INTERNATIONAL STANDARD

ISO 18400-104

First edition
2018-10

Soil quality — Sampling —

Part 104: Strategies

Qualité du sol — Échantillonnage —

Partie 104: Stratégies

Reference number
ISO 18400-104:2018(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 2, *Sampling*.

This first edition of ISO 18400-104, together with ISO 18400-101, ISO 18400-102, ISO 18400-105, ISO 18400-107, ISO 18400-202, ISO 18400-203 and ISO 18400-206, cancels and replaces the first editions of ISO 10381-1:2002, ISO 10381-4:2003, ISO 10381-5:2005, ISO 10381-6:2009 and ISO 10381-8:2006, which have been structurally and technically revised.

The new ISO 18400 series is based on a modular structure and cannot be compared to the ISO 10381 series clause by clause.

A list of all parts in the ISO 18400 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document is part of a series of sampling standards for soil (the role/position of the individual standards within the total investigation programme is shown in Figure 1). It provides guidance on the development of site investigation strategies in general (more specific guidance is given in other standards) and of sampling strategies [e.g. what to sample, where to sample (locations and depths) and the types of samples to take] taking into account the need to obtain representative samples and to have regard to relevant statistical principles.

Soils (and other soil materials) are composed of a mixture of mineral particles, organic matter, water, air (soil gas) and living organisms. In the case of some contaminated soils, a non-aqueous liquid phase might also be present. The solid matrix (phase), consists of particles of different size, shape and physical and chemical properties. The aim when carrying out soil sampling is usually to obtain sufficiently representative samples that can be used to characterize the properties of the whole soil entity (e.g. *in situ* soil in the form of a volume or horizon, or surface deposit such as a stockpile) or the portion considered relevant to the objectives of the investigation (e.g. <0,1 mm fraction for exposure assessment via hand-to-mouth activity). The properties of discrete entities such as individual soil particles are not addressed. As the soil as a whole cannot be analysed, soil samples are taken instead. The assumption that the results of these investigations on samples represent the total soil volume of interest is always an approximation, the reliability of which depends on additional information about the soil, the site and use of an appropriate sampling strategy. In other words, the sampling strategy should guarantee that, together with additional information (on-site observations, background information, previous investigation results, etc.), the results for the samples analysed allow a model to be developed of relevant properties of the soil volume of interest to a sufficiently reliable degree, in accordance with the investigation objectives.

Whatever the purpose of the investigation, a sound conceptual site model is required. Every property of a soil or soil material is a result of their dynamic development influenced by natural and human-induced processes such as weathering, leaching, dislocation, contamination, and many others. Without considering this, the results of any investigation of samples cannot be interpreted and evaluated properly. When spatial variability of soil properties (including contamination) is of particular interest, the conceptual site model includes what is known, or believed to be known, about the processes that led to the anticipated spatial distribution of properties.

The sampling strategy, especially when average properties are of interest, is preferably based on statistical methods, as far as practical and appropriate.

Having first defined key elements such as involved parties, objectives, properties of interest, phase of the investigation, background and site information, as well as health and safety aspects, a sampling strategy is developed that can form the basis of a sampling plan in accordance with ISO 18400-101 (the sampling plan covers a number of practical issues as well as the sampling strategy).

The appropriate sampling strategy in any particular case depends on

— the objectives of the investigation,

— the special situation and characteristics of the material to be sampled,

— the properties of interest, and

— the required degree of precision and reliability of the results.

Many other factors can also influence the design of the sampling strategy including:

— accessibility of the site as well as the sampled material;

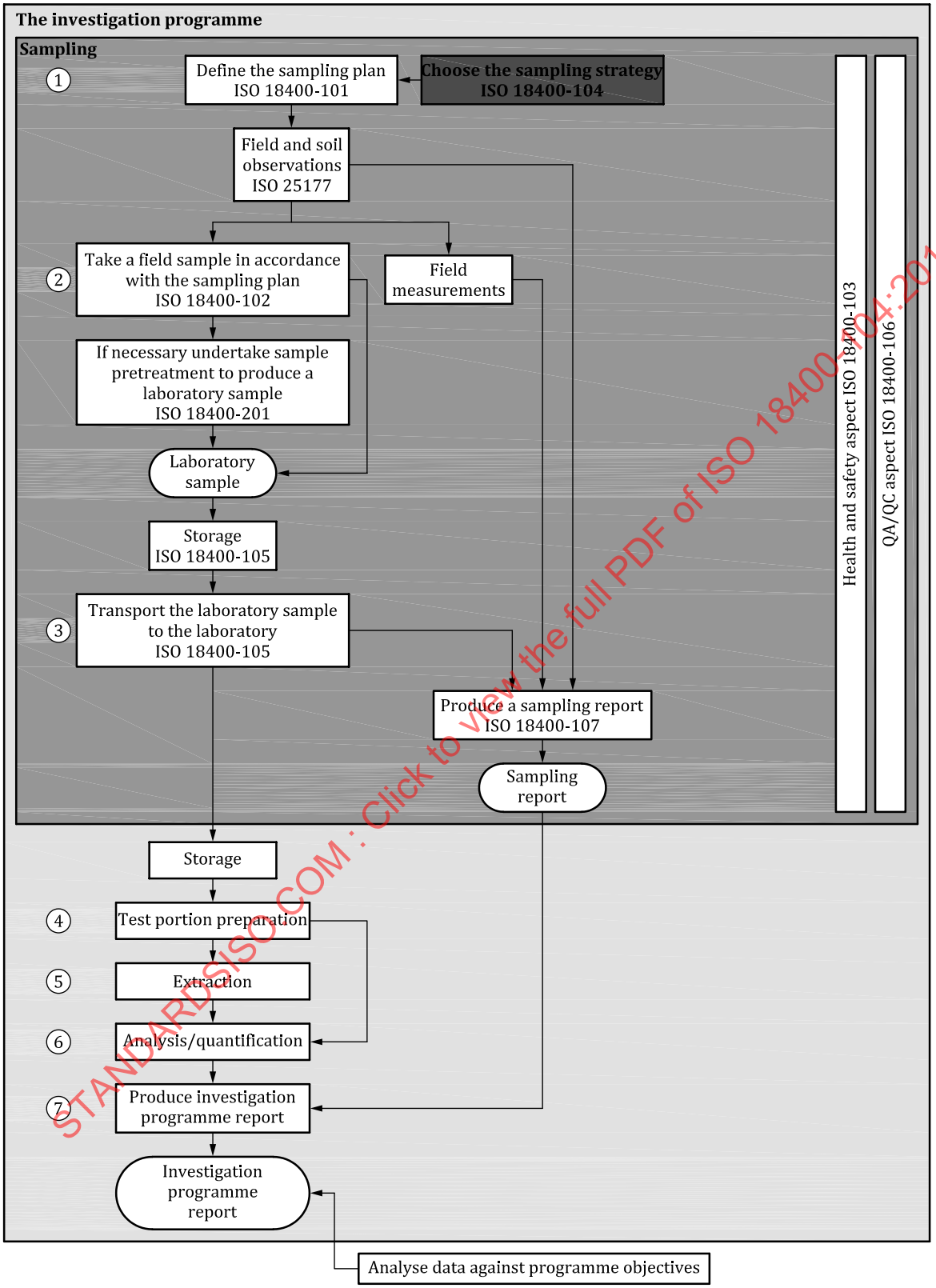
— financial, personnel, and technical resources;

— weather conditions;

— the time schedule/frame;

— legal/environmental restrictions.

Following the definition of the sampling approach, the appropriate sampling techniques are selected following the guidance in ISO 18400-102 with regard to health and safety (ISO 18400-103) and various practical considerations. The decisions made regarding sampling techniques form part of the sampling plan.



NOTE 1 The numbers in circles in Figure 1 define the key elements (1 to 7) of the investigation programme.

NOTE 2 Figure 1 displays a generic process which can be amended when necessary.

Figure 1 — Links between the essential elements of an investigation programme

Soil quality — Sampling —

Part 104: Strategies

1 Scope

This document gives general guidance on the development of site investigation strategies and detailed guidance on the development of sampling strategies, when collecting information on

— the average properties of soil,

— the variability of soil properties, and

— the spatial distribution of soil properties.

It is applicable to soil samples intended for chemical testing and determination of a variety of other properties (e.g. physical).

Although the main focus of this document is the collection of material (field samples) for transfer to a laboratory for testing, it is also applicable when measurements are made directly in the field.

NOTE 1 This document also provides information on the statistical principles underlying the development of appropriate sampling strategies and statistical methodologies.

NOTE 2 Guidance on other forms of related sampling activities are given in other International Standards [for soil gas (ISO 18400-204) and for biological testing purposes (ISO 18400-206)]. Guidance on sampling groundwater is provided in ISO 5667-11 and ISO 5667-22 and on sampling methods and groundwater measurements in geotechnical investigations in ISO 22475-1.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 11074, *Soil quality — Vocabulary*

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

ISO 18400-201, *Soil quality — Sampling — Part 201: Physical pretreatment in the field*

ISO 18400-202, *Soil quality — Sampling — Part 202: Preliminary investigations*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

NOTE The hierarchical approach and terms used in this document as well as the relationships between the sampling approaches and sampling patterns employed in this document are shown in Table 1.

3.1
above-ground sampling
taking samples from material that has been deposited on the ground surface

EXAMPLE Samples are taken from a stockpile (including bulk volumes of excavated soils), deposit of waste or embankment.

3.2
anthropogenic ground
deposits which have accumulated through human activity

3.3
cluster sample
composite sample (3.4) for which the *increments* (3.8) are taken over a small area around a predefined sampling point

Note 1 to entry: Sampled area is typically about 0,5 m^2 to 1,0 m^2.

Note 2 to entry: Material sampled is taken from within the same stratum or from material with the same characteristics.

3.4
composite sample
sample made of a number of increments (*cluster sample* (3.3) or *spatial composite sample* (3.22))

3.5
convenience sampling
taking samples based on accessibility, expediency, cost, efficiency, or another reason not directly concerned with sampling parameters

Note 1 to entry: The samples might be taken to a predetermined plan (locations, depths, etc.) or taken from locations and/or depths decided on site.

3.6
fill
anthropogenic ground (3.2) in which the material has been selected, placed and compacted in accordance with an engineering specification

3.7
ground
all materials below the ground surface, including natural materials (soil and rock) and anthropogenic materials

3.8
increment
material forming part of a *composite sample* (3.4) obtained by a single operation of a sampling device

Note 1 to entry: For instance, the filling of a scoop or auger.

3.9
in-ground sampling
taking samples from the ground surface and/or within the ground beneath the surface

3.10
judgemental sampling
targeted sampling
taking samples from particular zones or features of a site taking into account existing knowledge, including what is known about the history and layout of a site or a zone within a site

Note 1 to entry: Judgemental sampling could be required, for example, around underground storage tanks or pipelines where there might have been leaks, above ground storage tanks where there might have been spills, and for areas where raw materials or waste have been stored or deposited.

Note 2 to entry: Sampling locations are usually predetermined based on what is known about the target area but some locations may be selected in the field in response to on-site observations.

3.11
made ground
anthropogenic ground (3.2) comprising material placed without engineering control and/or manufactured by man in some way, such as through crushing or washing, or arising from an industrial process

3.12
macro-aggregate
soil aggregates consisting of micro-aggregates cemented together by organic matter, microbial polysaccharides, fungal hyphae, earthworm excretions and plant roots

Note 1 to entry: Generally, aggregates are >0,25 mm to 5 mm in size.

Note 2 to entry: Macro-aggregates are typically found in undisturbed soils.

3.13
micro-aggregate
soil aggregates consisting of primary particles, plant roots, and humin cemented together

Note 1 to entry: Generally, aggregates are less than 0,25 mm in size.

Note 2 to entry: Micro-aggregates are more typically found in disturbed or cultivated soils. Multiple micro-aggregates can form larger *macro-aggregates* (3.12) through microbial activity, plant root exudates and actions, fungal hyphae, and earthworm casts.

3.14
population
<soil sampling> entirety of a soil volume or mass about which information is to be sought via sampling

EXAMPLE A particular site, an *in situ* volume of soil, a stockpile, a truck load.

3.15
principal sampling situation
one of four sampling situations characterized by a combination of whether information is required on spatial distribution or average properties, with whether *in-ground sampling* (3.9) or *above-ground sampling* (3.1) is required

Note 1 to entry: The concept is illustrated in Table 2.

3.16
probabilistic sampling
taking samples to ensure that each particle or element in the *population* (3.14) has an equal chance of being part of the sample

Note 1 to entry: This means it is easy to obtain a quantifiable level of reliability (or uncertainty) in the estimated mean value and enables estimation of variability of the results for the population being tested.

[SOURCE: ISO 11074:2015, 4.2.10]

3.17
random sampling
simple random sampling
taking samples in locations selected arbitrarily within the area to be investigated

Note 1 to entry: The coordinates of the intended sampling locations are derived using pseudo-random or quasi-random numbers which can be found in tables included in manuals on statistics or generated by computer programs.

3.18
regular sampling
taking samples at the nodes of a regular pattern, such as a square or triangular grid, i.e. the sampling locations are evenly spaced

3.19
sampling pattern
set of predetermined sampling points

3.20
selective sample
sample deliberately chosen or formed based on some specific characteristic(s) of the material to be sampled

EXAMPLE Appearance, odour, particle sizes.

[SOURCE: ISO 11074:2015, 4.2.16, modified]

3.21
site investigation objective
statement regarding the information to be obtained during the investigation

3.22
spatial composite sample
composite sample (3.4) formed from small incremental point samples taken over an area (such as a field)

Note 1 to entry: The general premise is that the distribution of soil constituents is relatively homogeneous. The *increments* (3.8) may be located according to a regular grid, random, or other pattern. In agricultural/horticultural land investigations "N", "S", "W" and "X" *sampling patterns* (3.19) are commonly used. Along the outline of such a pattern, a number of samples or increments are taken which are bulked and mixed to provide one (composite) sample for analysis.

3.23
spot sample
sample from a discrete location

Note 1 to entry: Sample can be formed from one or more contiguous portions of material.

Note 2 to entry: Sample may be *disturbed* or *undisturbed*.

3.24
stratified random sampling
dividing the area to be sampled into a number of identical grid cells (strata) and taking samples arbitrarily in each cell

3.25
systematic sampling
taking samples from locations that have been pre-designated according to a geometric or other statistically derived pattern

Note 1 to entry: Systematic sampling can include a random positioning component.

Note 2 to entry: Some systematic sampling patterns are regarded as "*probabilistic*" (3.16).

Note 3 to entry: This definition is wider than the usual definition found in the literature, where systematic sampling commonly means *regular sampling* (3.18).

3.26
systematic unaligned sampling
taking samples using a *sampling pattern* (3.19) intermediate between a regular grid and *stratified random sampling* (3.24), where each row (respectively column) of the grid shows a similar pattern of unaligned points

Note 1 to entry: See Figure B.8.

4 Overall investigation strategy

4.1 General

The sampling strategy for soils and soil materials usually forms part of an overall site investigation strategy. This usually comprises a number of phases as described in 4.3.

NOTE 1 The soil sampling exercise that is being planned could be only one component of a wider investigation strategy that could also require collection of information relating to, for example, groundwater, geotechnical properties of the ground, archaeology, ecology, and soil gas and that for practical and logistical reasons a degree of integration between investigations into different aspects of the site might be required. How this can be done is outside of the scope of this document.

NOTE 2 Soils (and other soil materials) are composed of a mixture of mineral particles, organic matter, water, air (soil gas) and living organisms. In the case of some contaminated soils, a non-aqueous liquid phase might also be present. The solid matrix (phase), consists of particles of different size, shape and physical and chemical properties. The aim when carrying out soil sampling is usually to obtain sufficiently representative samples that can be used to characterize the properties of the whole soil entity (e.g. *in situ* soil in the form of a volume or horizon, or surface deposit such as a stockpile) or the portion considered relevant to the objectives of the investigation (e.g. <0,1 mm fraction for exposure assessment via hand-to-mouth activity). The properties of discrete entities such as individual soil particles are not addressed.

Sample is generally used in this document to mean the field sample. Sampling, therefore, means the collection of one or more field samples. The field sample usually equates to the laboratory sample [i.e. the material sent to the laboratory (ISO 11074:2015, 4.3.7)], although in some cases, the field sample is subjected to pretreatment in the field according to ISO 18400-201 to produce a sample of smaller size to send to the laboratory. The laboratory sample is usually subject, at least in the case of chemical analysis, to pretreatment in the laboratory (according to ISO 11464) in order to obtain a test sample. Physical and biological testing usually require a different approach and often require use of the whole of the laboratory sample. Information is provided in 6.6 and Clause 7 on the amounts of soil required primarily in the context of chemical testing but also for certain physical tests.

Table 1 — Sampling approaches and patterns

Convenience sampling	Judgemental sampling	Samples are taken from pre-designated locations							Spatial composite sampling
		Systematic sampling							
		Linear patterns	Circular patterns	Simple random sampling	Stratified random sampling	Unaligned sampling	Regular sampling		
							Rectangular grid	Non-rectangular grid	

NOTE 3 The terminology used to describe approaches to sampling and sampling patterns, etc. is not always applied consistently. Because of the variability in the use and understanding of terms related to sampling, it is important that those designing or carrying out site investigations explain what they intend to do, or have done, in ways that will be understood by non-experts and avoid reliance on specialist jargon.

The overall site investigation strategy should take into account:

a) the client's reasons for requesting the investigation to be undertaken (these should be clearly set out by the client);

b) the decisions that need to be made regarding the site or other area to be investigated;

c) the confidence required for making these decisions (this will determine the level of detail and accuracy of measurements required);

d) the findings of any investigations already carried out;

e) known data gaps and uncertainties (e.g. in the conceptual site model, see 4.4);

f) the findings of any risk assessment(s) completed to date.

A preliminary investigation (see 4.3.2) in accordance with ISO 18400-202 should always be carried out before any sampling or other intrusive investigation is carried out.

Once the preliminary investigation has been completed decisions should be made whether:

a) an exploratory and/or detailed (main) investigation is to be carried out, and in both cases, whether to carry out a single stage of sampling or two or more stages;

b) to zone the site, e.g. on the basis of past or current land use(s), intended use, or topography (if the site is zoned a separate sampling strategy will be required for each zone), see 4.2;

c) there are safety and environmental issues that could constrain the way that the sampling is carried out, see 5.9.

The objectives of the site investigation should be set before the investigation strategy is determined.

The objectives of a site investigation will vary, depending upon the stage in the process that has been reached and the underlying intentions for the land involved, but could, for example, be to provide:

— information on the pedological state of the site to be sampled;

— information on potential agricultural productivity;

— information on the physical and chemical state of the site to be sampled;

— information on contamination of the ground and groundwater;

— information on natural concentrations of potentially hazardous substances;

— the information needed to form, or further develop, a conceptual site model, including identification of potential pathways and receptors for the purposes of contamination-related risk assessment;

— support for a risk assessment;

— provide data for the design of remedial or protective works;

— provide data for re-use of soil materials (see ISO 15176) or for their disposal as waste.

As information is developed during an investigation, the impact on the objectives and the objectives themselves should be reviewed to determine whether these require modification or extension.

The formulation or refinement of a conceptual site model, see 4.4, should always be one of the investigation objectives and the model should be reviewed and revised in response to the results of the site investigation.

NOTE 4 The conceptual site model is to be no more detailed than required by the task in hand.

NOTE 5 Guidance on the investigations of particular types of site and in connection with the assessment for particular purposes is given in a number of other International Standards including: ISO 18400-205, ISO 18400-206, ISO 11504, ISO 15175, ISO 15176, ISO 15799, ISO 15800, ISO 16133, ISO 19258 and ISO 18400-203.

NOTE 6 An investigation (of potentially contaminated sites) are often carried out in one of the following contexts (see ISO 18400-203):

— in support of a proposal to develop or redevelop a site on behalf of the client (who might or might not be the site owner);

— in connection with the potential sale and purchase of a site to enable its value to be estimated, taking into account potential contaminated land liabilities and possible remediation requirements;

— to "benchmark" site conditions (e.g. contamination levels) for future reference;

— to determine whether it falls within a regulatory regime requiring remediation or some other form of response.

4.2 Zoning

Where logical and appropriate, the site should be divided into zones and separate conceptual site models and investigation strategies developed for each zone. Zoning may be based, for example, on:

— near-surface geology (e.g. made ground or natural ground);

— deeper geology;

— topography;

— probable absence or presence of contamination;

— previous, current or planned land uses;

— the nature of probable contaminants [e.g. volatile organic compounds (VOC) or inorganic compounds];

— known differences in soil types;

— observations on plant health;

— intended future use.

Zoning should be reviewed after each phase (and stage) of the investigation.

NOTE A stockpile or other above-ground deposit can be considered to be a single zone requiring separate investigation, or it can be divided into a number of zones, for example to reflect the method of formation (e.g. in layers or by end-tipping), or to reflect difference in appearance or on an arbitrary basis.

4.3 Types of investigation

4.3.1 General

Four principal phases of investigation are described in 4.3.2 to 4.3.5. The relationship between these phases and other investigation-related activities is shown in Figures 2 to 4. The person designing the overall site investigation should decide what phases of investigations are required, whether one or more of the phases should be carried out in stages, and the scope of each phase or stage of investigation.

Typically, investigations should comprise:

— a preliminary investigation (always required), see 4.3.2;

— an exploratory investigation (not necessarily required), see 4.3.3;

— a detailed investigation (required unless an exploratory investigation indicates otherwise), see 4.3.4;

— a supplementary investigation (sometimes required), see 4.3.5.

NOTE 4.3.2 to 4.3.5 have been written with particular emphasis on potentially contaminated sites because these are generally more complex to investigate than other sites, for example investigation of an agricultural field to determine the nutrient status would usually be a fairly simple task. However, the principles apply to all sites and are intended to be applied as appropriate in individual cases.

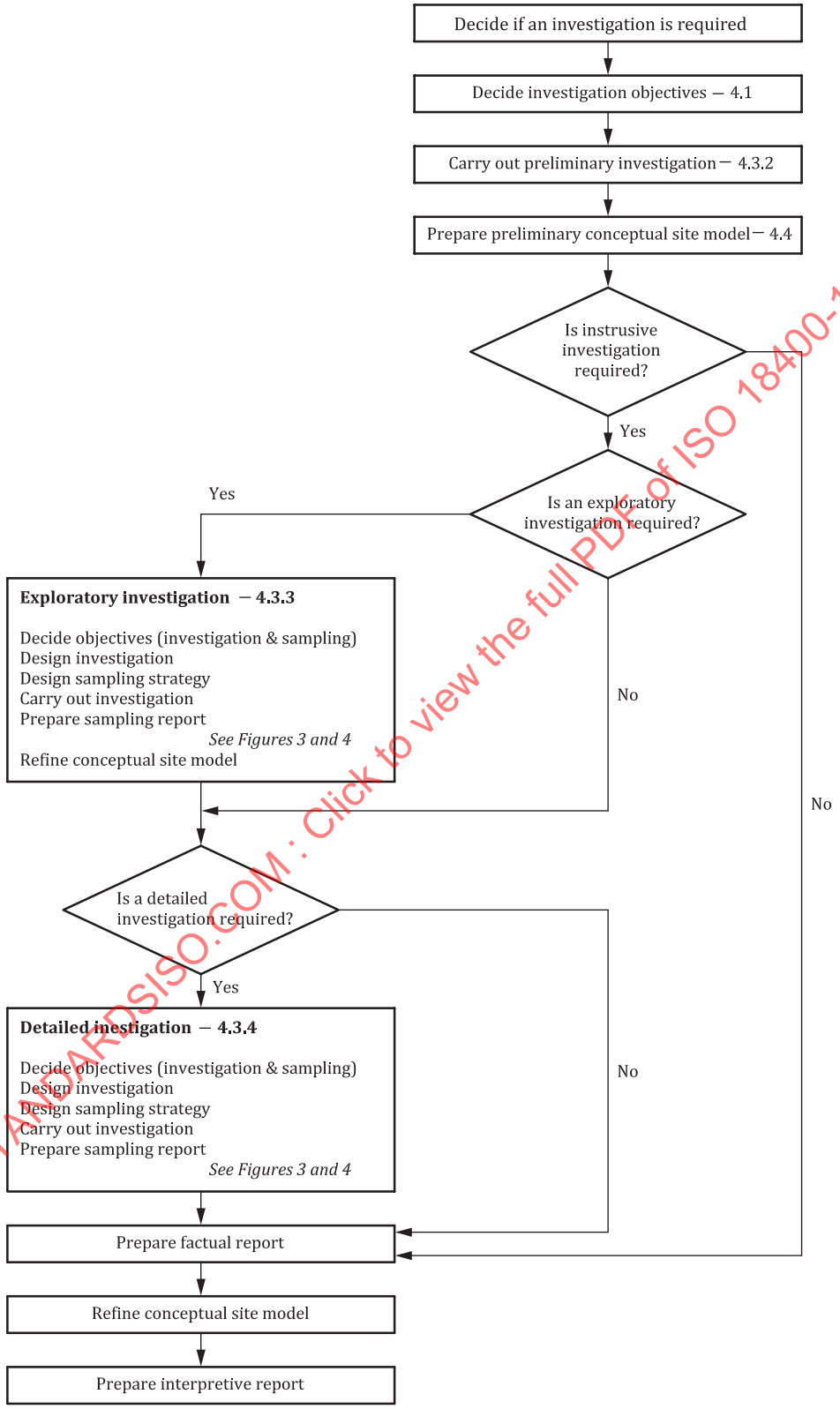


Figure 2 — Site investigation process

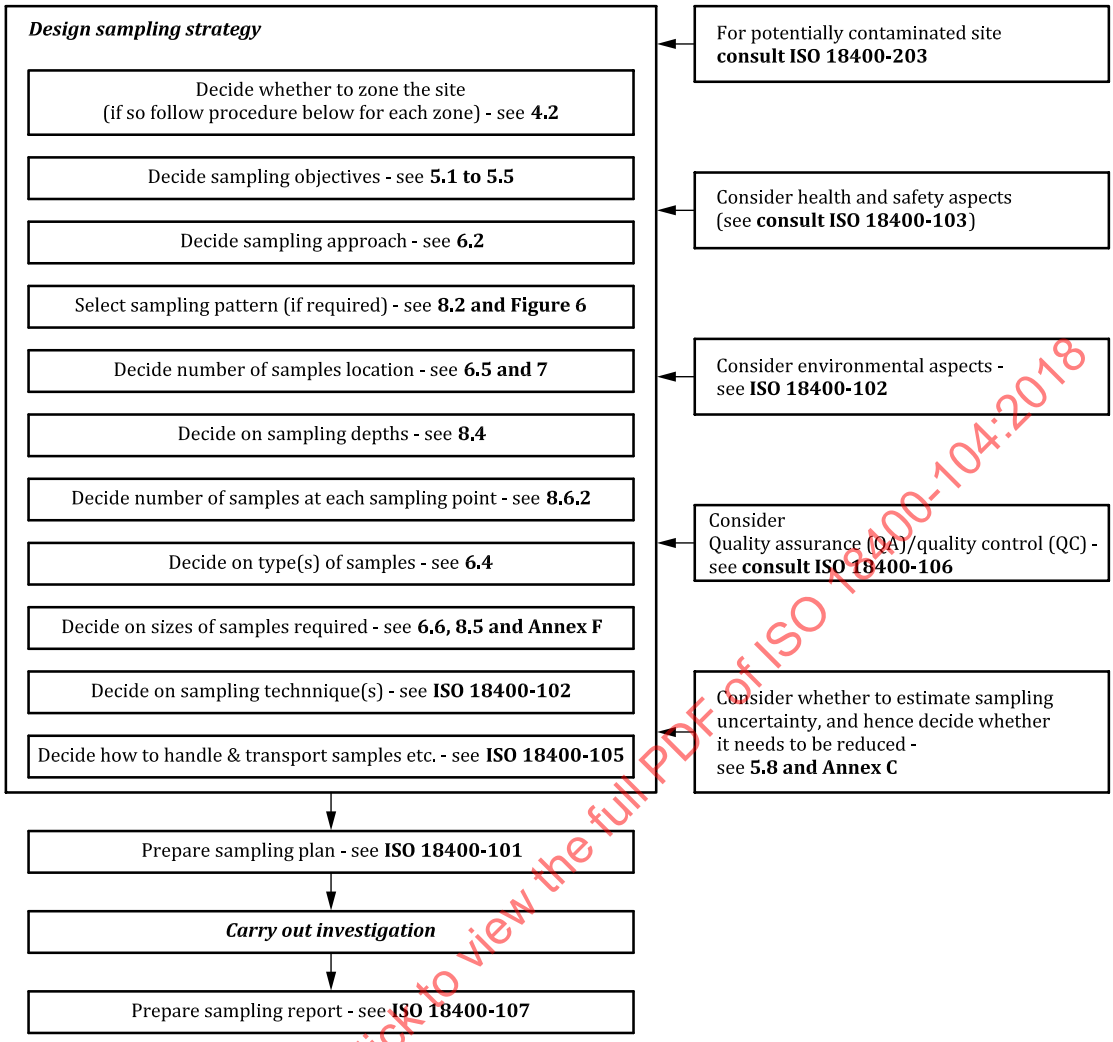


Figure 3 — Intrusive investigation process for potentially contaminated sites or above ground deposit

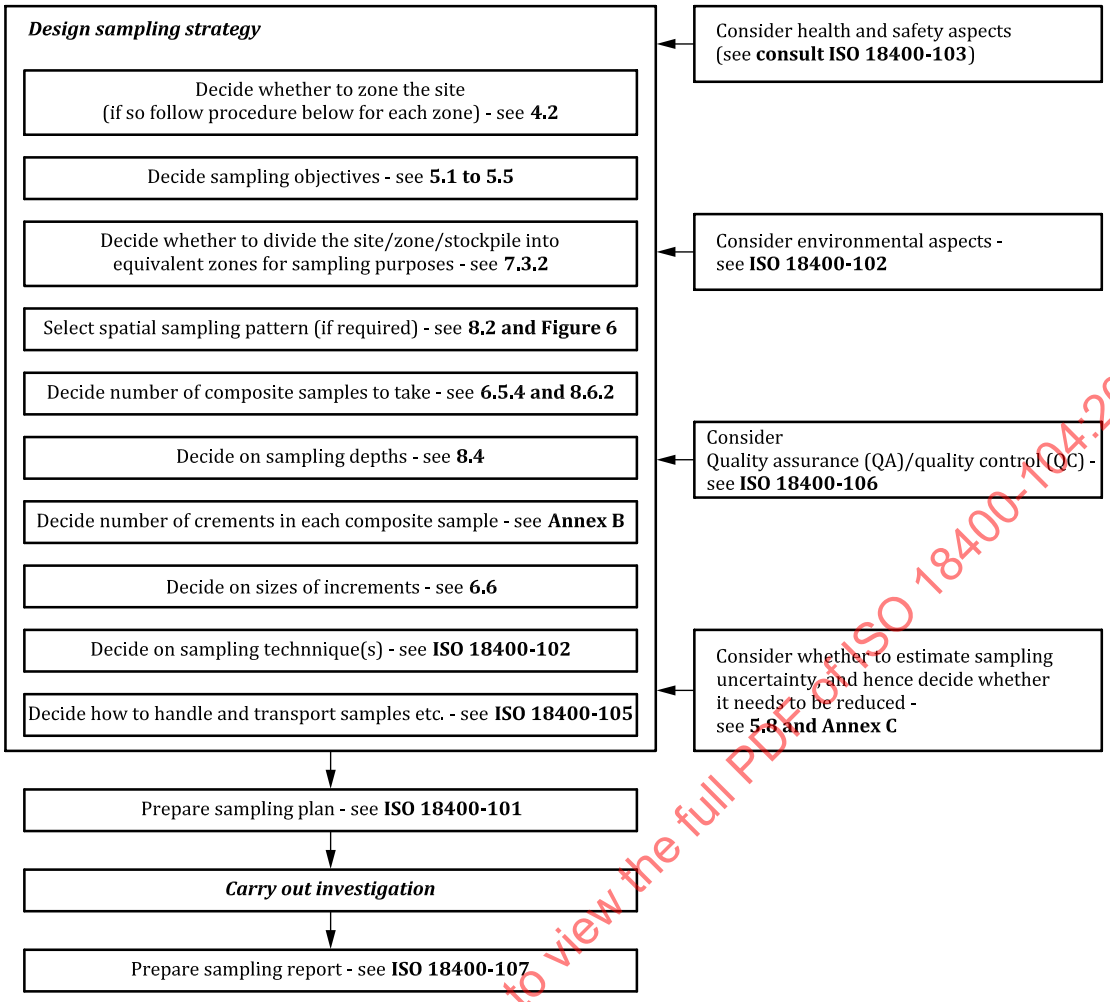


Figure 4 — Intrusive investigation process using spatial composite sampling to determine average properties

4.3.2 Preliminary investigation

The first phase in the overall investigation process should always be a preliminary investigation. This should include reference to historical records and other sources of information, consultation with relevant sources, and a site reconnaissance in accordance with ISO 18400-202.

The overall investigation strategy should provide for a review of the information obtained at the conclusion of the preliminary investigation to determine if the objectives have been achieved (and are still appropriate) and whether there is a need to carry out an exploratory investigation (see 4.3.3) and/or detailed investigation (see 4.3.4).

The output from the preliminary investigation should include an initial conceptual site model (see 4.4) and can include a preliminary risk assessment (see 4.5) based on the information available.

NOTE 1 Detailed guidance on preliminary investigations is provided in ISO 18400-202, including the type of information that could be required in relation to particular types of site, e.g. agricultural and near-natural sites, wooded sites and potentially contaminated sites.

NOTE 2 Preparation of a preliminary risk assessment requires interpretation of the information gathered during the preliminary investigation. Although in the case of a potentially contaminated site, a preliminary risk assessment will usually be an essential prerequisite for the design of further phases of investigation, guidance on how to do this is outside of the scope of this document.

4.3.3 Exploratory investigation

An exploratory investigation is a limited investigation and should be designed to:

— reduce uncertainty in knowledge of the site, including determining the validity or otherwise of contamination-related and other hypotheses developed in the preliminary investigation, thereby enabling the initial conceptual site model to be refined;

— provide information that helps the design of any subsequent detailed investigation.

NOTE 1 Exploratory investigations typically comprise one or more judgmental (targeted) sampling exercises although systematic sampling or composite sampling are also sometimes carried out.

NOTE 2 Detailed guidance on the design and execution of exploratory and detailed investigations of potentially contaminated sites, is provided in ISO 18400-203 and for natural and near-natural sites (e.g. agricultural) in ISO 18400-205.

NOTE 3 In the case of an agricultural site, an exploratory investigation could be used to confirm conclusions from the preliminary investigation about the soil type(s) present or to test a hypothesis, for example, that poor growth of vegetation is due to compaction and/or water logging. With this information available, the investigator can decide whether more detailed investigation is required, or that sufficient information is available to advise the client (e.g. the farmer).

4.3.4 Detailed investigation

The detailed investigation should, as far as is practical, provide all the information necessary to fulfil the objectives of the overall investigation.

In the case of potentially contaminated sites it should include collection of:

— all information necessary to test the validity or otherwise of contamination-related and other hypotheses developed in earlier phases of the overall investigation;

— all information necessary for the assessment of risks to all potential receptors;

— information relevant to the selection, design and costing of remedial or protective works.

NOTE 1 The detail required will depend upon the objectives of the investigation.

NOTE 2 When an exploratory investigation has not been carried out, it could be necessary to test a number of contamination-related and other hypotheses in the detailed investigation as would be done during an exploratory investigation.

NOTE 3 Detailed investigations of potentially contaminated sites typically comprise a combination of judgmental and systematic sampling exercises.

4.3.5 Supplementary investigations

A review of the outcome of the detailed investigation could identify aspects and areas where an unacceptable level of uncertainty or deficiency of information remains. Where such uncertainties or deficiencies are identified, a supplementary investigation should be carried out. This should be designed to produce specific information. It should use judgemental (targeted) and/or systematic sampling as appropriate.

NOTE In the case of potentially contaminated site, a supplementary investigation can be required, for example:

— to provide additional information needed to design and cost remedial works, for example, to delineate volumes of soil requiring remediation, or to carry out specific tests to evaluate the suitability of specific remedial treatment techniques;

— to provide chemical testing information for the classification of wastes;

— to monitor groundwater quality and levels, and/or soil gas conditions, after completion of the detailed investigation to determine any changes in site conditions and to provide additional data to assess risks and aid design of protective measures;

— to monitor groundwater and soil gas conditions during and after construction or remediation to determine whether there have been any adverse changes in site conditions.

4.4 Conceptual site model

A conceptual site model, embracing all information relevant to the objectives of the investigation, should be prepared prior to development of the sampling strategy. It should be presented in narrative, pictorial, tabular, or a combination of forms as appropriate.

The conceptual site model should be first formally formulated during the preliminary investigation (desk study and site reconnaissance) and should inform subsequent investigations, if these are necessary, to meet the objectives of the overall investigation. One of the objectives for the overall investigation should always be reduction of uncertainty in the conceptual site model.

NOTE 1 Although the conceptual site model is usually first formally prepared following a preliminary investigation, it first comes into existence the moment the question is asked whether the site needs to be investigated. At that stage, for example, it might be recognized that the site is agricultural land or is industrial land and the assessor will immediately form an initial picture about what the site might be like and act accordingly.

NOTE 2 The scope of, and level of detail in, the conceptual site model will depend on the objectives of the investigation and requires judgement on the part of the person preparing the model.

NOTE 3 The conceptual site model in the case of a potentially contaminated site is a description and/or representation of the site, incorporating what is known about the ground and groundwater conditions, the actual and potential contamination, the physical conditions and environmental setting, the current and past uses of the site, the receptors and potential pathway linkages between contamination sources and receptors, foreseeable events such as flooding, changes in groundwater level, global warming, extreme weather conditions, the closure of mines, etc.; in fact, all information relevant to the task in hand. Depending upon the objectives of the investigation, it could be relevant to consider new future receptors associated with the construction and completion of a new development, as well as existing receptors. The conceptual site model leads, for example, to the formulation of contamination-related hypotheses (see ISO 18400-203:2018, Annex A) which the investigation process examines through the collection of relevant data.

NOTE 4 An ISO Standard providing general guidance on how to develop an appropriate conceptual site model is planned. Guidance relating to particular objectives or site types is given in the relevant standards.

4.5 Preliminary risk assessment

In the case of a potentially contaminated site, a preliminary risk assessment should be undertaken once the initial conceptual site model has been formulated and the source-pathway-receptor linkages have been identified. Where adequate site investigation information has been obtained during the preliminary investigation, a qualitative, or generic-quantitative, risk assessment can be undertaken. Information from previous investigative works should be either verified or used with caution.

Where little or no previous investigation has been undertaken, only a qualitative assessment can be made. The effects of uncertainties in the information available on the outcome of a risk assessment should be identified.

NOTE Guidance on carrying out a formal risk assessment is outside the scope of this document (see also ISO 18400-203). Risk assessment covers:

a) identification of contaminants, pathways and receptors;

b) estimation of the likelihood, nature and extent of exposure to a hazard, and the risk of adverse effects;

c) assessment of the likely pollutant linkages and the degree of risk;

d) evaluation of the need for controlling the identified risk.

Site investigation provides baseline information for stage a) of the process.

5 Sampling strategies — General aspects

5.1 Sampling objectives

The sampling strategy, which forms the basis for the sampling plan (see ISO 18400-101), defines what samples are to be taken and where they are to be taken from. Before the soil sampling strategy is developed:

— a preliminary investigation should be carried out in accordance with ISO 18400-202;

— an initial conceptual site model should be developed, see 4.4;

— any safety and environmental issues (5.9) or other factors (5.10) that might constrain the way that the sampling is carried out should be identified;

— clear and appropriate objectives set.

Sampling objectives should be developed from the site investigation "objectives" (see 4.1) taking into account which of the principal situations defined in 5.4 apply to the site, whether any of the characteristic distribution of properties described in 5.6 is believed likely to be present, and any applicable regulations or guidance.

The objectives should include statements regarding what information is required including:

— whether information about spatial distribution of soil properties is required (see 5.6);

— whether information about average properties is required (see 6.3 and 7.3);

— what properties and characteristics are of interest;

— what aspects of variability are of interest;

— what "scale of sampling" is required (see Annex D and Annex E).

When intending to determine the average properties, the area, volume or feature for which the average is required should be carefully defined taking into account the purpose of the overall investigation. When the results of the investigation become available, the appropriateness of calculating an average for the previously defined entity should be carefully reviewed.

NOTE 1 It might, for example, be decided that the objective is to determine the average value of a property in the surface soil in a field. However, the investigation can show that there are two distinct soil types present and it is, therefore, more appropriate to calculate separate averages for the two soil types (this is of course not possible if only composite samples covering the whole field have been taken, see 6.4.5 and 7.3).

NOTE 2 There might be occasions when determination of other population parameters might be required.

There may be several reasons why information is required especially on a potentially contaminated site where it may be necessary, for example, to obtain analytical and other data to aid assessment of risks to human health and the environment, to assess materials for waste disposal and to comply with requirements in relation to the health and safety of construction workers. It is likely to be more economical if single samples, or at least a set of samples, can be taken to serve all purposes in a single operation. However, care should be taken not to compromise achievement of the different objectives for sampling.

5.2 Scope of the sampling strategy

The sampling objectives usually determine which type of sampling strategy is adequate and how reliable the sampling should be. When there is only an indirect relation between sampling objectives and sampling strategy, the programme manager has to define the quality and type of sampling explicitly.

Depending on the sampling objectives, the sampling strategy should define specific operational elements that will subsequently form the basis of the sampling plan (see ISO 18400-101) including:

— the soil to be sampled (the population), [this might, for example, be the site, a zone, horizon or above-ground deposit (e.g. stockpile)];

— whether the whole of the soil is to be sampled or whether there are particular fractions (e.g. based on size or appearance) that are relevant for the objectives of the investigation including whether selective sampling is required (see 6.4.6);

— the components to be determined and/or tests to be carried out on the samples;

— the statistical parameters to be determined (e.g. mean concentration, degree of heterogeneity, percentile);

— likely measurement uncertainty arising from different sampling approaches, see 5.8;

— the type of sampling (e.g. judgemental, systematic or composite, see 6.2.2 and 6.2.3);

— type(s) of sample to be taken, see 6.4;

— the density of sampling (e.g. distances between sampling points);

— sampling locations including sampling pattern;

— depth of sampling;

— size of sample(s), see 6.6;

— number of samples to be taken at each sampling point or over each area in the case of composite sampling, see 6.5 and Clause 7;

— definition of the desired level of uncertainty (i.e. bias and precision, see A.5.2, A.5.3) and confidence required (see also Annex C);

— phasing and staging of the investigation (see Clause 4).

It is important to recognize that there could be a variety of constraints on the works that can be carried out that should be taken into account when transposing the ideal sampling strategy into a sampling plan. For example, access, ground stability, the safety of those doing the work and the general public, and potential impacts on the environment while the works are in progress or at a later date (see 5.9 and 5.10).

NOTE Decisions on how the samples are to be obtained usually form part of the development of the sampling plan. However, the person developing the sampling strategy might wish to include preferences regarding the techniques to be used. Guidance on the techniques that can be used to obtain samples and their application is provided in ISO 18400-102.

5.3 Designing the sampling strategy

The designer of the soil sampling exercise should have an understanding of how the information that is to be gathered is to be used including any statistical analyses that are likely to be employed (see 5.6), of the factors that can contribute to sampling errors and uncertainty (see 5.7) and what would constitute a sufficiently representative sample in the context of the investigation (see 5.5).

The sampling strategy should take into account:

— the objectives of the investigation;

— the possibility of zoning the site (see 4.2);

— the conceptual site model developed to date (see 4.4);

— site-specific factors, (for example, the site size and topography, physical obstructions);

— the location of buried services, etc.;

— the need to avoid creating routes for migration of either gas or (contaminated) groundwater;

— protection of health and safety and the environment (see 5.9 and ISO 18400-103);

— temporal effects and the significance of such effects on the results of the investigation;

— locations and extent of any proposed buildings and infrastructure;

— off-site considerations;

— how data and other information used to inform risk assessment are to be analysed and presented so as to aid understanding of the data.

5.4 Principal sampling situations

The sampling strategy and the statistical methods to be used should take into account which of the following two sampling situations is applicable to the site or zone that is to be sampled:

a) sampling is required at the ground surface and/or in the ground beneath the surface, i.e. in-ground sampling;

b) sampling is required of materials that have been deposited on the ground surface, i.e. above-ground sampling.

NOTE 1 Ground is used here to mean all materials below the ground surface, including natural materials and anthropogenic materials (i.e. materials resulting from human activity). The ground surface might be formed by natural *in situ* materials or anthropogenic deposits in the form of (engineered) fill or "made ground".

For each of these sampling situations, decisions should also be made whether information is required about:

— spatial distribution of soil properties;

— average properties.

Thus, there are four principal sampling situations that might be encountered as shown in Table 2.

Table 2 — Principal sampling situations

	In-ground sampling	Above-ground sampling
Spatial distribution	**I** In-ground sampling to determine spatial distribution of properties	**III** Above-ground sampling to determine spatial distribution of properties
Average properties	**II** In-ground sampling to determine average properties	**IV** Above-ground sampling to determine average properties
NOTE "Spatial distribution" is used here with a general, geographical meaning: the arrangement or pattern of areas where a soil property has large values (e.g. exceeds a threshold), or the arrangement or pattern of contaminated areas (see also 5.6).		

Whatever the principal sampling situation, a decision should be made about how to address the variability of soil properties, taking into account the guidance in 5.6 about scale, etc. and 5.8 about sampling uncertainty.

When in-ground sampling is to be carried out, it is almost always possible to form exploratory holes in the surface in order to investigate the ground beneath. Obviously, not all sites are flat and the ground could be sloping, uneven, or in places of low bearing capacity; and access to all parts of the site might be difficult for a variety of reasons. Nevertheless, it is possible to envisage placing a two-dimensional pattern across the site and then sampling at varying depths at each sampling location. The depth

intervals for sampling will typically be much less than the spacing between the nodes of the sampling pattern (see B.1.3 and B.12).

In contrast, the starting point for sampling a stockpile or other above-ground deposit is to assume that the main interest is in average properties and that an equant three-dimensional sampling pattern is required. In practice, this will often be difficult to achieve for a variety of reasons (see Clause 9) and a pragmatic approach to sampling based on the expert judgment will often be required (see B.12). One of the major difficulties is that stockpiles and similar deposits are usually unconsolidated and thus unstable.

Engineered deposits such as rail and road embankments present other problems. Although there could be a level surface, this will often be occupied by a road or railway limiting access except when absolutely essential. Sampling on the sloping sides is then likely to be required. This presents considerable practical and safety issues, will often require the use of specialist and expensive equipment, and thus also require expert judgement about how and where to sample.

Some natural deposits (e.g. landslips, recent deposits of volcanic ash, scree slopes) can have similar characteristics to anthropogenic deposits in that they are generally unconsolidated and unstable, and are often underlain by sloping ground, e.g. landslips. The investigator will have to decide how best to approach such ground taking into account the information that is being sought and the paramount need for safety.

NOTE 2 Type a) sampling situations where in-ground sampling of soil materials is to be carried out include, for example, agricultural or sylvicultural soils, potentially contaminated sites and landfilled materials (see Note 4).

NOTE 3 Type b) sampling situations include, for example, embankments, waste dumps, and stockpiles of such materials as wastes, processed soils, manufactured soils and excavated materials.

NOTE 4 In industrial and other urban sites, the soil is seldom the original undisturbed soil: the soil might have been "disturbed" during building and other works, buildings might have been demolished and the waste spread around, the soil surface might have been smoothed with a bulldozer. Consequently, the soil material can be very heterogeneous. A dump from a mine or from steel works will usually have been formed over a number of years (sometimes decades) with a variable composition of the waste (some are now mined as true ore bodies). In both cases, the present soil results from these transformations and its properties can be studied as depending on the *x, y, z* coordinates. In contrast, a truck load or a temporary stockpile of loose material deposited on the surface of an otherwise level site has to be approached in a different way.

NOTE 5 Investigations to determine spatial distribution of properties can also provide information on average properties (see 6.3 and Clause 7).

5.5 Representative and sufficiently representative samples

Soils (and other soil materials) are composed of a mixture of mineral particles, organic matter, water, air (soil gas) and living organisms (see Note 2 in 4.1). As the soil as a whole cannot be analysed, soil samples are taken and investigated as representative of the whole soil. The assumption that the results of these investigations on samples represent the total soil volume of interest is always an approximation, the reliability of which depends on additional information about the soil, the site and use of an appropriate sampling strategy. In other words, the sampling strategy should guarantee that, together with additional information (on site observations, background information, previous investigation results, etc.), the results for the samples analysed allow estimation of relevant properties of the total soil volume under investigation (or that part of interest) to a sufficiently reliable degree, in accordance with the sampling objectives.

Whatever the purpose of the investigation, a sound conceptual site model should be developed, which when spatial variability of soil properties (including contamination) is of particular interest, should include what is known, or believed to be, the processes that led to the anticipated spatial distribution of properties. The sampling strategy, especially when average properties are of interest, should be based on statistical methods, as far as practical and appropriate.

5.6 Characteristics of the spatial distribution

Soil materials are inherently variable in composition. A key element of sampling programme design is an understanding of the main components of variability in the soil being sampled (the population).

Two distinctive types of variability can be distinguished:

— at very small scale, that of soil particles, i.e. fundamental variability;

— at large scales, i.e. spatial variability.

NOTE 1 Scale is an important concept, as heterogeneity is a scale-dependent characteristic: soil properties show less heterogeneity with large-scale measurements than with small-scale measurements. Two scales of sampling are usually considered: the scale of the sample and the scale of the assessment. These rarely coincide. A discussion about the scale of sampling is provided in Annex E.

NOTE 2 One cause of spatial variability can be temporal variability in the nature and composition of the soil or soil materials making up the soil volume (population that is to be sampled). Examples of when this might be the case include:

— when materials have been deposited in stockpiles over a period of time;

— when materials have been deposited on the ground surface by spreading or end tipping over a period of time;

— when there have been a number of flooding events;

— when there have been a number of land slips or similar events.

When temporal effects are expected based on the process wherein the stockpile or other deposit was formed, these effects should be accounted for in the assumptions on the spatial variability.

NOTE 3 Fundamental variability and spatial variability are discussed in detail in Annex A.

NOTE 4 Spatial variability usually occurs at all scales from that of sample increments to that of large volumes or even the whole site of interest.

NOTE 5 How a substance or particular material is distributed within a soil is of particular importance. For example:

— a contaminant (e.g. cadmium) might be concentrated in the finer fractions of a soil which form a reasonably constant portion of the soil. In this case, the analytical result obtained will be similar whether just a few hundred grams are taken to form a sample or whether many kilograms are taken, i.e. it is comparatively easy to obtain a representative sample; or

— the contaminant might be concentrated in slag particles, of say about 10 mm diameter distributed unevenly through the soil. In this case, if the laboratory sample is too small, it might not include any of the slag particles, or unless it is realized in advance that cadmium might be present in this way, the >10 mm might be rejected as "stones" and of "no interest" by the laboratory unless instructed otherwise thus leading to a misleading analytical result. In this case, a particle size analysis and separate analysis of fractions might be the best approach. However, for meaningful results, it would be necessary to take large samples (how large would depend on the number of particles/m^3); or

— the substance of concern might be present as discrete particles or fragments of material distributed unevenly through the soil, e.g. lead shot in areas where game birds are regularly hunted; fragments of asbestos-containing materials; asbestos fibres; or pieces of copper wire; plant roots.

In the third example there is usually no spatial relationship between the occurrence of one particle/fragment and another (e.g. there is no concentration gradient between them) although if it is known how they arrived in the soil it might sometimes be possible to predict areas where particles/fragments are more likely to be present.

Analogous to the third example is when there is combustible carbon present in a soil. Whether this is evenly distributed, or present in discrete lumps (e.g. fragments of coals or wood) the calorific value might be the same. However, the ignitability, combustibility and ability to sustain smouldering could be very different.

It is important to take into account that the substance of concern might:

— occur predominantly in solution in the pore water and that, consequently, care should be taken to capture and retain the water during sampling;

— be present as, or occur in, a non-aqueous phase;

— be concentrated in plant roots having been taken-up or adsorbed from the soil.

The anticipated variability at varying scales should be considered when developing the conceptual site model (see 4.4) and the subsequent investigation should include among its objectives an aim to "disclose" this variability. Achieving this aim requires that samples are taken from sufficient sampling locations selected by application of an appropriate sampling pattern(s), taking enough samples at each location (see 6.5) and taking samples of sufficient size (see also 6.6, Annex B and Annex F).

5.7 Statistical aspects

An understanding of certain basic statistical principles and concepts is essential for the design of sampling strategies and to the processing, presentation, analysis and assessment of the results of sampling exercises (see 6.1, Annex A and Annex H which describe basic statistical principles and concepts and their application in soil sampling programmes). Of particular importance is an understanding of such concepts as fundamental variability (see A.3.3) and spatial variability (see A.3.4).

5.8 Uncertainty of measurements caused by sampling and analysis

Overall uncertainties in the measurement results obtained during site investigations originate from a combination of measurement uncertainties arising from the laboratory procedures and those that arise from sampling and sample preparation prior to analysis or testing. The uncertainties arising from laboratory analytical procedures can be estimated and controlled, for example, by using a laboratory running appropriate quality assurance and quality control procedures (see ISO 18400-106).

The uncertainties associated with sampling tend to be much larger than those arising from analysis and testing and are more difficult to control and estimate. Depending on the heterogeneity of the site and the nature of the contamination or other property of interest, changing a sampling position by only 100 mm or so could in some cases have a marked effect on the result.

There are methods that can be applied when trial pitting, for example, that can be used to estimate sampling uncertainties (see Annex C). While it is an initial priority to have a sufficient number of sampling locations, it is also important to have an estimate of the sampling uncertainty, for example to decide whether cluster sampling might be employed in the detailed investigation, to reduce this uncertainty.

Uncertainties also arise because the process of sample preparation is not always well-controlled and varies between laboratories despite the existence of relevant International Standards intended to guide the process. For example, there can be differences in the size of material separated as "stones", the methods of grinding and subsampling, and the fineness of the analytical sample.

Further uncertainties can arise from mistakes when samples are taken in the field (see 5.10). These should be controlled by establishing standard working practices (see ISO 18400-106 and References [26] and [27]).

Because of these inherent measurement uncertainties, it is important not to treat laboratory measurements as "true" values, or to place undue reliance on single analytical results, and also that assessors employ their experience and professional judgement when interpreting data.

5.9 Safety and environmental protection

Sampling strategies should take into account the need to protect the health and safety of those carrying out the work and of the general public, and the need to avoid causing adverse environmental impacts during the works or at a later date (e.g. contamination of groundwater).

NOTE Guidance on the safety aspects of site investigation are provided in ISO 18400-103. Guidance on the selection of sampling techniques and their application taking into account health and safety and protection of the environment is provided in ISO 18400-102.

5.10 Barriers to sampling

It is not always possible to sample at a planned location (e.g. on a regular grid pattern) due to a variety of reasons (e.g. presence of a tree, large rock, building, buried foundations or services, difficulties of access). Contingency plans for dealing with such situations should be made in advance. The action to take depends on the circumstances. The investigator might ignore the point or follow predetermined rules for choosing a nearby substitute location (e.g. alternative position within 10 % of the grid space, or paired sampling along grid lines either side of the obstruction). *Ad hoc* decisions made in the field as they arise can lead to bias. An attempt should be made when marking out the site to identify such obstructions in advance of actual field work.

Similarly, when judgemental sampling is being employed, site constraints might limit where samples can be taken from so that an element of "convenience" is often inevitable when selecting sampling locations. For example, safety zones around underground storage tanks will limit where holes can be drilled on a vehicle fuelling station so that the locations that have to be used might not be those that would give the highest probability of detecting a leak. However, the locations where drilling is to take place can still be pre-selected so this is not "true convenience sampling".

In all cases when a sampling point has to be relocated, this and the reasons for relocation should be clearly indicated in the sampling report (see ISO 18400-107).

Preliminary investigations as described in 4.3.2 should provide as much detail as possible about conditions expected to exist on the site, and should therefore guide the design and execution of the sampling programme. However, it cannot totally protect against the danger of misinterpretation of the results of intrusive investigations, and the selection of sampling points should take this into account.

Examples of possible misinterpretation include:

— very thin soil layers or horizons which are not recognized in a core sample, sometimes due to smearing at tube wall contacts;

— erroneous correlation of horizons at different soil profiles in the same area;

— anomalies in the soil "missed" by intrusive investigations, e.g. foundations of buildings, spots of waste, barrels, tanks;

— erroneous indication of bed rock upper limit due to encountering lumps of rocks;

— erroneous indication of bed rock upper limit by "hitting" the shoulders of a groove;

— missing the natural sequence of layers/horizons due to near-vertical layering (occurs especially in geological material and in fill and wastes when "end-tipped").

Certain geophysical methods can give useful information that can help to avoid such misinterpretations. The merits of using such methods will depend on the objectives of the study and a variety of site-specific factors (see ISO 18400-203:2018, Annex B.

5.11 Timing of investigations

In some circumstances, it could be necessary to restrict sampling to specific periods of the year. For example, if the characteristic or substance to be determined is likely to be affected by seasonal factors

or human activities (weather, soil conditioning/fertilization, use of plant-protective agents or presence of nesting birds).

NOTE This is particularly important where monitoring lasts several months or years, or is continued periodically and therefore requires similar preconditions every time sampling is carried out.

6 Sampling strategies — Key aspects and concepts

6.1 Statistics and geostatistics

6.1.1 General

NOTE Clause 6 is primarily informative but there are some recommendations within some of the clauses (e.g. in 6.6). It is included in the main text of this document because an understanding of certain key concepts is essential for the application of the guidance provided in Clauses 7 to 9.

Statistical and geostatistical methods are used to characterize parameters of the variables of interest (e.g. contaminant concentrations) and the uncertainties in these parameters from the sample data. If the uncertainties are too large, these methods are also used to design further sampling in order to achieve objectives for parameters such as the concentration in a grid cell. This can also be done at an early sampling stage by reference to similar situations.

Both the spatial variability in concentration, and the measurement uncertainty of concentration caused by sampling and analysis, can be quantified as standard deviation (or its square, variance). The total variance can be sub-divided to identify its source (e.g. within- or between-sample) (see Annex C).

Neither statistical nor geostatistical methods can be used in the case of judgemental sampling (e.g. data specially located in areas assumed as likely to yield high contaminant concentrations) except in the context mentioned in 6.1.3 when judgemental sampling is used in combination with a regular pattern.

6.1.2 Statistics

Statistics of independent random variables offer techniques for estimating parameters of a statistical distribution and confidence intervals: mean value, standard deviation, probability not to exceed a given threshold, etc.

The approach is valid when the medium is so heterogeneous that there is no spatial structure. Another way to get independent samples is to use a random sampling pattern. This is, however, not a reason for doing so, because a stratified random pattern, as well as a regular grid, leads to more precise estimates.

NOTE Detailed information on statistical concepts is provided in Annex A and on their application in Annex G.

6.1.3 Geostatistics

A preliminary to the application of geostatistics is the characterization of spatial and/or temporal variability with the variogram and other spatial exploratory analysis tools.

NOTE 1 Values at locations that are close together are more similar than those further apart. They depend upon one another in a statistical sense. This property, known as spatial dependence, is the basis for geostatistics or spatial statistics. In this approach, the similarity or dissimilarity of the values taken by the variable of interest at two different sample points is statistically represented by a function of their distance, the variogram.

A geostatistical approach can be used in most situations not covered by statistical formulae, if:

— the scale of interest does not coincide with the sample scale (e.g. the interest lies in properties of large cells whereas only spot samples are available);

— the concern is in local properties rather than global properties (e.g. the average value of a property in a given cell, or the probability that this average property exceeds a given limit);

— the sampling pattern is random or regular;

and also:

— when sampling points are (within certain limits) unevenly distributed provided that sampling is not systematically focused on specific areas such as locations where high concentrations are expected;

EXAMPLE 1 Declustering techniques enable the use of a combination of a regular pattern and of judgemental samples in areas where large concentrations are likely.

— auxiliary information is to be incorporated.

EXAMPLE 2 A qualitative property, or a secondary property linked with the property of interest and known at any location.

Although application of geostatistics is proven when certain criteria are met, it can be unhelpful if they are not. For example:

— the objectives of the site investigation should require spatial resolved estimates of concentration; if the requirement is simply for the estimation of the mean concentration of a site, the application of geostatistics is unlikely to be worth the extra cost;

— the site should be suitable in terms of size and complexity; if the site is small there could be issues with justifying the large numbers of sample required ($n > 100$ is desirable even if a spatial exploratory data analysis can be of interest with much less data), and if it is known to be made up of several areas of different materials, there might well not be broad trends of geochemical variation that will fit sufficiently well to a single variogram model. It will be necessary to study each area separately, with the risk that some of them do not have enough samples for a sound geostatistical approach.

The sampling strategy also has to be suitable, not only in ensuring the required total number of samples, but also in ensuring that their coverage of the site is even enough to not leave gaps where the interpolation of concentration values is highly uncertain. Probably the most important requirement is for sufficient expertise in the personnel undertaking the designing of the sampling plan and the subsequent geostatistical interpretation.

Many software packages purport to provide geostatistical interpretation of measurements made from site investigations, but in inexperienced hands, they can produce apparently convincing output that could have little basis in reality. These comments more generally apply to all interpolation methods and models aiming at predicting concentration estimates at unsampled locations. Whatever the characterization approach chosen to support remediation decisions, expertise in sampling design and experience in site characterization is obviously fundamental to their proper application.

NOTE 2 See Annex H for a detailed discussion of geostatistical methods for sampling design and evaluation of soil quality.

A series of geostatistical methods, named “kriging”, enable the calculation of optimal estimates (in a statistical sense) and of the property of interest at unsampled locations or over grid cells. It also provides confidence intervals. An alternative to kriging, called “conditional simulations”, is to generate fields that mimic the variations of detail of the real variable; they are used as substitutes for the unknown reality to evaluate, for example, contaminated volumes, pollutant mass, or the uncertainty of the cost of remediation. These methods offer the possibility of anticipating the gain in precision that will be obtained with supplementary samples, and thus to optimize the design of further sampling plans.

6.2 Approaches to sampling

6.2.1 General

There are two principal approaches to the determination of spatial distributions:

— judgemental sampling is based on already available information (prior knowledge) and expert judgement – although this can provide valuable results at reasonable efforts, these results cannot be considered as representative in a statistical sense;

— systematic sampling is based on statistical considerations and requires a predefined sampling pattern before starting the field work (a variety of sampling patterns is available depending upon the situation to be explored).

Sound practice is often to combine the two approaches.

NOTE The approaches to sampling described in 6.2.2 to 6.2.4 and their relationship to one another are illustrated in Table 1.

6.2.2 Judgemental sampling

"Judgemental sampling", sometimes called "targeted sampling" or "preferential sampling", is where samples are collected taking into account what is known about the history and layout of a site or a zone within a site. It is the best approach for situations wherein a characteristic (e.g. concentration) of a constituent is to be determined at a specified location (e.g. a small area where spillage has occurred, for the delineation of the area or volume of soil affected). Other examples where judgemental sampling might be required include around underground storage tanks or pipelines where there might have been leaks, above ground storage tanks where there might have been spills, areas where raw materials or wastes have been stored and known deposits of wastes in the ground.

Judgemental sampling can be supplemented by systematic sampling (see 6.2.3) in order to confirm that there are no other areas of contamination. This is particularly important for old industrial sites and other potentially contaminated sites whose history is not precisely known.

Within the sub-area of a site to be subjected to judgemental sampling it is still possible, and often sensible, to adopt a localized sampling pattern to ensure that the area in question is examined in its entirety and as far as possible the extent of the affected area or volume can be delineated.

The use of judgemental sampling in situations where the average characteristic is to be determined will nearly always result in samples being taken from a sub-population, which is substantially more restrictive than the whole population. But within that sub-population it is feasible that the sampling could be "systematic", see 6.2.3. This means that provided that the sampling is indeed systematic for that sub-population, the results will still be representative for the part of the population sampled (within which the conditions for systematic sampling are met), though it still runs the risk of exhibiting a larger uncertainty for the whole population.

Judgemental sampling should not be confused with "convenience sampling" or "*ad hoc* sampling" which is undertaken on the basis of accessibility, expediency, cost, efficiency, or for other reasons not directly concerned with sampling parameters (see 6.2.4). There is no way of assessing the uncertainty in any subsequent data that results from the sampling steps.

The uncertainty resulting from judgemental sampling is highly dependent on the quality of the background information, on which any expert judgement and ultimately the sampling plan, is based. This will be especially sensitive for new sampling scenarios where there is an absence of suitable information or validation results.

Over reliance on "prior knowledge" can lead to unjustified assumptions being made that parts of a site are not contaminated and need not therefore be sampled at high spatial density, hence the need often to combine systematic sampling with judgemental sampling and/or to target areas assumed to be uncontaminated.

6.2.3 Systematic sampling

Systematic sampling as defined in this document involves taking samples from predetermined random locations (random sampling) or non-random locations determined by use of sampling patterns.

Some systematic sampling patterns are regarded as “probabilistic”, i.e. they ensure that each particle or element in the population (e.g. *in situ* volume of soil or stockpile) has an equal chance of being part of the sample. This means it is easy to obtain a quantifiable level of reliability (or uncertainty in the estimated mean value and enables estimation of variability) of the results for the population being tested. However, non-probabilistic sampling patterns can also yield this information if the appropriate statistical methods are applied to the results.

The principle types of systematic sampling are:

— simple random;

— stratified random;

— regular sampling; and

— unaligned sampling.

Simple random sampling involves selecting locations randomly in the domain of interest (e.g. a field or other site) and independently of one another.

Stratified random sampling involves selection of each sampling point randomly within the cells of a regular sampling pattern.

Regular (systematic) sampling involves taking samples at the nodes of a regular pattern, such as a square, triangular or hexagonal grid.

Systematic unaligned sampling involves taking samples off-set in both a systematic and random way from the nodes of a regular grid (this improves the chances of locating an elongated area of atypical properties).

When a regular grid is employed the initial location from which the nodes of the grid are derived is selected at random.

Systematic sampling can be performed stepwise: if the results exhibit too high an uncertainty, it is possible to take additional random samples or to refine the grid, which will result in an improved measure of uncertainty, at an additional testing cost.

Systematic sampling can be supplemented by judgemental sampling at locations where high concentrations are suspected.

For the same sampling effort (i.e. a given number of samples), simple random sampling is always less efficient than stratified random sampling, which is itself usually less efficient than regular sampling [efficiency measured in terms of estimation variance of the concentration, and at locating a hot spot (see 8.2.3 and Annex I)].

In terms of statistically efficient sampling, a regular equilateral triangular grid provides the best selection of sampling points. For a square grid with one node per unit area, the distance of a point to the nearest sampling point never exceeds 0,707 units of distance, i.e. the greater ease of use of the square grid is offset by the slightly greater area of un-sampled site

NOTE Further guidance on sampling patterns and their application is provided in 8.2 and 9.2 and Annex B and Annex I.

6.2.4 Convenience sampling

Some investigations are carried out without predetermined sampling locations. There could, for example, be circumstances in which the application of either of the above two approaches is not possible

because, for example, access around a site is severely limited, so that samples can only be taken in limited areas which might not be those that are actually of most interest on the basis of the site history. For example, on a vehicle dismantling site, the areas of interest could be beneath stockpiled vehicles or there might be growing crops in a field that limit access during the season when the work is being carried out (see 5.11). In such situations, only *convenience* sampling can be carried out. This should not be confused with the application of random distribution of sampling points, because persons usually cannot distribute sampling points randomly without preparation, i.e. they cannot ensure that at every point in the area, despite the position of the other sampling points, a sample will be obtained with equal probability.

When sampling is to be carried out without a predetermined plan, care should be taken that sampling is carried out by an appropriately experienced investigator.

6.3 Average properties

Average properties can be derived using composite samples or by averaging the results of investigations to determine spatial distributions. In both cases, it is essential to carefully define the area/zone/volume that is to be sampled and to understand what is being sampled. This can be particularly important when comparisons are to be made with published assessment criteria requiring consideration of, for example, upper and lower confidence limits (see 7.3.4).

When composite sampling is employed, taking a number of independent samples and averaging the results can reduce uncertainty in the estimated mean value and enable estimation of variability.

If average concentrations and variability parameters are to be derived from sampling used to determine spatial distributions, it is essential to define very carefully the volume of soil for which the average is being calculated. While an average is always a valid parameter, its practical and theoretical usefulness will be limited if, for example it includes different soil types, sampling horizons, or zones with different contamination histories.

NOTE Further guidance on determination of average properties and associated variability is provided in 7.3, A.5.4, and Annexes E, F and H.

6.4 Types of samples

6.4.1 General

NOTE This subclause is mainly informative. Guidance on the choice of sample type(s) for particular applications is provided in 8.2 and 9.2.

6.4.2 Disturbed and undisturbed samples

Two basic types of samples are collected for the purposes of investigating soil and ground conditions:

— disturbed samples (ISO 11074:2015, 4.4.8), and

— undisturbed samples (ISO 11074:2015, 4.4.31).

Disturbed samples are suitable for most purposes, except for some physical measurements, analysis for volatile substances, and microbiological examinations for which undisturbed samples could be required. Disturbed samples may be taken as single spot samples (6.4.3), cluster samples (6.4.4) or as spatial composite samples (6.4.5) where this is appropriate for the objectives of the investigation.

Undisturbed samples are inherently spot samples, i.e. taken from a specific material at a specific location and depth, although the initial undisturbed field sample might sometimes be taken over a depth range or be of extended lateral extent (e.g. when a core is taken for later examination and subsampling in the laboratory).

6.4.3 Spot (single) samples

Spot samples are taken as a single increment or formed from a limited number of increments of material that lies in direct contact. If there is a space left un-sampled between the increments, the result will be a cluster sample (6.4.4), which is not a true spot sample.

Spot samples are required in cases when information about the distribution of a substance or other property over a defined area or with depth is required. They are essential for estimating the degree of heterogeneity of a population or to detect limits of anomalies. Information resulting from spot (single) samples will be quite precise in general. To characterize a population, a large number of spot samples might be necessary.

Judgemental sampling around known or suspected sources of contamination, hot spots, or other anomalous areas, also requires single samples.

6.4.4 Cluster samples

Cluster samples made up of a small number of increments are appropriate when sampling top-soils for contamination (a number of small samples are taken over a defined area such as 1 m^2) or when using machines for excavating ground to obtain samples. In these latter circumstances, the samples should be formed by taking portions from locations within the bucket of excavated material. The use of cluster samples can reduce sampling uncertainties. This type of composite sampling is particularly appropriate when using trial pits, when surface samples (e.g. at depths of 0,0 m to 0,15 m) are being taken and when carrying out validation sampling of imported topsoil.

6.4.5 Spatial (composite) samples

6.4.5.1 Characteristics

Depending on the nature of the investigation, the defined sampling strategy and the objectives of the investigation, the use of spatial composite samples may be considered. In some situations the use of composite samples could result in dilution or loss of components, and hence the risk of not detecting contamination. However, composite samples can enhance the representativeness of the samples and may be considered in a number of specific situations. Composite samples may be produced in the field by combining increments from not identical locations or spot samples or subsamples of spot samples (in general the larger the number of increments, the more reliable the average result will be in case of more homogenously distributed properties). Alternatively, individual samples can be combined and thoroughly mixed in the laboratory.

Spatial composite samples are typically obtained by collecting a number of equally spaced increments of the same size, following a prescribed pattern, over a sampling area. These samples are bulked together to form the composite sample. This sample represents the mean quality of the area sampled. Composite sampling is often used where a sample is required to evaluate soil quality for agricultural purposes or for waste characterization of a stockpile. They can sometimes be used when investigating contaminated sites but only under very carefully defined conditions (see 6.4.5.3, Note 2).

More specifically, the use of composite samples may be considered where:

— there is homogeneous distribution;

— there is heterogeneous distribution over a small distance, but over a large distance the distribution is homogeneous;

— the components are non-volatile or semi-volatile.

Composite samples deliver, per se, averaged information with no information on variability, or sampling uncertainty (unless the composite sampling is replicated).

The use of composite samples could be of assistance in situations where there could be a large degree of variation of composition over a short-scale, but where on a larger-scale the distribution might be

considered to be homogeneous, for example, where ash or slag is mixed with soil. In this instance, composite samples might give much more representative analytical results.

Whether the composite samples are formed in the field or in the laboratory, only increments from within the same soil stratum should be combined.

NOTE 1 Combining samples/increments that come from different soil layers would result in a loss of information, and only combining neighbouring samples/increments will also ensure that any large-scale spatial variation in concentrations in the plane of the soil layers will be detected.

NOTE 2 There are various forms of composite sampling, mainly characterized by the size of the area from which the incremental samples that make up the composite are taken. This could vary, for example, between a few square metres and thousands of square metres. Cluster samples (see 6.4.4) are taken over a very small area (e.g. 1 m^2) and are used as an alternative to spot samples.

6.4.5.2 Advantages, disadvantages, limitations

Composite samples are often used to reduce the number of analyses required.

Information resulting from composite samples can neither reflect extreme values nor local variations or gradients. If the number of increments is too high, anomalies can become obscured.

6.4.5.3 Recommendations for application

Composite samples are typically used to characterize a population in total. But care should be taken if the results are intended to be considered representative. The increments also should be taken representatively according to a uniform approach and combined proportionally. Convenience or judgemental subsamples will not result in a representative composite sample.

Composite samples are not appropriate in all cases and the property of interest will determine, to a large degree, the possibility of forming composite samples. Composite samples cannot be made without significant loss of volatile compounds. Where semi-volatile compounds are present, composite soil samples may be prepared in the laboratory. The appropriate pre-treatment of the composite samples (e.g. thorough mixing) is of vital importance to ensure that representative results are obtained.

While the combination of a number of individual samples into a “composite sample” for analysis can increase the chance that widespread contamination will be detected, it is also possible that the mixing process will dilute a hot spot concentration to below detection and hence give a false indication in relation to contamination of the site. Any values used to judge the presence of contamination should be revised downwards to allow for this dilution effect.

Care should also be taken to avoid the combination of increments from different populations, subpopulations or soil strata.

NOTE 1 If, when taking an incremental sample out of a sampling device (e.g. a probe or an auger), this is done conveniently by selecting the loose material, the results could be heavily biased and will not be representative.

NOTE 2 Views differ as to whether spatial composite samples are suitable for the investigation of contaminated sites. In some countries, contaminated sites are usually only investigated using spot samples or cluster samples. However, in other jurisdictions (e.g. the Netherlands, see Reference [28] and in some circumstances in the USA, see Reference [29]), a form of composite sampling is either required by regulations or advised in formal guidance, especially when contamination levels are being compared with guideline values.

NOTE 3 Composite sampling can be unsuitable for the investigation of potentially contaminated land because of the:

— difficulty of comparing resultant data with guideline concentrations that relate to spot samples;

— possibility of disguising isolated locations of high concentration by mixing with samples of lower concentration;

— possibility of loss of volatile compounds during the compositing processes;

— difficulty of achieving an adequately mixed and representative sample;

— difficulty undertaking statistical analysis of composited data.

NOTE 4 Examples of the application of composite samples for sampling stockpiles for a number of different purposes is provided in Annexes E and F. The principles outlined are also applicable to *in situ* volumes of soil.

NOTE 5 The ability to form reliable composite samples depends upon the soil type, for example, it might not be possible to form composite samples from a clayey soil.

6.4.6 Selective samples

Selective samples are prepared by selecting materials on the basis of particle sizes, colour or some other readily discernible property. Two methods for selective subsampling, which can be applied in the field or laboratory, are described in ISO 18400-201:

— sieving (this should not be carried out when VOCs requiring analysis are present);

— hand picking.

Both methods can be applied, independent of whether the interest is in the fraction with the smaller or the larger particles. Hand picking can also be applied when other distinctive features of the material are a basis for the selection (e.g. colour, type of material).

Sieving is preferred when the mass ratio between the fraction with the larger particles and the fraction with the smaller particles of interest is small but is only possible when the material is suitably dry.

NOTE 1 With a small mass ratio between the two fractions, the amount of work for handpicking will be larger and the probability of missing particles will be higher.

NOTE 2 Selective subsampling per definition will result in a highly biased subsample and can only be allowed when indeed such a subsample is sought. This can for example be the case when there is an expected significant difference between the larger and smaller particles or elements in the sampled material, or, when apart from soil material, there are also bricks or other man-made materials present in the sampled material, that it has been decided need not be analysed or need to be analysed separately.

6.5 Number of samples

6.5.1 General

NOTE See also Clause 7, 8.6 and 9.6.

The total number of samples required in any sampling exercise will depend on a number of factors. In the case of an in-ground investigation in which spot or cluster samples are to be taken it will depend on the number of sampling locations (see below); the depths from which samples are taken at each location (8.4, 9.4); and whether one or more sample is taken from each sampling point (6.5.2).

When composite sampling is to be carried out, the total number of samples will depend on the number of zones into which the site is divided (7.3.2) and whether one or more sample is taken from each zone.

The number of sampling locations required when systematic sampling is employed will depend on sampling pattern used and the spacing between sampling locations (sampling density). As described in Clause 7 there are variety of factors governing the density of sampling required (e.g. a need to find a hot-spot of a particular size if it exists) and the total number of samples required to enable a statistically sound estimate, for example, of average properties (see also 6.3). Whether geostatistics are to be used will also have an influence (see 5.7 and Annex H).

Clause 7 also provides guidance on a number of situations when sampling is required for particular purposes [e.g. classification of materials for disposal as waste (7.5.2)].

When judgemental sampling is employed, the number of sampling locations required will, by definition, be a matter of expert judgement but important factors include:

— the number and size of features to be targeted,

— the need (or otherwise trying) to delineate a feature (as noted in 6.6.2, this can be aided by using a localized sampling pattern),

— the need to have information on non-target areas.

NOTE Illustrative examples of the numbers of samples required under particular circumstances can be found in Annexes D, E, H and I.

6.5.2 Number of samples at discrete sampling points

It might be necessary to take more than one sample from a discrete sampling point to:

— aid estimation of measurement uncertainty, and its sources (see 5.8 and Annex C);

— provide samples of more than one type to satisfy testing requirements (e.g. disturbed and undisturbed samples);

— provide samples of different size for test purposes;

— provide samples for different analytical and test regimes as required by the laboratories (see Note 1);

— provide duplicate samples for quality assurance purposes (see ISO 18400-106).

The practicality of providing near-equivalent samples will be constrained by the technique used to obtain them (see ISO 18400-102), the particle sizes (maximum and distribution), and the nature of the soil (e.g. clayey, granular, organic-rich).

NOTE 1 Often, laboratories carrying out the chemical analyses will require more than one sample from a location with those for different tests being of an appropriate size and placed in an appropriate container (e.g. plastic tub, glass jar, vial) rather than a single large sample that can be sub-divided or subsampled in the laboratory.

NOTE 2 There are obvious practical difficulties obtaining several equivalent samples, for example, when the material to be sampled is in the form of a 50 mm diameter core.

NOTE 3 Duplicate samples can be taken a small distance apart to show the effect of small-scale heterogeneity (see Annex C).

6.5.3 Number of cluster samples

It can be desirable to take more than one cluster sample from a proportion (e.g. 10 %) of the sampling points within an investigation, in order to estimate the uncertainty in the measurement results (see Annex C).

6.5.4 Number of composite samples

It can be desirable to take more than one composite sample from the area, zone or above-ground deposit under investigation in order to reduce uncertainty in the analytical and other results and/or to split the area, zone or deposit to be investigated into a number of sub-zones (see Annexes C and G). The latter will aid identification of any variation of average properties across the area, zone or deposit that might need further more detailed investigation.

NOTE 1 See also Clause 7.

NOTE 2 Examples of the application of composite sampling including the choice of the number of samples to take are provided in Annexes E and G.

6.6 Sample sizes

6.6.1 General

A number of factors govern the size of the samples sent to off-site laboratories including:

a) the range of pedological, chemical, physical and or biological examinations and tests that are to be carried out;

b) the specific requirements of the laboratory/laboratories carrying out the examinations and tests; and

c) the need for samples to be sufficiently representative (i.e. acceptable level of sampling uncertainty), taking into account the particle size distribution and the concentration distribution of the material to be sampled.

NOTE 1 More samples than required for the testing suite envisaged are usually taken, in case additional analysis or tests are decided on, in response to site observations and initial results.

Points a) and b) will usually be defined by following an established procedure and/or discussion with the laboratory/laboratories.

Point c) requires that

1) the sampling equipment enables the largest particles to be included in the samples or increments, and

2) the sample size is large enough for fundamental variability to be acceptably small.

The degree to which it is necessary to take account of the increment and sample size will very much depend on the type of material sampled.

The relationship between particle size distribution and the size of sample or increment required to obtain reliable analytical results is amenable to theoretical determination for a granular material in which the properties of interest are inherent within the particles. The guidance provided in Annex F is applicable when the property of concern is associated only with the dry particulate matter in the material being sampled. It is not applicable if the substance of interest is present as a non-aqueous phase or is dissolved in the non-aqueous phase. It is also not applicable when dealing with an inherently complex material such as soil which might contain solid particles of several types, water, biota, contaminants adhering to particles and even non-aqueous phase liquids.

In these more complex cases, the person designing the sampling exercise should, in consultation with the laboratory undertaking the planned testing, make a judgement about the minimum sample size required (see 6.6.2). Note, however, that such a specification will usually be based on assumptions about the character of the material to be sampled. The sampler should adjust the sample size to take account of observations in the field (or the results of an earlier Exploratory Investigation, including information about excessive sampling uncertainty).

In general:

— the smaller the sample taken, the more difficult it will be to ensure that the sample is sufficiently representative;

— when the sample to be sent to the laboratory is composed of a number of increments (i.e. is a composite sample), each increment should be of sufficient size to be representative of the material at the particular location from which it was taken;

— where the sample is of coarse-grained material, for example, gravels, a larger sample is required (see Note 1 to 6.6.2 for grain size definitions);

— when the contaminant or characteristic of interest is present only in a small proportion of the particles, a larger sample is required;

— the greater the range of particle sizes, the more difficult it can be to obtain a sufficiently representative sample, because of the tendency for segregation of different particle sizes during sampling and subsequent handling;

— if a larger volume is taken as a spot sample which then is homogenized and reduced in volume the investigation result will achieve a more average character — there is a transition to cluster sampling from the informational point of view.

While it is important that the sample taken from the ground is sufficiently representative, it is also important that the fraction of the sample actually analysed or tested is that which is of concern in relation to the objective of the investigation. For example, in human health risk assessment it will be the finest fractions that are of concern as only these are likely to be ingested, inhaled, or adhere to the skin. Segregation of the fractions of interest is best done in the laboratory but a degree of selectivity in the field can sometimes be appropriate (i.e. a "selective sample" might be taken by design). Some sampling techniques can only provide samples of restricted size and/or are not good at capturing larger particle sizes. Notwithstanding the desirability for samples sent to the laboratory to be truly representative, the persons designing and carrying out investigations, might need to decide that the samples that can be practically obtained are "fit for purpose" or at least "good enough" in the context of the investigation and/or that any deficiencies in the quality of the sample will not compromise the decisions that will in due course have to be made (e.g. sampling uncertainty is at a level that is "fit for purpose", see Reference [30]).

Even when a theoretical determination of minimum increment and sample size is available, it does not consider all components of sampling uncertainty (e.g. the uncertainty due to pretreatment in the field or in the laboratory, and the analytical uncertainty). In any case sample duplicates should be taken to enable estimation of the total sampling uncertainty resulting from the increment and sample sizes actually used and in order to decide whether they are fit-for-purpose for the detailed investigation (see Annex C).

NOTE 2 Liquids: The minimum increment and sample size have no specific relevance to the sampling plan design for liquids as the potential differences are at a molecular scale when compared to the size of the samples. The sample size is, in practice, governed by the quantity of material required by the laboratory for analysis, whereas the increment size is determined largely by the dimensions of the sampling equipment.

NOTE 3 Powders and sludges: Powders and sludges are basically particulate materials with a (very) small particle size; sludges also contain a substantial amount of liquid.

6.6.2 Practical considerations

The minimum sample size required in any particular case might be:

— the theoretical minimum to encompass expected variability at the scale of interest (see 6.6.1 and Annex F); or

— as dictated by other considerations (see 6.6.1 and below), whichever is the greater.

A minimum sample size of 500 g to 1 000 g will often be required (this applies both to single samples and composite samples, in the latter case after sufficient homogenization). The sampler should adjust the sample size to take account of observations in the field (or the results of an earlier Exploratory Investigation, including information about excessive sampling uncertainty). When spatial composite (or cluster) samples are to be prepared:

— the minimum increment should be of sufficient size to yield the required minimum sample size when the increments are combined; if this is not the case, either the size of the individual increments should be increased or the number of increments increased (or a combination of the two);

— the individual increments or samples to be combined should be of equal size (±25 %, mass fraction);

— the number of increments in each composite sample should be specified in the sampling plan; in most cases, the number of increments should be the same for all composite samples within the sampling programme — this simplifies statistical analysis.

For practical reasons, the minimum size for an increment taken in the field should be 200 g.

The sampling equipment being used, and the depth of layers of materials of different character, will influence the size of sample that can be taken. For example if a 50 mm core is taken, and the layer of interest is only 100 mm thick, all that material is likely to be required to form a sample, the mass of which might be less than otherwise considered desirable. This can be considered acceptable if this situation is not common. Otherwise cores with a larger diameter should be used.

Often the size of sampling equipment is such that more soil material is obtained than needed. Sample pretreatment is then required "in the field" in order to reduce the amount of material to be transported to the laboratory, while keeping it sufficiently representative. Guidance on pretreatment in the field is provided in ISO 18400-201.

In addition to the above, the following should be taken into account when deciding on the practical sample size(s) required:

— **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.

— **Boulders and other large material:** some soils (partly) contain (very) large boulders and other large material (see Note and Table 3 for named size classifications). If these 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 larger materials 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. It should be noted, to what extent boulders and other large materials contribute to the total volume of the material to be sampled.

— **Small particle sizes:** for small particle sizes, the minimum size of the sample can be very small, which is particularly relevant for subsampling in the laboratory in order to obtain the analytical sample.

— **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 samples are needed, because of the large particle size, the particle size should be reduced following the procedures described in ISO 18400-201 and ISO 11464 as appropriate. As particle size reduction is (in principle) only allowed under laboratory conditions, for these situations either a mobile laboratory or on-site laboratory is needed.

— **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 ensured. To ensure 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 (see ISO 18400-201).

— **Compounds to be analysed/tested**: the compounds to be analysed in the (sub)sample(s), or the test to be carried out, will in some cases affect the possibilities or methods of subsampling [e.g. a requirement to determine volatile organic compounds (VOCs) precludes oven drying, size reduction and sample splitting — subsamples are taken from the "as received" sample].

— **Laboratory provided containers**: analytical laboratories often provide the containers they want to use. For example: 400 ml to 600 ml plastic tubs for material to be dried and pretreated in the laboratory prior to analysis, for example for heavy metals, etc.: 250 ml glass jars for materials on which semi-volatile organic compounds (SVOCs) are to be determined without drying or other pretreatment and 70 ml glass jars for materials on which volatile organic compounds (VOCs) are to be determined without drying or other pretreatment. The assumption is that the material in each container is representative of the same portion of material in the field which is only likely to be true within limits. For example, the diameter of a 70 ml glass jar is only about 40 mm so that it is probable that coarser material will unavoidably excluded from the sample. The mass of fine dried

sand that can be held by a 70 ml glass is about 100 g (i.e. a bulk density of about 1,4 g/ml) which is less than the 500 g minimum recommended above.

NOTE ISO 14688-1 establishes the basic principles for the identification and classification of soils on the basis of those material and mass characteristics most commonly used for soils for engineering purposes. ISO 14688-1 is applicable to natural soils *in situ*, similar man-made materials *in situ* and soils redeposited by man. Named particle groups are defined in Table 3.

Table 3 — Defined particle size ranges

Name	Size mm	Name	Size mm
Large boulder	>630	Coarse sand	0,63 – 2,0
Boulder	200 – 630	Medium sand	0,2 – 0,63
Cobble	63 – 200	Fine sand	0,063 – 0,2
Coarse gravel	20 – 63	Coarse silt	0,02 – 0,063
Medium gravel	6,3 – 20	Medium silt	0,006 3 – 0,02
Fine gravel	2,0 – 6,3	Fine silt	0,002 – 0,006 3

7 Deciding how many samples to take

7.1 General

The number of samples that should be taken in any particular situation, whether these are to be spot samples, cluster samples or spatial composite samples should take into account:

— the purpose of the sampling exercise;

— the sampling objectives;

— prior knowledge or assumptions about the heterogeneity of the population (e.g. volume of soil) to be sampled; and

— the confidence required in the results and conclusions to be drawn from the results.

In all cases:

— the volume(s) of soil of interest (the “population” or “sub-populations” in statistical terms) should be carefully defined;

— any prior knowledge should be taken into account.

NOTE Attention is given to any specific requirements, such as how to decide if a prescribed limit is exceeded or has not been reached.

It is rare that site investigations are carried out without any knowledge about the site in question, indeed the purpose of the preliminary investigation (4.3.2) is to acquire information that can be used to develop a preliminary conceptual site model (4.4) and the sampling plan.

Available information is used to aid decisions about what, where and how to sample, and what substances and properties should be measured. By staging investigations, knowledge about what is, or is not present (and often variability), can be established to inform the design of subsequent stages of investigation and to improve the efficiency of the overall investigation (e.g. by reducing the total number of investigation points required or to enhance the value of information gained from each sampling point).

It can sometimes be possible to adopt a Bayesian approach to the design of a sampling plan. However, this should only be done with care and on the basis of expert advice (see B.1.2).

7.2 Basic situations

There are three basic situations.

a) It is beneficial to know the average concentration and in some instances to know whether this is above or below a particular value (e.g. guideline value for human health risk). This might be required in respect of, for example, human health risk assessment, ecological risk assessment, waste disposal, pesticide residues, nutrient status, etc. The scale can be from a small hot spot (a few m^2), garden (say 25 m^2), field or larger area. Spatial composite samples (but how many?) or spot samples (e.g. taken randomly or on a regular grid) could be used. Guidance can be dimensionless (i.e. applied irrespective of the area of interest), but also needs to be concrete in terms of sites of a specified size. All this requires an understanding of variability.

   NOTE 1 Some examples of when it might be necessary to determine average concentrations are provided in Annex D. Annex E discusses the "scale of sampling" required both to determine average concentrations reliably and to understand variability within the target volume of soil. The examples in both Annexes refer to stockpiles but the same principles can be applied to determination of the quality of soil in a field or to a volume of *in situ* soil.

b) It is necessary to locate a hot spot (or hot spots) or other area of interest (e.g. on a development site, the one garden in a hundred planned gardens that might be above a guideline value). Having found the area of interest (e.g. one high result in a hundred) it becomes a case of how to delineate and characterize the hot spot or area of interest. Delineation requires spot sampling using an appropriate sampling pattern, although the average concentration within the hot spot or variation within the hot spot might be of interest, depending on the objectives of the investigation.

   NOTE 2 There might be role for judgemental/targeted sampling if the planned layout of the houses is known, e.g. by taking one or more samples from each proposed garden area.

c) It is necessary to say which, if any, of the potential hypotheses in ISO 18400-203:2018, Annex A is true, i.e.:

   — hypothesis of a "probably uncontaminated" site or zone;

   — hypothesis of a "probably contaminated" site;

   — hypotheses relating to spatial distribution of contamination:

      — contamination is present with a homogeneous distribution;

      — contamination present with a heterogeneous distribution with point sources of contamination of known location;

      — contamination present with a heterogeneous distribution with point sources of contamination of unknown location;

      — contamination present with heterogeneous distribution but no point sources of contamination.

   NOTE 3 ISO 18400-203:2018, Annex A presents these as hypotheses to be addressed in the sampling strategy, but that carries with it an implicit requirement to say once the investigation has been carried out whether the preferred hypothesis has been proven and if so with what confidence.

7.3 Determining average concentrations

7.3.1 General

Annex G provides statistical expressions for estimating the mean of a population, its standard deviation, a percentile at the scale of the samples, and a percentage compliance with a given limit, as well as methods for calculating the uncertainty associated with these estimates.

These methods are designed for uncorrelated data, that is, in practice, when the property of interest displays no significant spatial correlation and/or in the case of random sampling.

7.3.2 Using spatial composite samples

As described in Annex D and Annex E, spatial composite samples can be used in various ways to determine average properties, with or without also providing information on variability at various scales.

Considering the case of a field in which soil quality is to be determined, decisions should be made about:

— whether to divide the site into zones on the basis of some attribute (e.g. soil type, perceived plant health);

— whether to sub-divide the field or zones into sub-areas (subpopulations) for the purposes of sampling (the assumption is that soil in each sub-area will have similar characteristics although this might not prove to be the case);

— whether to take one or more composite samples from the field or zone (the population) or from each sub-area if the field or zone has been divided in this way ;

— how many increments should be taken to form each composite sample.

EXAMPLE An undivided area of pasture land occupying several hectares is to be sampled. Two zones are distinguished on the basis of known soil types.

— Each zone is sub-divided into three sub-areas.

— Three composite samples are taken from each sub-area.

— Each composite sample comprises twenty five increments.

NOTE 1 As indicated in B.2, research has shown that a single composite sample prepared by combining at least 25 increments will yield a reliable estimate of average properties.

NOTE 2 The examples in Annexes E and G refer to composite samples made from 11 to 50 increments.

A further reason for sub-dividing a field or other site is that it is simply too large for it to be considered reasonable that a single (or even a few) composite sample taken across the whole area could properly portray the soil quality for the whole area. In such circumstances the site should be divided into a number of zones (preferably of similar size) as shown in Table 4. A separate decision should then be taken as to how many composite samples to take from each zone.

Table 4 — Number of zones for composite sampling in relation to the total area of the site

Area A ha	Minimum number of zones n
0 to 2	1
>2 to 5	2
>5 to 10	3
>10 to 15	4
>15 to 20	5
>20 to 30	6
For areas larger than those given, the following formula should be used to specify the number of zones to be sampled: $n = 1 + \sqrt{A}$. The underlying assumption is that properties are generally uniform within the area to be investigated – if this is not the case, the number of zones should be increased.	

7.3.3 Using spot samples

Average properties can be determined with reasonable confidence by taking the mean of values determined on spots samples taken using a probabilistic pattern provided sufficient number of samples are taken to encompass the full variability of the properties. If insufficient samples are taken from a heterogeneous population, there could be considerable bias in the average determined (see Annex I).

Ideally 20 to 30 samples should be taken from the exposure area/volume to provide a good estimate of the mean concentration (i.e. the 95 % confidence limits are close to the mean) while fewer than 10 samples will provide only a poor estimate (i.e. there is a large difference between the sample mean and the 95 % confidence limits), see I.7.2.

NOTE 1 Guidance on the calculation of mean values and associated statistical parameters (e.g. confidence limits) is provided in A.5.4 and in Annex G.

NOTE 2 The different levels of information on variability that can obtained when spot samples rather than composite samples are taken are illustrated in E.3.

7.3.4 Determining relation to threshold limit

Depending on the circumstances, it might be important to know whether the mean concentration of a substance of interest (e.g. nutrient, potentially hazardous substance), within an area is above or below a defined value.

If the calculated mean determined from a number of composite samples, spot or cluster samples, was the true mean it would be simple to say whether its value was above or below a limit value. However, the calculated mean is only an estimate of the true mean which, depending on the variance of the data, can only be said to probably lie, within a range of values defined by the upper and lower confidence limits. It is usual to work with 95 % confidence limits but there might be occasions when a lower level of confidence is required. Guidance on how to calculate confidence limits is provided in A.5.4.

— If the question to be answered is whether the mean concentration is below a certain threshold value, then the 95 % upper confidence limit of the calculated mean should be below the threshold value.

— If the question to be answered is whether the mean concentration is above a certain threshold value, then the 95 % lower confidence limit of the calculated mean concentration should be above the threshold value.

The magnitude of the confidence interval will depend on the variance of the data – in general, the more samples that are taken the lower this will be and the closer the calculated mean will be to the true mean.

For reliance on this approach, the data should be fully representative of the range of values present in the population. The fewer samples taken, the less likely is this to be the case, and the more likely that the calculated mean will be biased by inclusion of one or more high values or the absence of high values because by chance none of the samples analysed were at the upper or lower end of the actual range of values present (see 7.3.3 and I.7.2).

There can be circumstances where the interest is whether the results for individual samples are above or below a limit value (e.g. for waste disposal purposes). In these circumstances analytical variance will be important. It might be that guidance permits some “failures” within a set of values subject to the number of samples and certain statistical tests being met, see for example Reference [31].

7.4 Finding (hot spots and) areas of interest of a specified minimum size

Annex I provides information on the relationship between the size of an “area of interest” (e.g. a garden of specified size) and the probability of locating it in relation to a regular grid of specified dimensions.

As shown in Table 5, the information in Annex I indicates that in order to have 90 % confidence of finding an area of interest of 25 m^2, a grid size of 5 m is required and that the probability of finding an area of that size diminishes rapidly as the grid size is expanded so that it is only 25 % for a 10 m grid.

Table 5 — Effect of increasing grid spacing on the probability of detection

Grid size	Size of grid square	Nominal size of smallest area of concern	Relative hot spot area	Probability of detection
m	m^2	m^2	SAC/GS	%
5	25	25	1,00	90
10	100	25	0,25	25
15	225	25	0,11	10
20	400	25	0,06	6
30	900	25	0,03	3

7.5 Sampling for particular purposes

7.5.1 Determination of background values

Guidance on sampling to determine background values on a regional basis is provided in ISO 19258. This standard includes guidance on sampling numbers and statistical evaluation of the data collected.

7.5.2 Waste classification

Classification of soil and soil materials for waste disposal purposes is usually done in two stages:

a) decide whether the material is “hazardous”;

b) collect information relevant to disposal or treatment.

NOTE 1 Classification of whether a material is “hazardous” is usually done by reference to national or international requirements as are the information requirements for disposal or treatment. These are not things that can usually be done from first principles without reference to the regulatory context in which judgements are being made.

In the case of materials to be sent to landfill, information is likely to be required on the composition of the solid (e.g. the concentrations of polycyclic aromatic hydrocarbons (PAHs) and on leachability).

The information required relates to the average properties of the material in question in the form in which it is to be disposed off but with an understanding of the variability. This means, for example, that while analyses and leaching tests will typically be carried out on the <10 mm fraction, interpretation, in relation to whether limits have or have not been exceeded, can be made in respect of the bulk material, i.e. including >10 mm material and an appropriate amount of moisture.

It is important that unintentional bias is not introduced into the results. While it might be appropriate to take a sample from a small patch of soil contaminated with hydrocarbons in order to find out what it is, it could be inappropriate to include the analytical result perhaps representing only 1 m^3 of material, in a set of results in which each sample is taken to represent say 600 m^3. It could introduce significant bias to the results causing the average concentration to exceed the relevant limit when the correct approach would be to take a sample from more typical material and to delineate the volume affected by the hydrocarbons so that it can be removed and dealt with separately.

NOTE 2 Guidance on the sampling of wastes in general is provided in ISO 14899, and CEN/TR 15310 series. Reference [31] provides guidance in relation to soils in particular.

7.5.3 Supplementary investigations for remediation

Following completion of a site investigation of a contaminated site, decisions could be required about whether remediation is required. This might take the form, for example, of excavation of soil material for waste disposal or for *ex-situ* process-based treatment, or *insitu* treatment. In order to implement remediation, it is often necessary to carry out supplementary investigation (4.3.5).

If supplementary investigation is to be carried out in support of remediation decisions should be made about:

— scale;

— what to analyse and/or test for;

— what fraction of the soil to analyse or test.

The answers to these questions, particularly the first, should take into account both technical and economic factors. More data might enable the costs of remediation to be reduced but at some point, the additional costs will outweigh the savings. In contrast, more data might be essential to enable a process-based treatment method to be operated effectively and efficiently.

If excavation is to be carried out for disposal or to provide the feed to a process-based method of remediation, key questions that should be addressed include:

— the size of the area or volume that it is "safe" to leave in place;

— the practical volume for assessing materials for excavation and/or *ex-situ* treatment;

— the cost-benefit relationship between the scale of sampling and the cost of treatment or disposal;

— how to demonstrate conformance with any limiting values and the scale at which this needs to be done, e.g. for technical or regulatory reasons.

NOTE D.4 and E.2.3 provide useful illustration of how supplementary information might be obtained. Although it refers to already excavated and stockpiled soil material, the principles outlined are equally applicable to *in situ* soils. Reference [32] describes a case involving contamination with dioxins where small scale composite sampling led to significant savings in both analytical and remediation costs. Annex C and Annex G discuss some aspects of the cost-benefit equation relating sampling costs to project costs.

7.5.4 Validation of remediation and other works

During and following remediation, it is often necessary to carry out further sampling and other types of investigation to demonstrate that the works have been properly carried out (e.g. a specified volume of soil has been removed) or are working as planned (e.g. in the case of an *in situ* treatment method) and/or that the site is now "fit for purpose" as intended.

Clear objectives should be set for verification studies and they should be designed taking into account the guidance in this document on the scale of sampling and appropriate statistical approaches.

NOTE 1 Examples of where verification might be required are:

— removal of a "hot spot" of contamination including confirmation that the specified volume (area and depth) of material have been removed and that the sides and base of the excavation reveal the presence only of contamination below a predefined limit (remediation target value);

— covering of the site to a specified depth with "clean" soil including confirmation that the depth of cover is at least the minimum specified and that the physical, chemical and other attributes of the soil are as specified.

When seeking to demonstrate that a site is now fit for purpose the consideration should be site wide: it is not just a case of collecting together all the reports on localized remediation activities. Consideration should be given to the need to carry out systematic regular sampling across the whole of the site embracing both remediated and non-remediated areas and ignoring where the boundaries between these are assumed to be in order to provide a coherent set of data for the remediated site (this is what a prudent potential purchaser might in any case do to ensure themselves that what they are being told about the condition of the site is true).

NOTE 2 Reference [33] provides useful guidance on the verification of remedial works.

8 Sampling strategies for in-ground sampling

8.1 Approach to sampling

A decision should be made as to which of the following principal approaches should be employed:

— judgemental sampling, based on already available information and expert judgement (although this can provide valuable results at reasonable efforts, these results cannot be considered as representative in a statistical sense [see 6.2.2)];

— systematic sampling (see 6.2.3), based on statistical considerations which requires predefined sampling locations.

A combination of judgemental and systematic sampling should usually be employed when investigating potentially contaminated sites (see ISO 18400-203).

Investigations where the intention is to determine average properties for sites which are believed to be uncontaminated will usually require only systematic sampling or spatial composite sampling depending on the circumstances (see Clause 7).

NOTE In some cases, the power of judgemental sampling can be increased by employing an appropriate localized sampling pattern that takes into account the assumed shape of the target area (e.g. the area affected by a plume of hydrocarbons leaking from an underground storage tank).

8.2 Sampling patterns

8.2.1 General

When systematic sampling is to be carried out, an appropriate sampling pattern should be employed following the guidance given in 6.2.3 and Annex B, aided by Figure 5.

All fixed patterns should be adjusted to local conditions and modified as appropriate.

Sampling patterns are based on the probable distribution of soil constituents (in most cases chemical substances) within an area or on a type-of-substance input.

Four major fixed sampling patterns can be identified:

— patterns based on no specific estimate of substance distribution;

— patterns based on local substance distribution and known as a “hot spot”;

— patterns based on distributions along a line;

— patterns based on strip-like distributions.

In addition to these, several other patterns exist (e.g. based on deposition of substances from the air, input due to flooding).

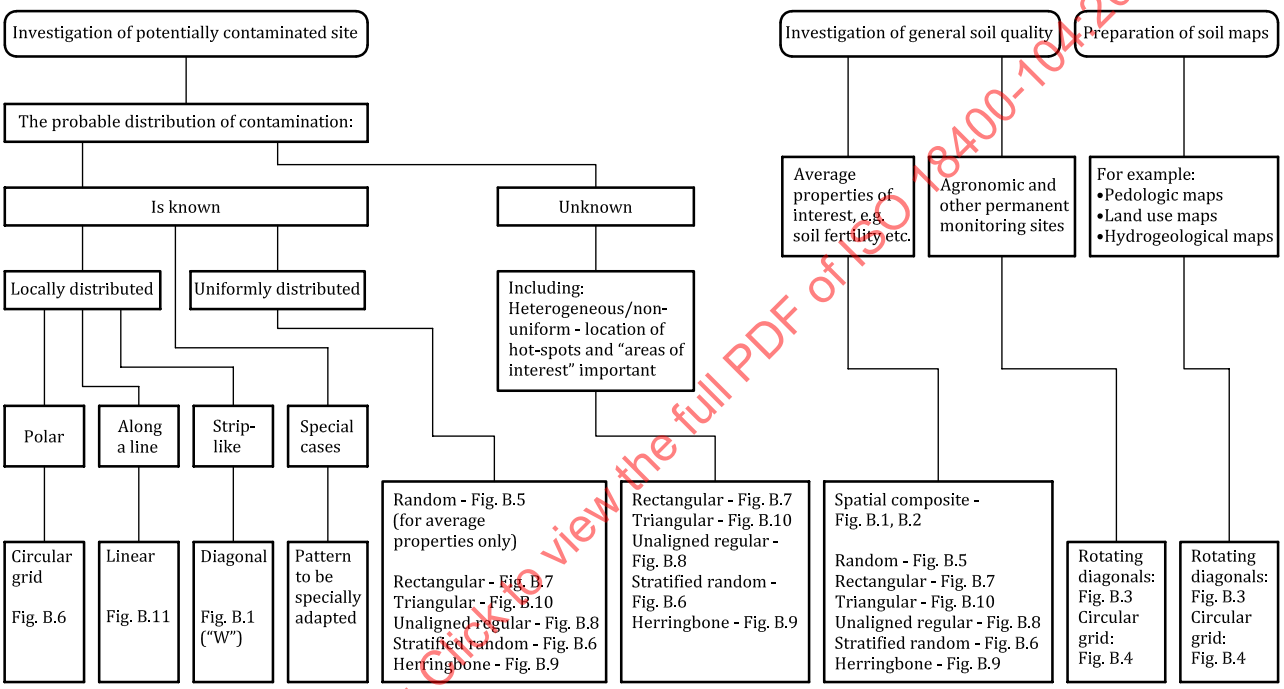
In agricultural sampling, one of the established convenient sampling patterns described in Annex B (see Figures B.1, B.2, B.3) should usually be used to obtain information, for example, on nutrient demand or the concentrations of pesticide residues across a wide area.

NOTE 1 Most predefined sampling patterns, especially grid patterns, are not very practical during the growing season, and are rarely applicable.

When sampling potentially contaminated sites, patterns should be employed that enable reliable determination of the spatial distribution of soil attributes including the concentrations of potentially hazardous substances.

NOTE 2 Annex B gives examples of a number of commonly applied sampling patterns which meet different statistical requirements (see Figures B.5 to B.10). Experience (and theoretical considerations) shows that, in many cases, systematic sampling on a regular rectangular grid is both practical and allows a sufficiently detailed picture of variations in soil properties to be established. The number of sampling points can be easily increased (e.g. in areas meriting more detailed investigation), the grid is easy to mark out on site, and sampling points are usually easily relocated.

Systematic sampling should be supplemented by judgemental and convenience sampling when appropriate.



NOTE The modification of patterns depends on: special situation on the site, e.g. rapidly changing topography, objective/hypothesis, applicability and validity of preliminary.

Figure 5 — Selection of sampling patterns

8.2.2 Potentially contaminated sites

Site investigations (both exploratory and detailed) should usually be carried out using systematic regular sampling so that the sampling locations are distributed throughout the site (or zone) according to a regular pattern. However, other sampling approaches (in particular, judgemental sampling) could be acceptable where there are good reasons for such use (for example, where it is important to check potential preferential pathways for migration), or to complement a systematic sampling pattern. Convenience sampling can be helpful for the characterization of small "target areas" identified on site as being atypical, for example differing in appearance (see 6.2.4).

The reasons for selecting a systematic regular sampling pattern are:

— sampling locations on a regular pattern are simpler to establish in the field;

— identification of areas of contamination including "hot spots" and design of further investigations are much easier.

Reliability of interpolation will depend substantially on the variability of soil characteristics. In many situations, including well stratified sediments, vertical variations in concentration could be much greater than horizontal variations.

If there are any regular topographical patterns on the site (for example, ditches at regular intervals, systematic undulations of the terrain, etc.), the sampling pattern should not coincide with the topography in a way that could introduce a bias or systematic error in the samples. This can be avoided by careful selection of the base or starting point of the sampling grid and, where necessary, careful selection of the spacing of the grid.

The sampling pattern used, and the number of the sample locations, will vary and be dependent on the hypotheses formulated and the phase of the investigation.

— In an exploratory site investigation, fewer samples will be collected from fewer locations than in detailed site investigation. For exploratory site investigation, sampling locations should be selected with the aim of confirming the hypotheses and establishing the areas that will require greater attention in the detailed site investigation.

— In contrast, the principal aim of the detailed site investigation will be to establish a comprehensive picture of the contamination status of all parts of the site. The number of samples collected and the spacing between sample locations should be related to the objectives and degree of confidence required in the resultant assessment of contamination and associated risk assessment and need for remedial works.

— Greater density of sampling will usually be appropriate in areas of suspected contamination with less intense sampling in areas not suspected of contamination, in accordance with the hypotheses relating to different areas of the site.

NOTE Under some circumstances, there can be greater monetary value in demonstrating, with a high degree of confidence, that a particular part of the site is "uncontaminated". This will usually require a high sampling density.

8.2.3 Hot spot detection, site investigation design and sampling

The "efficiency" of a sampling pattern is often expressed in terms of the confidence there is, in whether a "hot spot" of a given size will, or will not, be identified (see 7.4 and Annex I). However, the concept and definition of the hot spots should also be carefully considered during the design stage of the investigation, and particularly so for the detailed site investigation.

A hot spot can be defined as:

— an area of contamination in an otherwise uncontaminated area;

— an area of much greater contamination in a site that is generally contaminated.

In practice, the probability of detecting a hot spot can be improved by the careful design of the exploratory site investigation and subsequent detailed site investigation.

If the contamination is expected to occur in known localized hot spots, each suspect location should be subject to detailed investigation.

NOTE 1 In some circumstances, e.g. in agriculture-related investigations, the area of interest (hot spot) might be characterized by an atypical deficiency (e.g. in nutrients) rather than higher concentrations of a determinant or might be characterized by variation in some other property (e.g. compaction).

NOTE 2 The size of a hot spot is not a fixed parameter and depends on

— the source and nature of the contaminative process (for example, a hot spot of contamination due to possibly leaking buried drums will present a different sampling problem than that for identifying a hot spot due to a leakage from an identified storage tank), and

— the definition of what concentration of a particular contaminant is judged to be noteworthy when assessing the results of the investigation.

In the case of a substance potentially harmful to human health, the size of a hot spot or area to be detected will be related to the consideration of the maximum area that will not pose unacceptable health risks, if it is not detected in the site investigation and subsequent assessment of results. In this respect, it is important to bear in mind the area of contamination that could be of concern where the evaluation of human health risks is involved (see also 8.4 and Annex I).

EXAMPLE In the case of a housing development, this could require the identification of a contaminated area the size of a small garden, or even part of a small garden. This could be only 50 m^2 (or just 0,5 % of a 1 ha site).

NOTE 3 In the case of circular hot spots, location can be achieved with a square grid where the square diagonals are equal to or smaller than the diameter of the smallest hot spots to be detected; a triangular grid would be more efficient see B.10.

8.3 Types of samples

The samples taken should be of appropriate type(s) to enable the objectives of the investigation to be achieved. Special consideration is required regarding:

— whether to take disturbed or undisturbed samples (6.4.3);

— whether to take spot samples (6.4.4);

— whether cluster samples should be taken (6.4.5);

— whether to employ a form of composite sampling (6.4.5);

— how to comply with any authoritative guidance relating to judging whether guideline values (assessment criteria) have been exceeded;

— statistical analysis of the data obtained;

— the expected distribution of contaminants or other substances of interest;

— whether and how to reduce uncertainty in the measurements in the investigation (5.8 and Annex C).

Undisturbed samples should be collected if it is intended to determine the presence and concentration of volatile organic compounds, since disturbance will result in loss of these compounds to the atmosphere (see ISO 18512).

Composite samples should not be used when soil characteristics that could change during the composition process, such as concentrations of volatile compounds, are to be determined. They also should not be used if peak concentrations of any substance or variations of soil characteristics are to be determined.

When composite sampling is used to determine the characteristics of *in situ* soil the sample should represent a single stratum.

When samples are taken from a surface (ground surface, surface of a stockpile or from the walls or bottom of a trial pit) the whole sample or increments can be taken directly.

When samples are taken at depth using boring or drilling techniques (see ISO 18400-102), the samples can be taken completely as one single sample or can be subsampled or sampled selectively.

Disturbed samples are suitable for most purposes, except for some physical measurements, profiles and microbiological examinations for which undisturbed samples are often required. Disturbed samples

may be taken as single spot samples, cluster samples or as composite samples where this is appropriate for the objectives of the investigation.

NOTE Undisturbed samples are inherently spot samples, i.e. taken from a specific material at a specific location and depth, although the initial undisturbed field sample can sometimes be taken over a depth range or be of extended lateral extent (e.g. when a core is taken for later examination and subsampled in the laboratory).

8.4 Sampling depths

8.4.1 General

No general recommendation can be given on the depths at which samples should be taken or on the final depths to which trial pits or boring/drilling should extend. This depends on the objectives and might be subject to change during a running programme. The investigation of soil for chemical characteristics can be divided into two general cases:

— the investigation of agricultural and similar near-natural sites, where information is required mostly on the top soil or ploughed horizon or arable zone but often over an extended area;

— the investigation of sites which are known or suspected to be contaminated, where information is required from deeper layers, sometimes to a depth of several tens of metres.

A mixture of both these cases is found in so-called "permanently monitored soil sites", which represent larger areas of homogeneous soil development and in most cases are established to monitor environmental effects on the complete profile over a long time-scale (see ISO 16133).

If a profile is to be sampled, care should be taken that every horizon/layer of interest is sampled and that different horizons/layers are not mixed. In general, contaminated sites should be sampled horizon by horizon.

If trial pits are used, it could be appropriate to sample from more than one side. Ideally, this should be done by taking cluster samples from multiple sides of the pit. However, due to safety considerations in deep pits, the only possibility is to sample from multiple sides of the pile of excavated material (the material from different depths can be segregated by placing it on separate heaps).

A depth-related sampling programme is based on a number of conventions, depending on the project. It is not as representative with regard to the soil as a horizon-related sampling programme can be. The mode of sampling from each depth should be carefully specified, e.g. the maximum depth range (usually not more than 0,1 m) and how horizontal variations are to be dealt with.

Mountainous regions or hilly areas with pronounced slopes require special consideration. For slopes of 10° and greater, vertical drilling lengths should be extended according to the cosine rule to maintain slope-parallel thicknesses of soil layers constant. The extension factor is 1/cos of slope. Without correction, for example, the error will be 2 % at a slope of 11,5°.

8.4.2 Potentially contaminated sites

Where a site has been identified as potentially contaminated, ideally those horizons of the soil strata which are expected to be contaminated should be sampled (sampling will often be concentrated on suspect areas in accordance with the hypotheses). In some situations, where the site is underlain by an impermeable stratum at fairly shallow depth, it will be possible to identify specific depths which should be sampled. However, it is often very difficult, prior to any intrusive investigation, to have a clear indication of where contamination might exist.

NOTE 1 The distributions of different contaminants on a site can vary with depth, because they have different origins and, even if they originate from the same source, they behave differently in the ground.

Sampling depths should reflect what is known about intentions for the use of the site (i.e. the receptors which might be at risk) and the probable pathways by which contaminants could enter the environment.

The following should be taken into account as appropriate:

— varying physical and chemical soil properties over depth, particularly where substantial thicknesses of made ground are present or where large differences are present in natural deposits;

— contamination sources (e.g. solid, leachable and gas- or vapour-generating materials, leaking pipe) that might be located at any depth in the soil profile;

— that the relevant depth in exposure terms can be at any level in the soil profile (e.g. in redevelopment projects, the final formation level could be lower than existing site level; close contact between soils and site services can be at some depth below ground level);

— movement of gases and liquids along vertical (and perhaps deep lateral) pathways will be determined by physical soil properties at the relevant depths;

— the usage of the site.

NOTE 2 Reasonable sampling depths can be geared to excavation depths needed for construction planned on the site. See the following examples.

EXAMPLE 1 On most housing developments, excavation to at least 1,5 m is likely to be required to install services and strip foundations. Deeper excavations could be required for the installation of sewers.

EXAMPLE 2 On commercial developments, excavation to considerable depths could be required to construct several floors of basements. Thus, the construction work force could encounter contaminated materials at these depths, and materials from these depths could be brought to the surface and either become spread about (if there is inadequate control) or have to be taken off-site for treatment or disposal.

Individual samples should be taken over a limited depth range (say 0,1 m to 0,2 m) to be representative of no more than 1,0 m of the soil profile, taking into account the contamination-related hypotheses. They should be restricted to a specific stratum. Samples should be taken to represent any strata of particular interest (e.g. appearance) that are encountered. Where samples are not restricted to a single stratum, the reasons should be stated in the report of the investigation.

At all sampling locations, samples should be taken through the full depth of interest in accordance with the hypotheses. Where contamination is indicated at greater depth than anticipated prior to the site investigation, sampling should be carried out to whatever greater depth seems appropriate and is practicable.

The sampling plan should also take into account whether there is a likelihood that the surface level of the site is to be lowered, which will thus expose deeper layers of the site.

NOTE 3 Taking samples of natural strata beneath deposited fill or disturbed natural ground is always beneficial: if uncontaminated, these will indicate the natural background chemical conditions which are essential to the evaluation of risks and decisions on target remediation values. In many sites, a correlation between contamination in the topsoil and the subsoil will be found. Often a correlation will also exist between contamination in the subsoil and contamination in the groundwater.

8.4.3 Sampling in relation to the groundwater profile and aquifer

Frequently, contamination migrates into groundwater in such a way that it is preferentially concentrated in, or along, the upper layers of the groundwater profile. Therefore, it is prudent to collect soil samples from these depths.

However, this depends highly on the density of the contaminant; contaminants like chlorinated hydrocarbons [Dense Non Aqueous Phase Liquids (DNAPLs)] show just the opposite behaviour and can be found in high concentrations at the base of the aquifer.

8.5 Size of samples

The size of the samples sent to off-site laboratories should take into account:

— the mass required to be sufficiently representative of the specified location (or sampling target), see 6.6;

— the range of pedological, chemical, physical and or biological examinations and tests that are to be carried out;

— the particle size distribution of the material be sampled;

— the specific requirements of the laboratory/laboratories carrying out the examinations and tests.

The guidance in 6.6 and Annex F on the size of samples should be followed. More sample than is required for the testing suite envisaged should be taken, in case additional analysis is required on receipt of the results, e.g. dependant options or repetition of tests to clarify or confirm the initial result.

A useful rule of thumb for fine soils is that at least 500 g to 1 000 g of soil as sampled should be obtained for chemical analysis (this applies both to single samples and composite samples, in the latter case after sufficient homogenization). Where the sample is of coarse-grained material, for example, gravels, a larger sample should be taken (see 6.6).

NOTE 1 Some of the techniques used to obtain samples are not capable of providing the precision required in sampling depth (see 9.4). In addition, segregation of coarse particles can occur with some sampling techniques. The persons designing the sampling strategy and implementing it in the field might need to make judgements as to what will constitute a "fit for purpose" or "good enough" sample that will meet the overall objectives of the investigation (e.g. by inspection of the sampling uncertainty to judge whether it is fit for purpose, see Reference [30]).

Samples obtained to serve as reference material or to be stored in a soil specimen bank should usually be larger than 2 000 g.

Any subsampling from the sample extracted by the equipment or measures to reduce the volume of material to be transported to the laboratory should be carried out according to ISO 18400-201.

NOTE 2 With smaller volumes of sample, it could be more difficult to ensure that the sample is sufficiently representative (e.g. as judged by the sampling uncertainty, see Reference [30]).

NOTE 3 Often, the size of sampling equipment is such that more soil material is obtained than needed. Sample pretreatment is then required "in the field", following the procedures described in ISO 18400-201 in order to limit the amount of material to be transported to the laboratory.

When potentially expansive slags are to be sampled for expansion tests specialist advice should be sought. These tests commonly require samples of about 50 kg or more to be taken.

NOTE 4 Some steel slags (both current production and old) and some old blastfurnace slags can have a tendency to expand. This expansion can occur decades after the slag has been deposited and can be triggered by disturbance that admits water and air, or by changing water levels (e.g. following a burst water main). Modern blastfurnace slags are, however, inert stable materials, see References [34] and [35].

Where the sampling of soil involves the separation of oversized material (i.e. mineral grains, sand, pebble and all other materials) due to very coarse-grained or heterogeneous soil conditions, the material removed should be weighed or estimated and recorded and described to enable the analytical results to be given with reference to the composition of the original sample. These procedures should be carried out according to ISO 11464.

8.6 Number of samples

8.6.1 General

The number of samples to be taken from the area, zone or other area of interest, should be decided in accordance with the guidance in 6.5 and Clause 7.

There might be several reasons why information is required especially on a potentially contaminated site where it might be necessary, for example, to obtain analytical and other data to aid assessment of risks to human health and the environment, to assess materials for waste disposal and to comply with requirements in relation to the health and safety of construction workers. When technically appropriate and practical, single samples, or at least a set of samples, should be taken to serve all purposes in a single operation.

8.6.2 Number of samples at individual sampling points

The number of samples taken should take into account as appropriate the need to:

— reduce uncertainty in the analytical and other results, e.g. taking more increments within a cluster sample reduces uncertainty in measurements arising from sampling (see 5.7 and Annex C);

— provide samples of more than one type to satisfy testing requirements (e.g. disturbed and undisturbed samples);

— provide samples of different size for test purposes;

— provide samples for different analytical and test regimes as required by the laboratories (see Note 1);

— provide duplicate samples for quality assurance purposes (see ISO 18400-106).

The practicality of providing near-equivalent samples will be constrained by the technique used to obtain them (ISO 18400-102), the particle sizes (maximum and distribution), and the nature of the soil (e.g. clayey, granular, organic-rich).

NOTE 1 Duplicate samples are usually taken a small distance apart to show the effect of small-scale heterogeneity (see Annex C).

NOTE 2 Often laboratories carrying out the chemical analyses will require more than one sample from a location with those for different tests being of an appropriate size and placed in an appropriate container (e.g. plastic tub, glass jar, vial).

NOTE 3 There are obvious practical difficulties, for example, when the material to be sampled is in the form of a 100 mm diameter core.

8.6.3 Number of composite samples

As appropriate, a number of spatial composite samples should be taken from the area or zone under investigation in order to reduce uncertainty in the analytical and other results and to meet the objectives of the investigation (see Clause 7).

The area or zone to be investigated can also be split into a number of sub-zones (this will aid identification of any variation of average properties across the area or zone that might require further more detailed investigation).

9 Sampling of above-ground deposits

9.1 General

It could be necessary to determine:

— the average properties of the deposit;

— the spatial distribution of properties.

The first step in sampling above-ground deposits (e.g. a stockpile or number of stockpiles) should be to carefully delineate the volume(s) to be sampled. In the case of a stockpile which cannot be satisfactorily differentiated from adjoining stockpiles, a "safety margin" should be applied in connection with the spatial definition of the stockpile such that adjoining stockpiles do not partially overlap.

If the deposit is non-consolidated, it can be difficult or even impossible to reach the 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. A final option would be not to sample the inside of the stockpile (see B.12.4), but this would mean that a non-probabilistic sampling method is used (see Annex E.).

If the deposited material to be sampled is effectively homogeneous (within the limits of interest) the spatial arrangement of samples (or increments in the case of composite samples) does not matter, i.e. random location of sampling points should suffice.

If the deposited material is known to be heterogeneous (e.g. the soil material has different origins or was disposed at different times):

— the average value of the property of interest can be obtained by taking a number of composite samples;

— the spatial distribution of the property of interest can be obtained using systematic sampling.

— the number of samples to be taken depends on the degree of reliability of the results that is required.

NOTE 1 These principle situations in general also apply for moving material on a conveyor belt.

NOTE 2 The sampling techniques used to obtain samples from stockpiles, etc. often differ from those employed in in-ground sampling (see ISO 18400-102:2018, Annex D). This can impose constraints on the selection of sampling locations, and the types and size of samples that can be taken.

NOTE 3 Annex D provides examples of sampling strategies suitable for stockpiles including for when the purpose is basic characterization of the soil population (D.2), determination of compliance or otherwise with guideline values (D.3), and verification, such as quality top-soil delivered to a site (D.4). Annex E discusses the scale of sampling required with reference to stockpiles.

9.2 Sampling patterns

Similar sampling patterns to those used for in-ground sampling should be used (see 8.2), although in many cases, these need to be implemented in 3-dimensions rather than 2-dimensions (see B.12).

9.3 Types of sample

9.3.1 General

In principle, the same options apply as for *in situ* sampling (see 6.4) but in the case of composite sampling the sampling pattern from which the increments are taken has usually to be implemented in three dimensions rather than two.

Convenience sampling can play a particularly useful role as described in 9.3.2.

9.3.2 Convenience sampling

A sample of the appropriate size is taken at a specific spot or location that has either been:

— 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.

NOTE 1 Convenience sampling will for instance be useful, when:

— a quick characterization of the material is necessary and representativeness is not directly relevant;

— specific parts of the soil stockpile seem to deviate from the bulk and characterization of these parts is desired (for ease of reference these areas are termed "target areas").

NOTE 2 Target areas might, following investigation, be deemed to be "hot spots" (8.2.3)

In an exploratory investigation, one sample location should be placed at the assumed centre of the target area.

NOTE 3 When there are obvious signs of contamination or other differences in the character of the material in the target area, this one sample might be sufficient to confirm that contamination is present and/or the nature of any differences from the bulk of the material.

When some information on the extent of the contamination is required in the exploratory investigation, another four sample locations should be placed at the expected perimeter of the contamination. Samples should be taken from each location and all relevant depths.

If hot spots are found (by chance) during the exploratory investigation, these hot spots should be investigated with a similar approach during the detailed investigation.

In the detailed site investigation, the number of additional sample locations should take into account the anticipated size of the target area (e.g. already identified hot spot) and the desired confidence of the delineation.

NOTE 4 If there is an area of considerable deviation in characteristics from the rest (hot spot), that is to be delineated, sampling profiles arranged orthogonally to the (estimated) border will provide information on the position and the slope of the gradient of the transition.

9.4 Sampling depths

In general, the question of the depth of sampling is encompassed by the adoption of a sampling pattern in three-dimensions.

9.5 Size of samples

The guidance relating to in-ground sampling provided in 6.6 should be followed.

9.6 Number of samples

The guidance relating to the number of samples required in 6.5, Clause 7, and 8.6 should be followed as appropriate having regard also to the examples in Annex D (especially D.2) and the information regarding the scale of sampling provided in Annex E.

Annex A
(informative)

Basic statistical concepts

A.1 General

This annex provides a basic outline of the statistical elements relevant for the ISO 18400 series.

The basic statistical concepts are the same for the different sampling strategies (e.g. the term "population" is used with the same meaning for in-ground sampling of a hectare of land or above-ground sampling of a stockpile), however, different concepts may be applied for *in situ* sampling than for above-ground sampling.

Formal definitions for many of the concepts discussed below are provided in ISO 11074 and/or Clause 3.

A.2 Population and subpopulation

A.2.1 General

These are statistical terms defining the entirety of material about which information is required. Specification of the population should be the starting point of any sampling exercise. Related specifications concern the statistical parameters of interest as well as the scale of interest if the material is granular and/or presents spatial variability. Indeed, these specifications can greatly influence both the type of sampling and the number of samples needed.

A.2.2 Population

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

In the context of soil quality investigations, the "population" is the entirety of soil (e.g. particular site, stockpile, truck load) about which information is to be sought via sampling. It is important for this to be defined explicitly over space and/or time. If this is not done, it is impossible to say whether a particular sampling programme will result in sufficiently representative samples.

Given the risk of multiple interpretations, it is important always to check that all involved parties are talking about "the same amount of soil", i.e. population, when carrying out a sampling exercise.

A.2.3 Subpopulation

It can be useful to define a subset of the population (termed the "subpopulation") and restrict the sampling to that more convenient region. This might be done because:

— doing so enables more information to be gathered about the characteristics of the whole population (see Clause 7 and Annex E); or

— for practical reasons – for example, it is difficult or even impossible to sample certain parts of the population; this might be the case if to sample the material at the core of a large soil population (e.g. a stockpile of substantial size) would require expensive equipment or be very time consuming.

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).

A.2.4 Population parameters

The range of the values taken for a physical or chemical property of a population can be displayed as a histogram of the sample data. If the range of property values is suspected to depend on, for example, differences in lithology, sampling tool, analysis method, etc., different histograms should be drawn to determine if these differences have to be taken into account. To simplify comparisons, histograms can be summarized in box plots showing the minimum and maximum values as well as several quantiles of the distribution (e.g. the median and the 25th and 75th percentiles).

Histograms can also enable approximate identification of type of distribution (e.g. normal, non-normal) and identification of possible outliers.

Other graphical presentations could also be helpful.

A variety of summary measures, or "parameters", can be used to characterize a population (e.g. the mean, the standard deviation, the median or the 90th percentile). An alternative term sometimes used is "statistical parameter" – in order to emphasize the distinction between this and other usages of the word "parameter".

A key step in planning a sampling programme is to specify the population parameters that should be estimated. Because most parameters depend on the scale considered – with the noticeable exception of the mean – the scale should also be specified, as discussed below.

A.2.5 Dependence on sample scale

The properties of interest are usually not homogeneous. If the material is examined at the scale of small samples (e.g. 500 g), it will display much more dispersed values than when it is described at the size of large cells making sense for remediation or intended future use. The probability distribution, and therefore the statistical parameters of the population, depend on the scale considered. Specification of the sample scale is therefore part of the specification of the population.

A noticeable exception is the mean, because it does not vary with scale. For example, if the site of interest (the entirety of the soil) is divided into identical cells (e.g. cubic cells or prisms with a hexagonal base), the mean of the cell values is the mean of point values in the whole site and therefore does not vary with the size of the cells. However, the dispersion of the individual values around the mean depends on the shape and size of the cells. This is therefore the case for dispersion parameters such as the variance of the population and for mixed parameters such as the median and the 95th percentile of the population.

If the scale of interest is very large, it is usually not possible to directly sample at that scale. Suitably defined composite samples can, however, approximate the scale of interest, as shown in Annex E. Geostatistics provides tools for relating the scale of spot samples to a larger scale. They can be used when spot samples are available and a study of spatial variability is of interest (see Annex H).

A.2.6 Dispersion variance

When the site of interest is fully divided into discrete identical cells, the variance of the population is named "dispersion variance". It depends on the size of the cells and on the size of the site. If in the case of a regular or random pattern of spot samples, the site can be seen as divided into very small cells similar in scale to the samples; the variance of the samples can be considered as an approximation of the dispersion variance of these cells within the site. This approximation becomes more reliable as the number of samples increases. The dispersion variance for cells larger than the sample scale [e.g. at a scale that makes sense for remediation such as 100 m^3 (about 170 t)] can be obtained with geostatistical techniques.

In the case of a regular or random pattern of composite samples (cluster samples or spatial composite samples deemed representative of an area of medium size), the variance of the composite samples can be considered as an approximation to the dispersion variance at the scale of the area where the increments of a composite sample are selected. This approximation becomes more reliable as the number of composite samples and the number of increments per composite sample increase. Note that the areas encompassed by the separate composite samples should not overlap (something that might

happen by chance with a random sampling pattern for sampling locations that happen to be close together).

A.3 Variability

A.3.1 General

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 "size" of the samples, i.e. the minimum volume of material within which variations in quality are of no concern.

NOTE In the case of composite samples, scale is that of the area where the increments are selected, whereas size is the total volume of the increments. The fundamental error variance is related to size and not to scale.

A.3.2 Types of variability

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

Two distinctive types of variability can be distinguished:

— at very small scale, that of soil particles, i.e. fundamental variability;

— at larger scales, i.e. spatial variability.

In stockpiles and other anthropogenic deposits, spatial variability can be linked with temporal variability when temporal processes determine the spatial variability within the deposit (e.g. a stockpile) or between deposits (e.g. a number of stockpiles). When temporal effects are expected based on the process wherein the stockpile or other above-ground deposit was formed, these effects should be accounted for in the assumptions on the spatial variability.

A.3.3 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 F provides the details of a method that can be used to estimate the minimum size of increments and samples of particulate materials when the property of interest is inherent to the particles 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 size of a sample (the total volume of its increments in case of a composite sample) should be such that the effect of individual particles on the results from the sample is small.

Note that fundamental variability acts twice: (i) in the field when extracting a sample, and (ii) in the laboratory when extracting a fraction of the sample (after suitable grinding) for chemical analysis. This text focuses on the former, the laboratory being left to cope with the latter.

A.3.4 Spatial variability

The soil usually comprises different soil units (type, horizon) with different statistical characteristics. These characteristics should be determined for each soil unit (each soil unit is a distinct population). Even within a soil unit, the samples usually display variations: this is due to spatial variability. The spatial variability is an inherent characteristic of the population. Without manipulation of the material (e.g. mixing the soil unit), it will not change.

Recognition of this spatial variability is essential for the proper assessment of soil quality (e.g. in sites, stockpiles). Whether one is interested in the variability within the whole site or within a soil unit, the potential occurrence of spatial variability has a direct effect on the sampling strategy to be chosen. This effect exists even if there is no specific interest in the spatial variability (e.g. the objective is only to know the average value of a property), because spatial variability has an impact on the definition of a sample that would be representative of the whole population. However, when spatial variability is likely to occur, it is not safe to represent the whole population by one or two composite samples. Indeed these can mask locally high concentrations within a background of low concentrations. Or, on the contrary, very high local concentrations can so much impact the average grade that the whole site or above-ground deposit could be considered to exceed a regulatory or similar limit. It is, therefore, necessary to restrict composite samples to a reasonable scale, for example the size of the cells or units for which separate decisions can or will be made about whether remediation is required.

Setting aside fundamental variability, spatial variability usually occurs at all scales from that of sample increments to that of large units or even the whole site of interest. If spatial variability is of interest, it can be characterized with geostatistical tools such as the variogram if a certain number of spot samples or cluster samples are available (a minimum of 30 samples is commonly reported as being required but a higher number is required to obtain truly sound results). See Annex H.

A.3.5 Variability between soil units

For quantification of the variability between soil units, the variability within the individual soil units should either be quantified or should be incorporated in the composite samples that are used to characterize the quality of each individual soil unit.

A.4 Estimation 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 called "estimation error". It is also known as "statistical sampling error".

A.5 Reliability

A.5.1 General

The greater the amount and quality of sampling, the smaller the estimation errors are likely to be and 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.

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

A.5.2 Bias

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

Bias can also be due to a lack in the sampling procedure or the sample pretreatment (e.g. the large size particles, or on the contrary the fines, are not included in the sample), or a lack of calibration of the chemical analysis procedure. In the case of several sources of bias, the total bias is the sum of the partial biases.

If the bias is known, it is essential to correct the estimate in order to obtain an unbiased estimate. Otherwise it is often not possible to detect a bias by simply inspecting the data, except by comparison with reference data. It is therefore essential to check every possible source of bias.

A.5.3 Precision and confidence

Assuming that an estimate is unbiased, another desirable property is that it is as precise as required, that is, the magnitude of the estimation error be as low as required to achieve fitness for purpose. The magnitude of the estimation error is usually characterized by its variance, called "estimation variance", or by its square root, the "estimation standard deviation". This variance can be calculated with geostatistical techniques. In specific cases, approximations to these variances or upper bounds can be obtained using simple statistical formulas.

Assuming a type of distribution for the estimation error, the estimation variance can be translated into an error band – known as a "confidence interval" — to be placed around the property estimate. The "precision" of the estimation is often defined as the semi-width of the 95 % confidence interval (or equivalently as twice the estimation standard deviation in the case of an assumed normal distribution for the estimation error). It is also sometimes defined as equal to the estimation standard deviation itself. When giving a precision, it is therefore necessary to specify the adopted definition. The precision depends on

— the variability in the population or subpopulation,

— the pattern of sampling (see 6.2 and 9.2), 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.

A.5.4 Estimation of statistical parameters

When the scale of the samples coincides with the scale of interest and the samples can be considered "independent" (i.e. uncorrelated), statistical methods enable the estimation of commonly used parameters. For example, in the case of a simple random pattern of spot or cluster samples, the population mean can be estimated by determining the mean of the samples and the estimation variance of the mean can be approximated by one nth the dispersion variance where n is the number of samples (in the case of a stratified random or regular pattern, the estimate is more accurate than for a simple random pattern, so that this expression is an upper bound for the estimation variance of the mean). G.2 provides statistical expressions for estimating the mean of a population, its standard deviation or a percentile at the scale of the samples, a percentage compliance with a given limit, as well as methods for calculating the uncertainty associated with these estimates. The last 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.

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.

Because each sample can be considered as an estimate of the mean with an estimation variance of 35^2 = 1 225, and because the samples are independent, the mean of the 30 samples is an estimate of the true mean with an estimation variance of 1 225/30 = 41, corresponding to an estimation standard deviation of 6,4 mg/kg (i.e. $\sqrt{41}$).

Assuming a normal distribution of the estimation error, a 90 % confidence interval for the mean cadmium would be of 1,645 standard deviations around the mean, that is

45 mg/kg ± 11 mg/kg, i.e. between 34 mg/kg and 56 mg/kg.

NOTE In the presence of spatial variability the above calculation is an approximation to the exact calculation and is valid only for simple random sampling. This is however not a reason for selecting a simple random sampling pattern. In the case of stratified random sampling or regular sampling, the estimation variance is smaller — and often much smaller — than with simple random sampling. A geostatistical approach is needed to show how much smaller it is.

In most situations not covered by statistical formulae, a geostatistical approach can be used, not only when:

— the scale of interest does not coincide with the sample scale (e.g. the interest lies in properties of large cells whereas spot samples are available);

— the concern is in local properties rather than global properties (e.g. the average value of a property in a given cell, or the probability that this average property exceeds a given limit);

— the sampling pattern is random or regular, but also when the sampling points are (witihin certain limits- see below) unevenly distributed provided that sampling is not systematically focused on specific areas such as locations where high concentrations are expected; for example, declustering techniques enable the use of a combination of a regular pattern and of judgemental samples in areas where large concentrations are likely;

— auxiliary information is to be incorporated (e.g. a qualitative property, or a secondary property linked with the property of interest and known at any location).

A series of geostatistical methods enable the calculation of optimal estimates (in a statistical sense) and of the corresponding estimation variances. A preliminary to the application of geostatistics is the characterization of spatial and/or temporal variability with the variogram and other spatial exploratory analysis tools. See Annex H for further information.

Annex B
(informative)

Sampling patterns

B.1 General

B.1.1 Overview

Most natural properties of the soil vary more or less continuously in space and, as a consequence, the values at locations that are close together are more similar than those further apart. They depend upon one another in a statistical sense. This property is known as spatial dependence. Geostatistics, i.e. spatial statistics, provide tools such as the variogram for the characterization of spatial variability, and methods for rationalizing sampling and analysing its results, including for multi-stage or nested sampling (see Annex H).

A fruitful property of geostatistical methods is that, once spatial variability has been characterized, the accuracy of a sampling scheme for estimating, for example, an average concentration, does not depend on the observed values at the sampling points, but on the configuration of the sampling points in relation to the area or block (if three dimensions are considered) to be estimated. This accuracy can therefore be evaluated prior to sampling assuming there is no sampling or analytical bias

However, at early stages of the characterization of a site, when no data enable the characterization of its spatial variability, more general considerations are used to choose a sampling pattern. Theoretical results enable the comparison of standard patterns such as simple random pattern, stratified random pattern and a regular square grid.

Another approach is to determine which pattern will be best for detecting hot spots of limited extension. A square grid, for example, will detect any circular hot spot whose diameter exceeds the square diagonal. From this point of view and for the same sampling effort, a regular grid of equilateral triangles enables the detection of smaller hot spots. The square pattern is however usually preferred because it is viewed as simpler to put into practice. If the presence of thin elongated hot spots is suspected, less regular patterns can be preferred, such as stratified random sampling or systematic unaligned sampling.

NOTE Figures B.5 to B.8 show a site in which there are areas of interest (of contamination in this case) randomly distributed. The figures show that the number of these areas that will be sampled depends on the sampling pattern. Which ones are sampled will depend on the "origin" of the sampling pattern. The number of sampling locations is 10 to 12 in each case. Further illustrations of sampling patterns are provided in Annex I.

B.1.2 Bayesian approaches to investigation and sampling

In practice, very few site investigations, and hence sampling exercises, are carried out without at least some knowledge of what to expect. The available information is used to aid decisions about what, where and how to sample, and what substances and properties should be measured. By staging investigations, knowledge about what is, or is not present, can be used to inform the design of subsequent stages of investigation and to improve the efficiency of the overall investigation, e.g. by reducing the total number of investigation points required or to enhance the value of information gained from each sampling point. This process can be aided by application of statistical techniques including sometimes Bayesian statistics.

Bayesian statistics should only be used with expert advice. Proper application of a Bayesian approach requires that formal hypotheses (e.g. the probability of a hot spot of contamination being present) are explicitly stated before the sampling plan is prepared — it is not something that can be done with hindsight.

A simple application would be to use Bayesian approach to justify statistically a decision that on a "green field" site, sampling density can be less than on a brownfield site (there is a formally stated low expectation of finding anything untoward). If any evidence of contamination is found, however, a second stage of investigation is required – reliance on average concentrations or properties for the subsequent assessment of risks, etc. would be inappropriate.

Bayesian statistics requires "prior knowledge" of the situation. In site investigation, it is difficult to ensure the accuracy and applicability of this prior knowledge. Application of Bayesian statistics cannot, therefore, be regarded as a customary method with widespread application. They should only be used with advice from a statistical expert.

B.1.3 Sampling in three-dimensions

Sampling patterns and the associated statistical considerations are usually restricted to two dimensions. Having decided on a sampling approach, e.g. random or application of a regular grid, sampling depths are decided on taking into account the considerations listed in Clauses 8 and 9 about particular sampling situations. This approach reflects the practicality of taking samples at depth and also the fact that in most site investigations, the variation in properties with depth occurs on a much smaller scale than laterally. Although sampling is sometimes carried out at regular depth intervals (e.g. every 0,5 m or 1,0 m), it is also common to sample individual horizons or materials that are of "interest" which might be only 100 mm or so thick. Thus, a large element of judgement is usually employed when deciding on sampling depths.

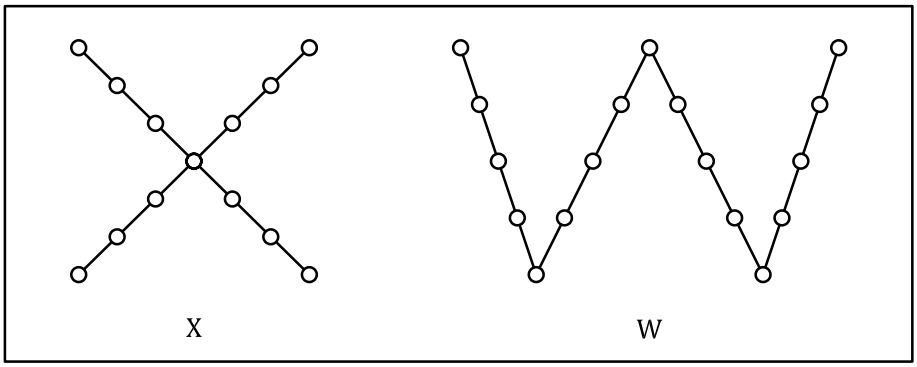
When samples are taken at fixed depth intervals at locations on a predetermined sampling pattern, a three-dimensional pattern is in fact established, albeit one that is usually far from equant in its dimensions. For example, when samples are taken at fixed depth intervals this is likely to be at 0,5 m or 1,0 m whereas the sampling grid might employ intervals of 10 m, 15 m, or 20 m between sampling points. As discussed in B.12, more equant grids, or even random sampling in three dimensions might be necessary or possible when investigating ore bodies or above-ground deposits (e.g. large consolidated stockpiles) but the practicality of doing so is often limited because of the technical complexity of doing so in relation to their value compared to other possible approaches.

B.2 Patterns for spatial composite sampling

Widely used in agricultural/horticultural land investigation are the "N", "S", "W" and "X" patterns of sampling (Figure B.1). The general premise is that the distribution of soil constituents is relatively homogeneous. Along the outline of such a pattern, a number of increments are taken and then bulked and mixed to provide one composite sample for analysis.

For the reasons explained below the "W" pattern is generally favoured. Increments should be taken from at least 25 locations in the area to be sampled. This applies irrespective of the size of the area, whether it is a small garden bed or a 4 ha field (above this size is best to sub-divide the site into at least two zones [see 7.3.2)]. Research has shown that a composite sample composed of 25 increments will usually constitute a sufficiently representative sample of soil. See Reference [36].

The distribution of sampling points is likely to be inadequate to identify the location of point contamination, and in any event high contaminant levels will be lost in mixing of these samples. Thus in the majority of contaminated land investigations these patterns are unlikely to be useful, because they obscure high levels of point contamination.



Key

○ points from which increment is taken to form composite sample (number of points illustrative only)

Figure B.1 — Spatial composite sampling patterns

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

There can be advantages in splitting a site into zones in this way and taking a composite sample from each zone irrespective of whether there is an obvious basis for such zoning such as those mentioned above because it can reveal unexpected differences or confirm the assumed relative homogeneity of properties.

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

Application of diagonal patterns should be based on the following:

— estimation of uniform distribution of substances;

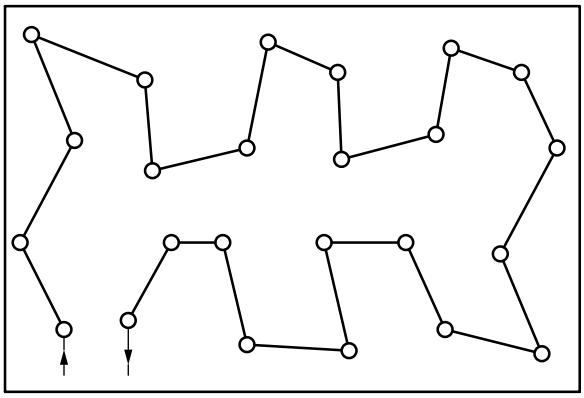
— useful only for uniformly developed areas (deviating parts of the area should be sampled separately);

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

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

— selection of sampling points is independent of local characteristics (points should preferably be fixed by measurement or pacing).

Traversing the area in a zigzag manner similar to that shown in Figure B.2 is another way of obtaining increments to make up a composite sample.



Key

○ points from which increment is taken to form composite sample (number of points illustrative only)

Figure B.2 — Zigzag traverse sampling pattern

B.3 Sampling patterns for permanent monitoring sites

A general exception to the "biased diagonals pattern" was developed for permanently monitored areas to obtain information about long-term changes within selected sites due to human influence. The aim is to make available samples from an area representative of the surrounding environment for a defined number of examinations to be carried out over a period of some years.

The following procedure is recommended (see Figure B.3):

— select a representative area of approximately 1 000 m^2;

— divide this area into four squares, each of 250 m^2;

— within each square, draw two diagonals along each of which nine increments are obtained;

— take samples according to the specified requirements;

— prepare composite samples 1, 2, and 3 by:

  — mixing increments from positions 1, 4, 7, 10, 13, and 16 to give composite sample 1;

  — mixing increments from positions 2, 5, 8, 11, 14, and 17 to give composite sample 2;

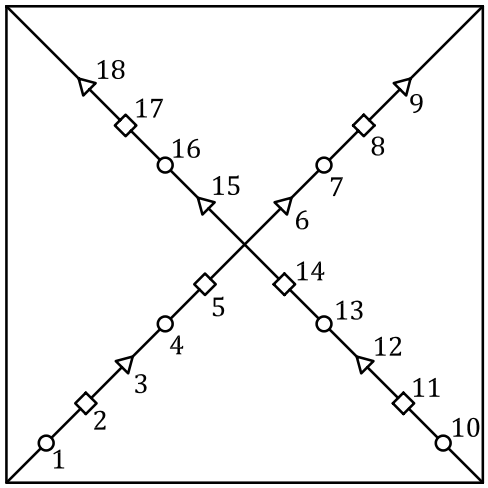
  — mixing increments from positions 3, 6, 9, 12, 15, and 18 to give composite sample 3.

Rotational sampling of the area can be conducted by:

— taking increments at the intersections of the sampling points (positions 1 to 18) as described above;

— rotating the diagonals clockwise around the centre of the square in steps of 22,5 ° so that, all in all, four sampling exercises can be carried out at undisturbed positions.

An area selected and sampled according to the above-mentioned scheme serves for eight sampling episodes. After the final episode, the area might be considered unsuitable for further sampling. Extension or reduction of the dimensions of the test area might require changes in the total number of samples and therefore also affect composite samples.



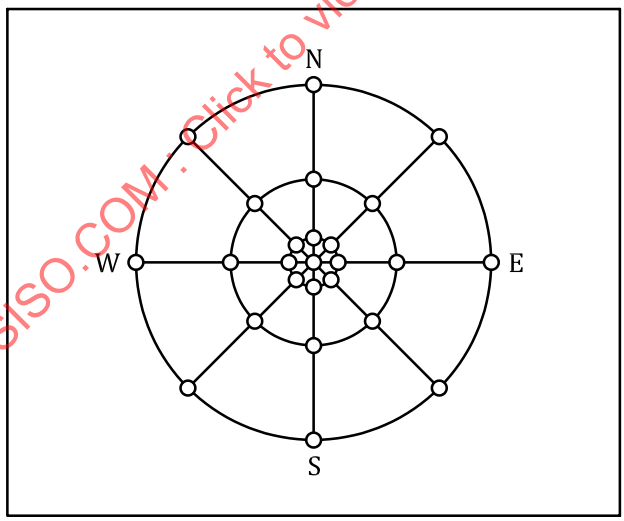
Key

○, ⍰, Δ points from which increments are taken (see text for number sequence)

Figure B.3 — Pattern of rotating diagonals for permanently monitored areas

B.4 Circular grids

Circular grids are useful for delineating localized contamination areas such as storage tanks but also to indicate influences around a regional emitting source, e.g. precipitation from industrial plants. Spot samples are taken at the intersection of concentric circles (the radii of which depend upon the suspected area of contamination) and the lines of the main eight points of the compass (see Figure B.4).



Key

○ points from which samples are taken

Figure B.4 — Circular grid

Sampling based on circular grids can provide a range of information. For example:

— information on substance concentrations in the grid centre (maximum values);

— information on distribution of contamination (dimensions of particular area with increased contamination);

— shape of distribution of contamination.

Disadvantages of circular grids are:

— star-shaped (radial) location of sampling points is practicable but not optimal (rotation of concentric circles of 22,5° leads to a higher quality of the pattern);

— relationship of sampling point densities of the (usually) eight samples close to the centre and those (usually) eight samples at a greater distance might not be optimal in every case (if, for example, borders of distribution of a contaminated area are sought, fewer central points should be sampled and more samples at the margins of the grid);

— circular grids might imply a uniform extension of contamination in all directions. This is usually not the case. Preferred directions, e.g. due to main wind direction in case of airborne contaminants, should be considered in modifications of the circular grid, e.g. an increased number of sampling points in critical directions, extending the distance of sampling from the centre in critical directions;

— circular grids generally are not applicable for taking composite samples, because the values measured give information neither on the average nor on the maximum concentration of the area sampled.

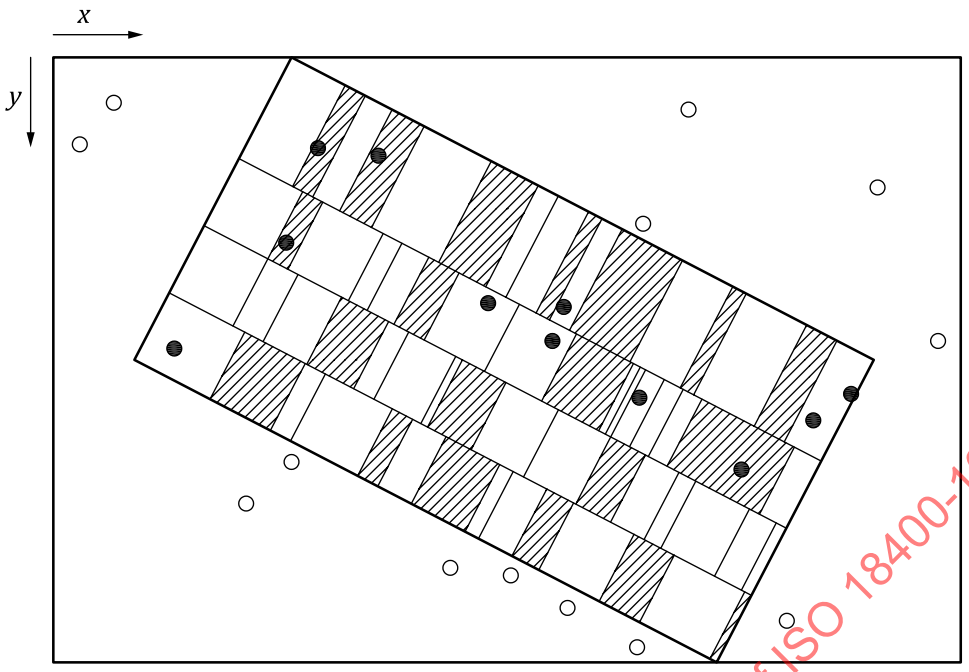
B.5 Simple random sampling

In cases of presumed irregular occurrences of contaminated zones, random sampling may be applied. Sampling points within the area are selected by using random numbers which can be found in tables included in manuals on statistics or generated by computer programmes. This technique has the disadvantage of irregular coverage and makes interpolation between sampling points difficult (see Figure B.5). It is also not efficient for the identification of hot spots.

A suitable procedure is the following. Encompass the site of interest in a rectangle parallel to the coordinate axes. Sample coordinates are selected independently and randomly within this rectangle. When the coordinates define a point outside the site, another set of coordinates is selected.

In general, random sampling can also be applied for soil fertility investigations, etc.

In practice, random sampling (in its purest form) is rarely used in soil surveys.



Key

- contamination
- sampling point
- invalid point outside of the site boundary

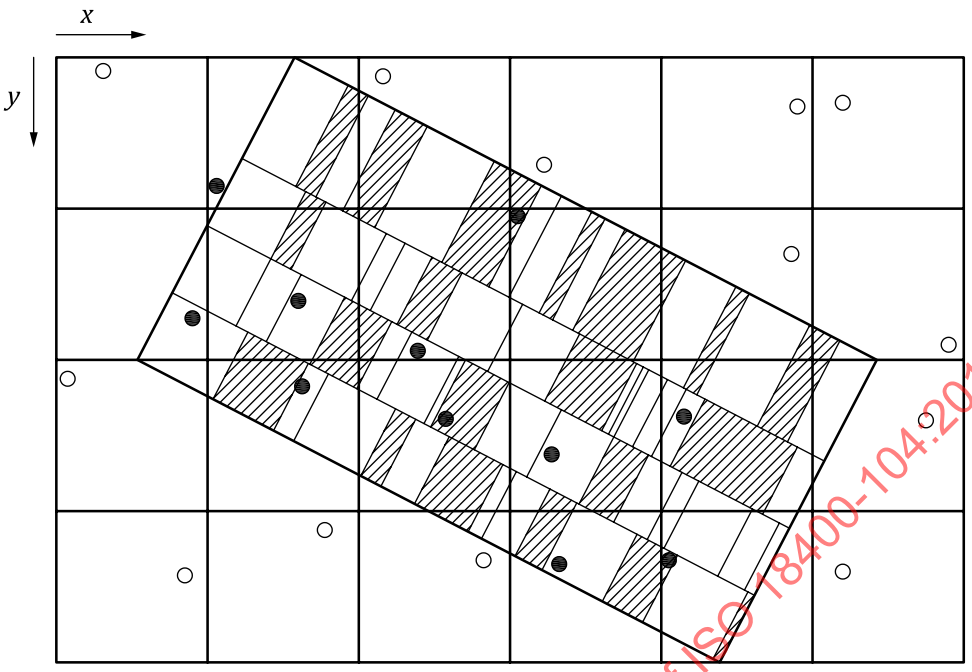
Figure B.5 — Random sampling pattern

B.6 Stratified random sampling

This method avoids some of the disadvantages of random sampling. The site is divided into a number of grid cells, and a given number (usually one) of randomly distributed sampling points is chosen in each cell (Figure B.6).

In practice, no point is chosen in a cell that has no intersection with the site. In the case where the cell belongs partly to the site, the random point is retained if it falls within the site, and is rejected otherwise; in the latter case no other point is chosen.

Stratified random sampling and systematic unaligned sampling (see B.8) are recommended for soil fertility investigations to avoid the influence of previous field operations, fertilizer banding, and other factors that tend to follow straight lines.



Key

contamination

sampling point

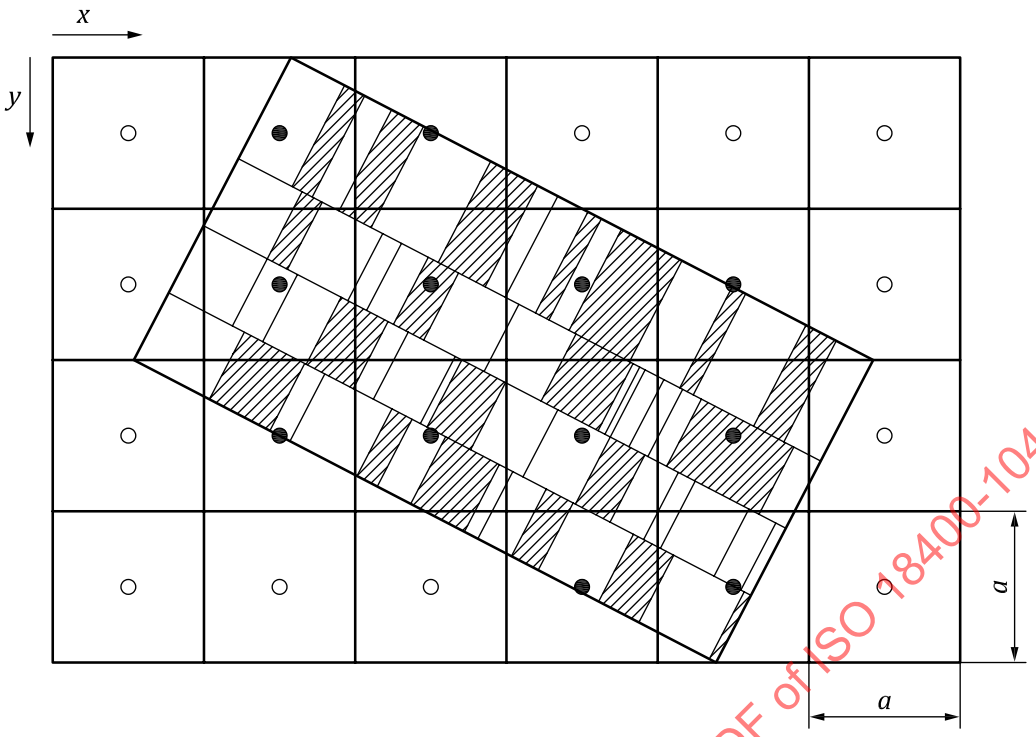
invalid point outside of the site boundary

Figure B.6 — Stratified random sampling pattern

B.7 Regular grids

In many cases, a regular grid is selected for soil sampling (Figure B.7). There is a direct relationship between the spacing of sampling points and the detection of hot spots of contamination. Circular hot spots, for example, are detected with certainty if their radius is larger than 0,71 a, or equivalently if their area is larger than 1,57 a^2, where a is the grid spacing.

The assigned grid spacing depends on how much detail is required. It will differ according to the objective of the sampling, e.g. to collect samples of average degree of contamination, to locate isolated sources of contamination or to establish the extent of contaminated zones (horizontal and vertical). The latter is of particular importance in cases where a contaminated area has already been located and a follow-up sampling programme becomes necessary (see also Annex I).



Key

contamination

sampling point

invalid point outside of the site boundary

Figure B.7 — Distribution of sampling points on a regular grid

Although more frequently used for the investigation of soil contamination, regular grids are also suitable for soil fertility investigations, etc.

An advantage of a regular grid is that it can be set up easily and grid dimensions can be readily varied. It has the disadvantage of being prone to bias from linear features across the site (e.g. drains that are either under or over represented). This disadvantage can be overcome by using unaligned grids, (see B.8 and B.9).

Interpolation between sampling points and return to the grid to carry out a more intensive sampling in localized areas to further delineate point sources of contamination is easy.

NOTE Figure B.7 shows the sampling points located in the centres of cells demarcated by the grid to better illustrate how this regular pattern relates to those shown in the other figures. In practice, it is more usual to fix the sampling points at the intersections of the grid lines.

B.8 Systematic unaligned sampling

Systematic unaligned sampling is an intermediate between a regular grid and stratified random sampling. It involves taking samples off set in a both random and systematic way from the nodes of a regular grid (see Figure B.8). This improves the chances of locating an elongated area of atypical properties.

Unlike a regular grid, the samples of a row or column are not aligned, which avoids possible sampling bias when a spatial periodicity in the contaminant concentration is suspected, due to the previous use of the site.

The samples of a row (resp. column) always display the same pattern — up to a translation, giving globally a more systematic effect than stratified random sampling.

When compared to a regular grid or even to stratified random sampling, systematic unaligned sampling improves the chances of locating an elongated (e.g. oval or tear drop) area of atypical properties or a "long thin" hot spot (e.g. along line of old pipeline or cable) where this might by chance be approximately aligned with one of the grid axes.

The procedure giving a pattern such as that illustrated in Figure B.8 is as follows.

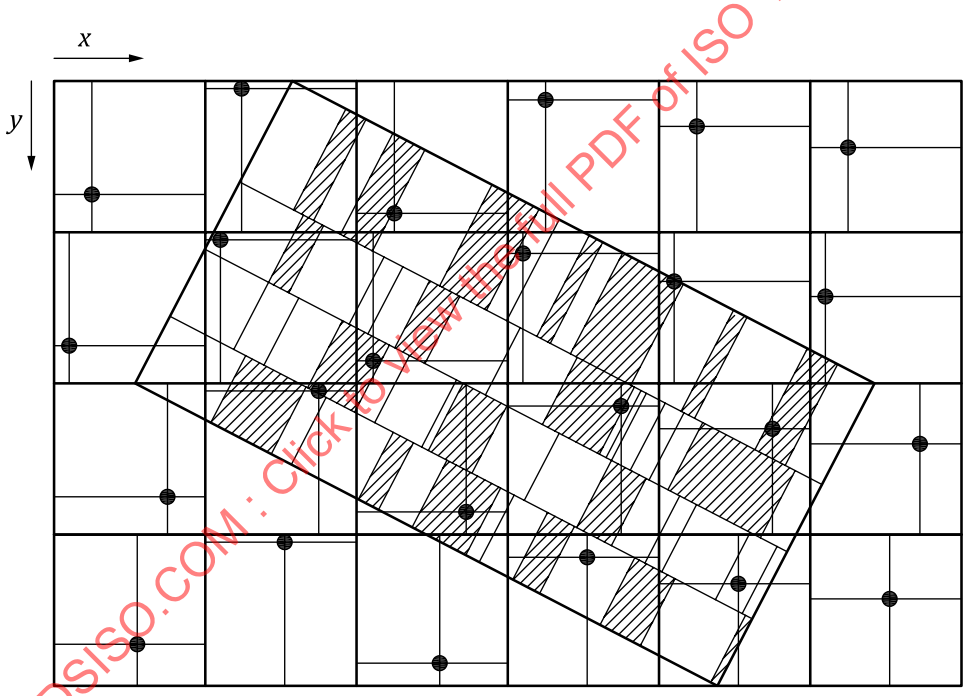
EXAMPLE Given a grid with 24 cells (squares), arranged in 4 lines and 6 columns (Figure B.8).

— For the first cell (line 1, column 1) x- and y-coordinates are chosen at random.

— For cells 2, 3, 4, 5 and 6 (upper row) only the y-coordinates are chosen at random.

— For cells 7, 13 and 19 (left-hand column) only the x-coordinates are chosen at random.

— All sampling points are now located on the grid. For all sampling points in the columns, the y coordinates of cells 2, 3, 4, 5 and 6 are valid and for all sampling points in the lines the x-coordinates of cells 7, 13 and 19 are valid.



Key

contamination

sampling point

Figure B.8 — Systematic unaligned sampling pattern

B.9 Herringbone pattern

The "herringbone" pattern is a modification of a regular grid achieved by alternately offsetting the x and y coordinates of the grid nodes by a constant amount, see Reference [37]. It is a special case of the systematic unaligned pattern. It can be set out easily on site, as it can be generated by four square grids, each with the same orientation and spacing.

The "herringbone" pattern was designed for improving the probability of detecting elongated (e.g. oval or tear drop) or "long thin" hot spots (e.g. along line of old pipeline or cable) in unfavourable orientations.

Its properties depend heavily on the two relative offsets selected. In practice the pattern displayed in Figure B.9 is used because it represents the best choice, as shown by computer experiments.

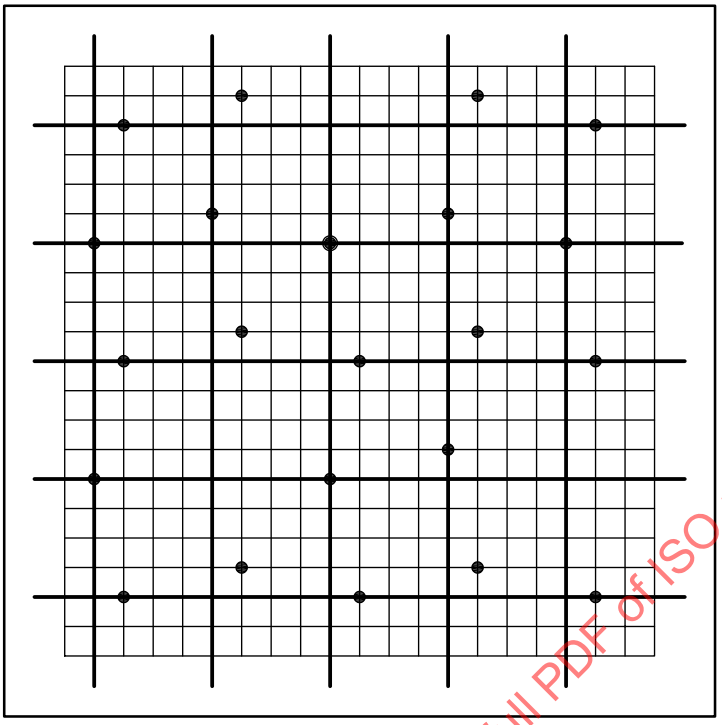


Figure B.9 — Herringbone pattern

B.10 Systematic sampling on a triangular grid

In case of an equilateral triangular grid (Figure B.10), each grid point is neighboured by six grid points at the unique distance, d.

To put this pattern into practice, sampling points are fixed at a distance of d in parallel rows spaced at a distance of $(\sqrt{3}/2)d$, i.e. approximately 0,87 d. The sampling points on the parallel rows are staggered by $d/2$.

The free, unsampled disk defined by three adjacent points has a radius of $(\sqrt{3}/3)d$, i.e. approximately 0,58 d.

Therefore, any circular contamination is certain to be detected when its radius is larger than 0,58 d, or equivalently its area is larger than 1,05 d^2. Compared with a square grid with the same sampling density (that is, with $a^2 = d^2\sqrt{3}/2$), this area is 23 % smaller.

Thus, just by changing the pattern (and with the same sampling effort) the size of the unsampled circular area decreases by 23 %.

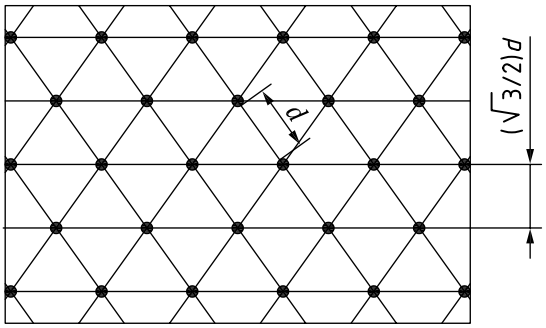


Figure B.10 — Triangular grid

B.11 Sampling along a linear source

In the case of contamination following a line, e.g. caused by leaking pipelines, sampling points can be arranged in the covering soil directly above the pipeline or, if this not practicable for certain reasons (e.g. safety), close to the pipeline. If the distribution of contaminants caused by a line-like structure is also of interest, it is recommended to take samples at a distance "*x*" one from another above the line and further samples at increasing distances (e.g. 2*x*) parallel to the line (see Figure B.11).

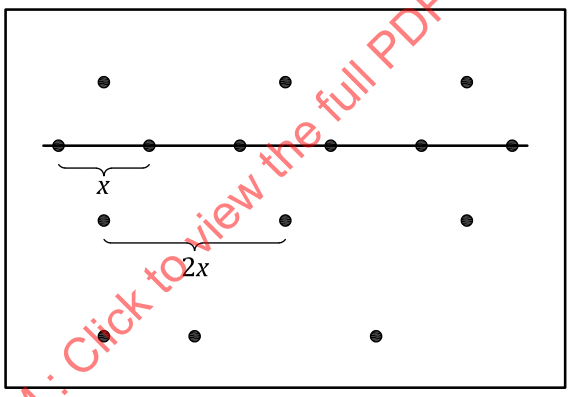


Figure B.11 — Sampling along a linear source

B.12 Sampling in three dimensions

B.12.1 Simple random sampling in 3 dimensions

Random sampling can be applied in three dimensions, e.g. when sampling a stockpile, following the procedure described below. However, as noted above (B.5) random sampling is more usually applied in two-dimensions, by specifying the location of boreholes randomly and then taking samples from each borehole either at fixed intervals or using a technique which provides a continuous sample (e.g. a core) from which (sub)samples for testing can be taken in the field or following transfer to the laboratory.

Three-dimensional random sampling is only feasible where application of the chosen sampling equipment in conjunction with the appearance of the ground or stockpile (e.g. maximum particle size, degree of consolidation, size/height) enables the sampler to take the samples in a safe way while the identity of the sampled material is guaranteed.

A suitable procedure is the following: Define the soil to be sampled (the population) precisely, and encompass it in a rectangular parallelepiped oriented like the coordinate axes. Samples will be taken on coordinates that are randomly chosen within this parallelepiped. When the coordinates define a point outside the soil to be sampled, another set of coordinates is chosen.

B.12.2 Stratified random sampling in 3 dimensions

Stratified random sampling in three dimensions requires samples to be taken from each of a set of three-dimensional cells. A three-dimensional grid is established in the rectangular parallelepiped described in B.12.1. A random location is then defined for each cell. If this location does not belong to the soil to be sampled, it is rejected and no sample is selected in the cell.

If spatial variability is greater vertically than horizontally, which is a common situation, the dimensions of the grid cells are set to be smaller vertically than horizontally.

When a single point is chosen in each cell, the number of samples is approximately the ratio of the volume of the soil to be sampled to the cell volume.

B.12.3 Systematic regular sampling

The regular grid pattern applied in a horizontal plane can also be applied e.g. in case of a stockpile or *in situ* volume, in a vertical direction. The principle of the regular sampling pattern is that the distance between sampling locations both in horizontal and, if relevant, vertical direction is constant. However there is no need for the horizontal distance to be equal to the vertical distance. The vertical distance is usually set to be smaller due to the often larger variability along the vertical than along the horizontal.

A three-dimensional grid is established in the rectangular parallelepiped described in B.12.1. A sample is placed at the centre of each cell, provided that it belongs to the soil to be sampled. The number of samples is approximately the ratio of the volume of the soil to be sampled to the cell volume.

B.12.4 Sampling the surface of above-ground deposits

Samples can be taken from the surface of a stockpile or other above-ground deposit at locations determined by a grid or random sampling pattern laid over its surface or at intervals around the base when sampling over the surface is not practical (e.g. because the sides are steep and/or not sufficiently firm to be trafficked); see Figure B.12. Such 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 could be feasible for the sampling to be sufficiently representative. 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 can be taken according to a systematic pattern from the top 0,50 m 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), while 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 30 mm auger from a soil with a maximum particle size of 50 mm. The larger particles (i.e. those >30 mm) will not be part of the sample. In addition, as it is usually necessary in order to obtain a sufficiently representative sample for the diameter of the sampling tool to be greater than about three times the diameter of the largest particles present (see F.2) it is very difficult to know what part of the overall population the samples represent. For example, particles of 25 mm diameter, will have only a small probability of entering the auger and therefore are likely to be under represented.

Spot samples of the appropriate size can be taken at specific locations that have 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.

Convenience sampling might be appropriate when:

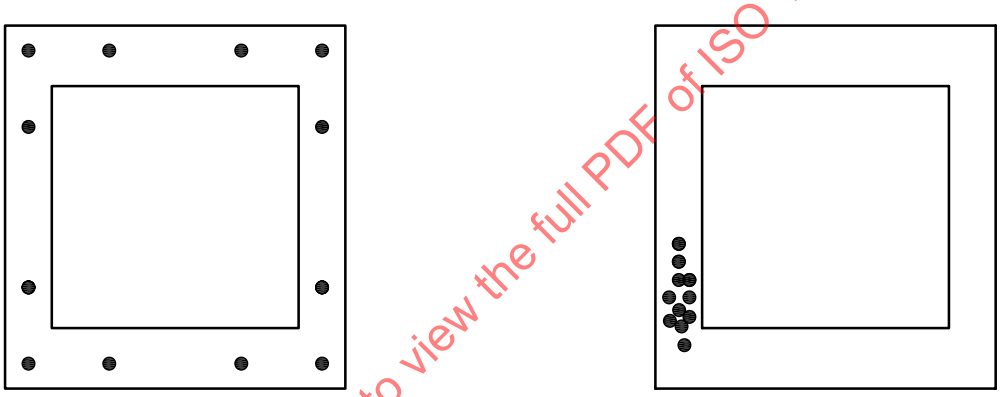
— a quick characterization of the material is necessary and representativity is not directly relevant;

— specific parts of the soil stockpile seem to deviate from the bulk and characterization of these parts is desired.

During an exploratory investigation, one sample location should be placed at the assumed centre of any target of interest, e.g. hot spot. For contamination that can be observed in the field, this one sample might be sufficient. If contamination is not observable in the field, and when some information on the extent of the contamination is required from the exploratory investigation, another four sample locations should be placed at the expected perimeter of the assumed contaminated area. Samples should be taken from each location and all relevant depths. If hot spots are found (by chance) during the exploratory investigation, these hot spots should be investigated with a similar approach;

During a main/detailed site investigation, the number of additional sample locations depends on the extent of the contamination and the desired confidence of the delineation.

NOTE If there is an area of considerable deviation in characteristics from the rest (hot spot), which is to be delineated, sampling profiles arranged orthogonally to the (estimated) border will provide information on the position and the slope of the gradient of the transition.



a) Sampling around a stockpile

b) Convenience sampling around an area of interest or restricted point of access

Figure B12 — Surface sampling of above-ground deposits

B.12.5 Directional sampling

A directional sample may 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. A number of samples or a full-length sample can be taken in a specific direction through the soil stockpile, resulting in a composite sample throughout the full direction of sampling. However, this has the disadvantage that any differences between layers are masked – it depends on the circumstances whether this is acceptable.

Annex C
(informative)

Assessment and modification of sampling uncertainty

C.1 General

Total measurement uncertainty includes random (precision) and systematic (bias) effects from sampling in the field, sample preparation (e.g. subsampling within the laboratory), and from the process of chemical analysis. For this purpose, it is not necessary to identify or quantify the individual sources of errors in terms of sampling theory, such as fundamental sampling error applied in Annex F. The uncertainty arising from sampling process is generally greater than that arising from the analytical process.

Estimates of total measurement uncertainty (including sampling uncertainty) are useful for the following purposes.

— Judging, demonstrating and, if necessary, improving the fitness-for-purpose of the measurements for any particular site (e.g. in an exploratory investigation). The resulting uncertainty estimates can be used to minimize overall expenditure on site development; see Reference [30].

— Making a probabilistic interpretation of the contamination at a site that allows for the uncertainty of the measurements, for either laboratory or field methods; see Reference [52].

— Improving the confidence, robustness and transparency of decisions based on the information from a site investigation.

Methods to estimate the systematic effects of sampling exist (see Reference [38]) but are not well established and are generally ignored when assessing sampling uncertainty. In contrast methods are well established for estimating systematic effects of analysis.

The duplicate method for estimating the random sampling uncertainty involves the collection of duplicate samples at a number of sampling locations (called sampling targets) across a site; see Reference [38]. A minimum of eight duplicate samples is recommended to ensure that the uncertainty estimates are suitably robust. For larger investigations, duplicates are taken at 10 % of the targets, selected at random. The duplicate samples are taken by a fresh interpretation of the sampling protocol, and are not simply two splits of one sample. Two test portions are then taken at the laboratory from both duplicate samples, prepared separately, to give four test materials for analysis. This scheme is illustrated in Figure C.1.

The analytical data from the four samples are then used to estimate sampling uncertainty by a technique called “analysis of variance”. Traditional classical ANOVA can be applied if the frequency distribution is broadly Gaussian, but robust ANOVA is more effective to quantify the underlying uncertainty if there up to 10 % of outlying values. If the distribution is more heavily skewed, then transformation of the raw data (e.g. using logarithms) is advisable before the application of classical ANOVA. Further details on how to estimate sampling uncertainty are provided in the example in C.2 (from Reference [38], Example A2), and six worked examples are given in Reference [30]. The uncertainty from the sample preparation can be estimated separately using a modified sampling design.

Estimating sampling uncertainty using the duplicate method might be particularly appropriate when:

a) the analytical results are close to the critical level of interest and the total uncertainty of measurement therefore needs to be explicit and allowed for in interpretation;

b) the ground is expected to be highly heterogeneous and cluster sampling could be required to modify the uncertainty and thereby achieve fitness-for-purpose, see Reference [30].

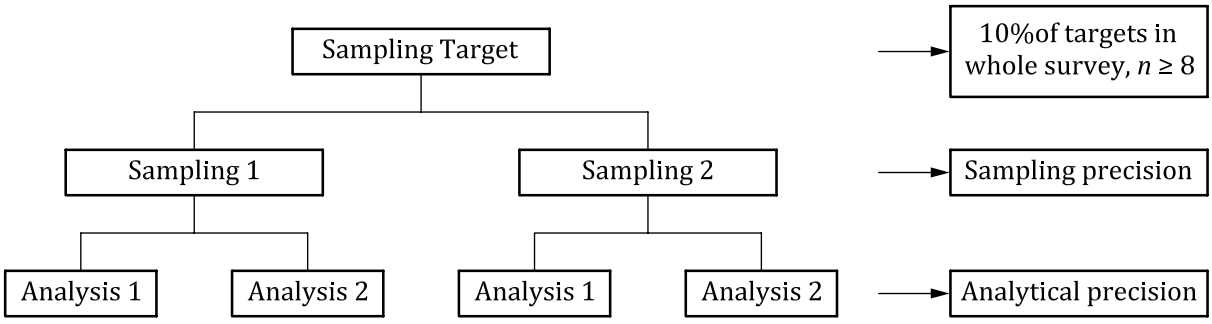


Figure C.1 — Duplicate method sampling design

C.2 Example of estimation of uncertainty and improvement in fitness-for-purpose

Lead concentrations (Table C.1) were measured for 10 randomly selected sampling targets, out of a total of 100, using the balanced design (Figure C.1), to estimate measurement uncertainty for the investigation of a 9 ha site using a regular grid, see Reference [38]. The standard uncertainty was estimated, using robust ANOVA, from the analytical repeatability standard deviation (s_{anal} = 11,1 mg/kg), sampling (s_{samp} = 123,8 mg/kg) and measurement ($s_{meas} = \sqrt{[s_{samp}^2 + s_{anal}^2]}$ = 124,3 mg/kg). The random component of the expanded measurement uncertainty, relative to the robust mean (297,3 mg/kg) at 95 % confidence, was therefore U_{meas} = 200 * 124,3/297,3 = 83,6 %. When the estimated analytical bias of the measurements (−3,4 %) was included, this estimate increased marginally to 83,9 %. These results reveal that 99,2 % of the uncertainty arises from the procedures of sampling (including sample preparation), and only 0,8 % from the chemical analysis. Reducing the uncertainty appreciably would therefore only be possible by improving the sampling.

This same procedure was applied to routine investigations of six sites with a range of different contaminates, areas, and former and proposed land uses, see Reference [30] The expanded measurement uncertainty ranged from 25 % to 158 % across the six sites, with 64 % to 96 % of that uncertainty arising from the sampling process.

Table C.1 — Measurement of Pb concentration (mg/kg) in 10 duplicated samples for first example, taken in the design from Figure C.1

Sample 1 Analysis 1	Sample 1 Analysis 2	Sample 2 Analysis 1	Sample 2 Analysis 2
787	769	811	780
338	327	651	563
289	297	211	204
662	702	238	246
229	215	208	218
346	374	525	520
324	321	77	73
56	61	116	120
189	189	176	168
61	61	91	119

NOTE The generally close agreement between the analytical duplicates for one sample, but poorer agreement between results for the duplicate samples for one target, shows qualitatively that the sampling is the larger source of uncertainty in the measurement results.

A further procedure (the Optimised Contaminated Land Investigation, OCLI method, see Reference [39]) was applied to decide whether the estimated uncertainty was acceptable [i.e. whether the measurements

were fit for their particular purpose (FFP) and the samples therefore sufficiently representative]. This technique judges FFP to be achieved at the minimum overall cost (expressed as "expectation of loss"), considering not just the cost of the investigation but also of the consequences of any misclassification of the land caused by the measurement uncertainty. (Figure C.2).

The function that is minimized in the OCLI method is shown in Formula (C.1):

$$E(L) = C[1 - \Phi(\varepsilon_1/s_{meas})] + D/s^2_{meas} \quad (C.1)$$

where

E(L) is the expectation of financial loss, which is equivalent to the predicted overall cost, expressed in any currency;

s_{meas} is the measurement uncertainty, calculated by the duplicate method;

C is the consequence cost, such as potential losses resulting from misclassification, for either a "false positive" situation (e.g. cost of unnecessary remediation), or a "false negative" situation, when missed contamination is detected at a later stage leading to the cost of unexpected delays or litigation;

Φ is the standard normal cumulative distribution function (e.g. the function NORMSDIST in Excel);

ε_1 is the error limit = | T−c | in which T is the appropriate threshold value used to classify the land, and c is the contaminant concentration for which the optimization is considered. For example, for a "false positive" case this could be 1,4*T, or the average of measured concentration values that are above T, but have lower uncertainty limits below T (i.e. "probably contaminated"). Similarly for the "false negative" case, c could be set at 0,6*T or the average of the measured concentration value that are below T, but have upper uncertainty limits above T (i.e. "possibly contaminated").

D is the combined optimal cost for sampling and analysis, where $D = \left(\sqrt{A} + \sqrt{B}\right)$, and $A = L_{samp} * s^2_{samp}$ and $B = L_{anal} * s^2_{anal}$, when L_{samp} is the real cost of taking each sample and L_{anal} is the real cost of making a chemical analysis for the contaminant under consideration;

This approach can be extended to also calculate the optimal allocation of variance and costs to achieve the optimal level of uncertainty between the sampling and the chemical analysis.

The optimal sampling variance ($v'_{samp} = [s'_{samp}]^2$), and optimal analytical variance (v'_{anal}), can be derived from the optimal measurement variance (v'_{meas}).

$$v'_{samp} = v'_{meas}\left\{\sqrt{A}/\left(\sqrt{A} + \sqrt{B}\right)\right\} \quad (C.2)$$

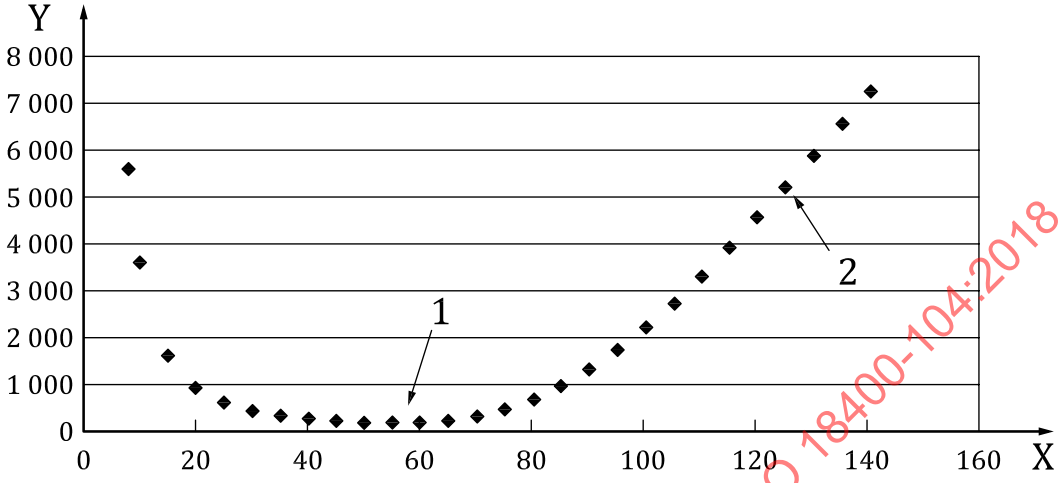
$$v'_{anal} = v'_{meas}\left\{\sqrt{B}/\left(\sqrt{A} + \sqrt{B}\right)\right\} \quad (C.3)$$

The optimal costs of sampling, and of chemical analysis, are then:

$$L_{samp} = A/v'_{samp} \quad (C.4)$$

$$L_{anal} = A/v'_{anal} \quad (C.5)$$

EXAMPLE When the OCLI method is applied to the case study above (see Reference [39]), the input values for the "false positive" scenario are:- C = £ 94 230 (for unnecessary remediation per target misclassified), ε_1 = 200 mg/kg from T = 500 mg/kg and c = 700 mg/kg (i.e. 1,4T), s_{meas} = 124,3 mg/kg (calculated above), cost per sample L_{samp} = £ 15, cost per analysis L_{anal} = £ 20.



Key

X uncertainty (s_{meas}) mg/kg Pb

Y expectation of loss (£)

1 optimal uncertainty calculated as 56 mg/kg Pb using the OCLI method

2 actual uncertainty calculated as 124 mg/kg Pb using the duplicate method

NOTE Achieving FFP is shown to require reducing the measurement uncertainty by a factor of 2,2 (i.e. 124/56), which would reduce the expectation of loss by over 97 %.

Figure C.2 — Judging the fitness-for-purpose (FFP) of a sampling procedure by applying the OCLI method to the case study (Table C.1) using Formula (C.1)

The expectation of loss, calculated from Formula (C.1) for a range of measurement uncertainty in Figure C.2 shows a "U" shape, caused by increasing costs of both measurement (e.g. sampling) at lower uncertainty, and of land misclassification at higher uncertainty. In between there is a visible minimum value that can be established either by visual inspection, or by numerical analysis using the Newton-Raphson method. This optimal value can be used to judge the fitness-for-purpose (FFP) of the whole measurement procedure, and thereby the sampling. For this case study FFP is achieved around the optimal measurement uncertainty (s'_{meas}) of 56 mg/kg. The actual uncertainty caused by the current sampling protocol, indicating a potential saving of over 97 % in this part of the whole site development. This saving could be achieved most cost-effectively by reducing s_{samp} from 124,3 mg/kg to 54 mg/kg (using Formula C.2), which would require increasing the expenditure on sampling by 427 % (Formula C.4).

Applying this OCLI method to the six other routine site investigations, see Reference [30] only one investigation had measurements judged to be FFP. In the other cases, the sampling uncertainty needed to be reduced by a large factor (e.g. from 4 to 9) to reduce the uncertainty, and thereby to achieve FFP. In one case the sampling protocol was modified in a supplementary phase of the investigation in order to reduce the sampling uncertainty, by increasing the number of increments in each composite/cluster sample to better approach FFP. The use of fourfold cluster samples to increase the sample mass reduced the sampling uncertainty by 2,6-fold and reduced the overall expectation of loss substantially (i.e. by >80 %). The reduction in sampling uncertainty is close to the twofold change, predicted by sampling theory, in which sampling variance is inversely proportional to sample mass (i.e. a √4 reduction for a fourfold cluster, and fourfold increased sample mass, as predicted by Formula F.5). One main benefit of this approach is that it can provide quantitative evidence of the need, and the benefits, of taking larger primary soil samples during site investigations.

Annex D
(informative)

Examples of sampling for particular purposes

D.1 General

The sampling required is determined by the purpose of the sampling exercise and the sampling objectives. Defining the sampling objectives results in other essential elements of the sampling plan also being defined. In this annex, examples are given of how to derive the sampling objectives (the detailed definition of the technical aspects necessary for defining the sampling plan) in subsequent steps from the purpose of sampling (the reason why the population is sampled). Typical sampling objectives include definition of the:

— population to be sampled (the population, e.g. the above-ground deposit, the site);

— components to be determined and/or tests to be carried out on the samples;

— statistical parameter to be determined (e.g. mean concentration, degree of heterogeneity, percentile);

— type of sampling (probabilistic or judgemental);

— scale of sampling (the use of increments and composite samples or spot samples and the scale on which the soil should be tested);

— desired precision and confidence.

The three examples provided in D2, D.3 and D.4 all refer to stockpiles but the principles underlying them can equally be applied to *in situ* materials. For example, the approach described in example 3 (D.4) could be adopted to characterize excavated materials prior to treatment or disposal to ensure that each defined volume of soil ("unit") complies with the specification that has been set.

D.2 Example 1: Determination of the compliance of a soil stockpile with national limit values for re-usability

D.2.1 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 (i.e. below) 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 disposed of as waste.

D.2.2 Definition of sampling objectives

Testing is required to determine compliance with specific reference conditions (e.g. limits set by legislation).

The following sampling objectives are defined:

— definition of the stockpile:

  — the stockpile is known, as this is the material that has to be tested prior to re-use;

— definition of the components and tests:

  — the components to be tested are given in legislation: 8 (heavy) metals, sum of 10 specified polycyclic aromatic hydrocarbons (PAHs), mineral oil and extractable organic halides (EOX);

  — the two composite samples are to be analysed after thorough sample pretreatment in the laboratory;

— definition of the statistical parameter:

  — the mean concentration of the stockpile has to comply with the reference levels given in legislation;

— definition of the type of sampling:

  — probabilistic sampling;

    — the number of composite samples (2) and increments (50) forming each composite sample is defined in legislation, i.e. 2 × 50 increments, to be taken at random locations throughout the soil stockpile;

— definition of the scale of sampling:

  — the scale of interest is the size of the whole stockpile (see E.2.1 and E.2.3);

— definition of the desired precision and confidence:

  — the true mean concentration should be within the 90 % confidence interval of the determined mean concentration.

Based on a large number of investigated stockpiles, it is known that the true mean for 85 % to 98 % of the stockpiles examined (depending on the components to be determined) will be within the 90 % confidence interval of the estimated mean when 100 increments are taken.

NOTE 1 When *n* composite samples are available, each made of *m* increments selected randomly throughout the stockpile (which is supposed to be the case here), and when the mean concentration over the site is estimated by taking the average of the *n* composite samples, the estimation variance of the mean can be approximated by the empirical variance of the *n* composite samples divided by *n*-1 (the approximation is better when *n* is larger). Note that this result is apparently not dependent on *m*, but of course the empirical variance depends on *m*. As a consequence, assuming a normal distribution for the estimation error, the 90 % confidence interval can be obtained from several composite samples (all at the scale of the stockpile) without the need of a more general characterization of spatial variability, which would require many spot samples.

NOTE 2 The example sampling method described is used in Dutch legislation, and has been 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 in Table D.1.

Table D.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 %

D.3 Example 2: Verification of physical characteristics of supplied soil

D.3.1 Purpose of sampling

In this example, the soil delivered to a client appears to consist of a different soil type than that which was ordered (e.g. peat instead of clay).

D.3.2 Definition of sampling objectives

Testing comprises a "quick check" to establish consistency with soil characteristics for clay (e.g. percentage fines, dry matter and organic matter content).

The following sampling objectives are defined:

— definition of the stockpile:

  — the material as delivered at the farm;

  — 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;

  — therefore, the stockpile should not be sampled in this "zone";

— 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;

— definition of the statistical parameter:

  — the concentration or other measured property for a composite sample obtained from different locations throughout the soil stockpile;

— definition of the type of sampling:

  — simple random sampling at a limited number of locations (e.g. 10) throughout the soil stockpile will be sufficient;

  — full probabilistic sampling will not be necessary (for example in the centre of the stockpile) as long as there is no tendency to biased sampling;

— definition of the scale of sampling:

  — there is now no real need for a predefined 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), however, see Note 1;

— definition of the desired precision and confidence:

  — limited precision and confidence required; these are not to be quantified.

NOTE 1 According to Annex E, the scale of sampling is the whole stockpile (or a subpopulation of the stockpile if sampling is not carried out in the centre of the stockpile), even if the mass of the composite sample is 2 kg.

NOTE 2 If the failure to supply the correct soil type becomes a contractual issue that is contested by the supplier, it might be necessary to carry out further sampling, e.g. to take further composite samples, to take a number of spot samples, or to sub-divide the stockpile and to take composite samples from each sub-division.

D.4 Example 3: Detailed characterization of a stockpiled soil

D.4.1 Purpose of sampling

This example deals with the detailed characterization 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, 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 (essentially at a smaller scale).

D.4.2 Definition of sampling objectives

The testing required is to enable a comprehensive characterization of the soil at the requisite scale and consists of a thorough determination of the behaviour and properties of interest of the material. The following sampling objectives are defined:

— definition of the stockpile:

  — the stockpile is a predefined heap of soil material already available at the laboratory that will conduct the planned experiments;

— definition of the components and tests:

  — as the experiments involve the plant uptake of heavy metals, the concentrations of the heavy metals are to be determined in order to know the original concentrations in the soil;

  — other factors that might affect uptake (e.g. pH, soil organic matter, clay fraction) are also to be determined;

— definition of the statistical parameter:

  — 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);

  — in order to determine the variability on this scale, the mean concentration in a 1 m^3 unit has to be determined, as do the mean values of the other relevant parameters;

— definition of the type of sampling:

  — for a thorough quality check of the soil, a probabilistic sampling approach is essential;

— definition of the scale of sampling:

  — as mentioned, the laboratory experiments will be conducted at a 1 m^3 scale;

  — the individual experiments need the same soil quality (for heavy metals and other relevant parameters) on this 1 m^3 scale;

  — therefore, the variability of the soil has to be determined on a 1 m^3 scale;

  — this implies that for each potential part of 1 m^3, the mean concentration or value should be known to lie within a specified bandwidth;

— definition of the desired precision and confidence:

  — a reliable estimate has to be obtained for the mean concentration and values of other relevant characteristics within a soil volume of 1 m^3;

— 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 vertical spot samples of 1 m each. This results in 30 × 3 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 vertical spot samples are 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.

NOTE Analysing the individual spot samples enables a check that the concentrations of the three samples of a chosen unit are not too dispersed. Otherwise, for the same analytical cost it would be better to group the three spot samples of a unit in a composite sample and start with the analysis of 15 composite samples, that is, 15 units.

Annex E
(informative)

Scale of sampling

E.1 Spatial variability and scale

Scale is an important concept, as heterogeneity is a scale-dependent characteristic: soil properties show less heterogeneity with large-scale measurements than with small-scale measurements.

EXAMPLE 1 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.

Two main scales should be considered: the scale of the samples, and the scale of the assessment. These scales usually do not coincide. For example, spot samples of 200 g are collected but the interest lies in the assessment of units of one cubic metre. The properties at the scale of these units can be obtained by averaging properties of several spot samples taken from within each unit or, in the framework of geostatistics, by kriging from spot data (which delivers both an estimate and an estimation variance for each unit). This requires chemical analysis (or physical testing as appropriate) of every spot sample. If this analysis is expensive, a similar result can be obtained directly with composite samples representing the unit scale (with the usual advantages and limitations of composite samples). This annex is devoted to the application of such composite samples.

Although the examples and the discussion below all refer to sampling stockpiles, the same principles can be applied to in-ground sampling, for example when the quality of the soil in a field is to be determined. Indeed, as all the examples are based on random sampling (in the sense that the individual increments making up the composite samples are collected at random locations), which is truly difficult to achieve for a stockpile or otherwise in three dimensions, these principles are easier to apply in two dimensions.

EXAMPLE 2 An example of a policy-defined scale of sampling can 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 contaminants in this soil volume. Thus, when assessing the seriousness of contaminated soil, it is the mean concentration within this volume of 25 m^3 that is most relevant. 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 to form a single composite sample. An estimate of the true mean concentration on the scale of 25 m^3 can thus be obtained.

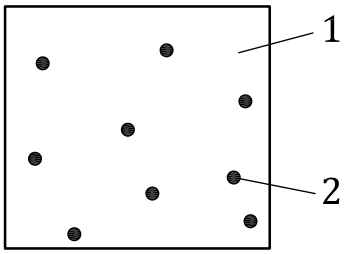
E.2 Three specific situations for which the scale is defined

E.2.1 Situation 1

Situation 1, as shown in Figure E.1, describes a soil stockpile of 2 000 t from which randomly 50 increments of 200 g are taken [the effect of fundamental variability is assumed to be negligible at this scale (see 5.6 and Annex A)]. 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, i.e. the one composite sample represents 2 000 t of soil.

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 soil stockpile from which population 2 000 t

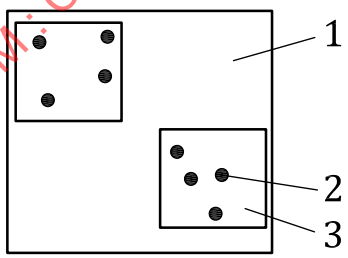
2 increment 200 g (50 increments in a composite sample of 10 kg)

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

E.2.2 Situation 2

Situation 2, as shown in Figure E.2, describes a stockpile of 2 000 t. Within this stockpile, perhaps only for the purpose of sampling, 40 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, i.e. 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 soil stockpile from which population 2 000 t

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

3 subpopulation 50 t

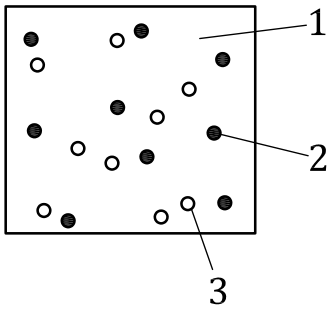
Figure E.2 — Situation 2: Soil stockpile of 2 000 t with 50 increments from each subpopulation

E.2.3 Situation 3

Situation 3, as shown in Figure E.3, is 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, i.e. 2 000 t.

The scale for each composite sample in this example is 2 000 t. If the results for several composite samples composed in the same way are available, their variance suitably scaled, gives an approximation to the variance of 200 g spot samples in the stockpile but they do not give access to the detailed spatial variability represented by a variogram.

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 soil stockpile from which 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 E.3 — Situation 3: Soil stockpile of 2 000 t with more than one composite sample taken

NOTE In these three situations random sampling has been considered for simplicity (which is of course difficult to do in three dimensions in many situations). As usual, stratified random sampling and in general regular sampling provide better estimates.

E.3 Effects of different definitions of the scale on sampling results

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

Consider within a stockpile the three subpopulations as shown in Table E.1 (comparable to situation 2 as described in E.2.2). Each subpopulation consists of 13 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 E.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, this results in 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 (CVs = 33,3/84,2 and 33,2 %). Comparing the subpopulations in Table E.1 now still gives the same result for the mean of the whole subpopulation, but additionally, it becomes clear that subpopulation 2 has a higher degree of variability than subpopulations 1 and 3.

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

Table E.1 — Example of three different subpopulations, characterized on the individual samples, the mean and coefficient of variation (CV) of the contaminant concentrations[a]

Statistical parameter	Subpopulation 1	Subpopulation 2	Subpopulation 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 %	

[a] A high CV indicates a heterogeneous sample.

E.4 Choices about the scale of sampling

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

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

   — For each defined (sub)population, a number of individual 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. Furthermore, spatial variability can be studied by means of geostatistical tools such as the variogram. 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 (if that is indeed the case).

   — For example, it can be argued that subpopulation 2 in Table E.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;

   — Many subpopulations of high heterogeneity could lead to a re-evaluation of the sampling plan. An important disadvantage is the cost for measuring the individual samples, in this example, 13 per subpopulation.

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

   — No information on individual samples within a subpopulation is gathered.

   — 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 E.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.

— An important advantage of this approach is the low costs for analysis and testing. An important disadvantage is the assumption that a composite sample can be obtained without a considerable sampling error.

— The analysis of a composite sample might pose problems as the amount of material in the sample will be (much) larger than the amount of material needed for the analysis and thus proper sample 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.

— In the example given in Table E.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.

— An important advantage is the (very) low costs for analysis and testing, 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.

— In the case of a heterogeneous stockpile (e.g. subpopulation 2 in Table E.1), sampling on the scale of subpopulations or individual samples would have given the involved parties information that might 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 of 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.

Annex F
(informative)

Determination of size and number of samples and increments

F.1 General

This annex provides methods for the determination of the size and number of samples and/or increments when the material is particulate or granular and the constituent of interest occurs in separate solid particles. These methods require specific knowledge about the particles that constitute the material to be sampled.

For a good understanding of this annex, the distinction between the terms "increment", "sample" and "composite sample" is essential (see 6.4.5). An increment is the amount of material obtained by a single operation of a sampling device, for instance, the filling of a scoop or auger. It is, per definition, put together with other increments in a composite sample. Conversely, a spot sample is made of a single increment or a number of contiguous increments. The term "sample" can either be used for a spot sample or a composite sample.

F.2 Background to the determination of the minimum increment size

In order to achieve unbiased sampling, it is essential that every particle in the target to be sampled (site, stratum, zone, 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 5 cm when the diameter of the auger is only 3 cm. Also, when the diameter of the auger is little over 5 cm, 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.

There is, in general, a wide particle size distribution. It is therefore assumed in practical applications that the opening of the sampling device is large enough when it is equal to three times the 95th percentile of the distribution of particle sizes. This 95th percentile is known as D_{95} and is called the "maximum particle size" for simplicity. 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.

The consequences for the size of the sampling equipment required depend on the governing dimensions of the sampling equipment used. 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 D_{95}. (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 the front size, the height and the depth of the scoop would all have to be over three times D_{95}.

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.

The minimum increment size depends on its physical background; therefore, the dimensions can also be defined in terms of volume or weight of the increment. The volume of the minimum increment size can be easily defined as $(3D_{95})^3 = 27(D_{95})^3$. See Formula (F.1):

$$V_{\text{min}} = (3D_{95})^3 = 27(D_{95})^3 \quad \text{(F.1)}$$

where

$V_{\text{min. increment}}$ is the minimal volume of the increment, in cm^3;

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

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. The minimum mass of the increment is defined according to Formula (F.2):

$$m_{\text{min. increment}} = 27\ (D_{95})^3\ \rho_{\text{bulk}} \quad \text{(F.2)}$$

where

$m_{\text{min. increment}}$ is the minimal mass of the increment, in g;

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

ρ_{bulk} is the bulk density of the material, in g/cm^3.

For practical reasons an increment should nevertheless have a mass of at least 200 g. When Formula (F.2) leads to a minimum mass less than 200 g (for a bulk density of 2 g/cm^3, for example, this is the case when D_{95} is less than 1,6 cm), [Formula (F.2) should be replaced by Formula (F.3)]:

$$m_{\text{min. increment}} = 200\ \text{g} \quad \text{(F.3)}$$

Note that this applies to sampling in the field. Smaller increments or samples can be defined in laboratory conditions.

F.3 Background to determination of the minimum sample size

The characteristic of interest of the material to be sampled is, for example, a metal grade or a constituent concentration. In a particulate material, the metal or constituent is borne by a fraction of the particles. Moreover, the particles possess different shapes, sizes, densities (sometimes termed "volumetric mass densities" to distinguish them from "bulk densities" as used in F.2) and chemical compositions, and therefore vary from each other in a way that is specific for that material. In consequence, the material always displays a certain degree of variability at the scale of the particles. This fundamental variability results in what is known as the "fundamental error" in sampling (note that small scale heterogeneity also affects sampling uncertainty). In order to limit this undesired degree of variability to an acceptable level, the samples should at least contain such an amount of particles that the variability between the particles has no significant influence on the mean grade or concentration of the sample. To ease the presentation, the particles that bear the metal or constituent of interest will be named mineral particles, and the other particles will be named matrix particles.

A simple and basic situation is a batch of material made of a very large number of particles that are identical except that a proportion w of them are mineral particles while the others are matrix particles. If n particles in that batch are selected, the observed proportion w_{sample} of mineral particles will depend on the n particles selected. The sampling error attached to the selection is the difference between w_{sample} and w. It is called fundamental error because it is inherent to the material and exists even if

the sampling operation is perfectly carried out. Binomial sampling theory shows that the observed proportion will be on average equal to *w*, and that the sampling error has a variance

$$Var\left[w_{\text{sample}}\right]=\frac{(1-w)w}{n}$$

Said differently, w_{sample} is an unbiased estimate of *w*, with a relative sampling variance

$$\sigma_{rel}^2=\frac{Var\left[w_{\text{sample}}\right]}{w^2}=\frac{1-w}{nw}$$

This variance is inversely proportional to *n*. As a consequence, the sample should have a minimum size to be regarded as sufficiently representative of the material.

Statistical sampling theory generalizes this result to more realistic materials, where particles vary in shape and size, possibly with different densities for mineral particles and matrix particles. Further, the assumptions are made that the mass of the batch is several orders of magnitude larger than the sample mass and that the material has been crushed below the liberation size.

Particles are randomly selected until a sample of mass M_{sample} is obtained. The sampling process is unbiased and the magnitude of the fundamental error can be characterized by its relative error variance given in Formula (F.4):

$$\sigma_{\text{rel}}^2=\frac{Var\left[w_{\text{sample}}\right]}{w^2} \tag{F.4}$$

Equivalently, σ_{rel} is the coefficient of variation of the fundamental error.

where

$Var[w_{\text{sample}}]$ is the variance;

W is the mass fraction of mineral particles in the batch (dimensionless).

This relative variance is given by Gy's formula [Formula (F.5)]:

$$\sigma_{\text{rel}}^2=\frac{1}{M_{\text{sample}}}fg\frac{1-w}{w}\left[(1-w)\rho_{\text{mineral}}+w\rho_{\text{matrix}}\right](D_{95})^3 \tag{F.5}$$

Simple calculations show that this is equivalent to Formula (F.6):

$$\sigma_{\text{rel}}^2=\frac{1}{M_{\text{sample}}}fg\frac{1-w}{w}\frac{\rho_{\text{mineral}}\rho_{\text{matrix}}}{\rho}(D_{95})^3 \tag{F.6}$$

with [Formula (F.7)]:

$$\rho=w\rho_{\text{mineral}}+(1-w)\rho_{\text{matrix}} \tag{F.7}$$

where

M_{sample} is the mass of the sample, expressed in g;

f is the particle shape factor (dimensionless);

g is the factor of granulometric dispersion (dimensionless);

w is the mass fraction of mineral particles in the batch (dimensionless);

$\rho_{mineral}$ is the density of the particles of mineral, in g/cm^3;

ρ_{matrix} is the density of the particles of the matrix, in g/cm^3;

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

The shape factor f is equal to 1 for cubic particles, to π/6 for spherical particles, and to 0,1 for needles. In practice, the value 0,5 is selected, close to π/6.

The granulometric dispersion factor g depends on the spread of the distribution of particle size as follows:

— broad distribution: $D_{95}/D_{05} > 4$, $g = 0{,}25$;

— medium distribution: $2 < D_{95}/D_{05} \leq 4$, $g = 0{,}50$;

— narrow distribution: $1 < D_{95}/D_{05} \leq 2$, $g = 0{,}75$;

— uniform particles: $D_{95}/D_{05} = 1$, $g = 1{,}00$.

where

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

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

When the particles have not been calibrated, the value $g = 0{,}25$ is used.

In the applications considered here, the proportion of particles of interest is low, so that ρ is close to ρ_{matrix}. Formulae (F.5) and (F.6) therefore amount to (with $f = 0{,}5$):

$$\sigma_{rel}^2 = \frac{1}{2M_{sample}} g \frac{1-w}{w} \rho_{mineral} \left(D_{95}\right)^3 \quad \text{(F.8)}$$

where

g is the factor of granulometric dispersion (dimensionless);

w is the mass fraction of mineral particles in the batch (dimensionless);

M_{sample} is the mass of the sample, expressed in g;

$\rho_{mineral}$ is the density of the particles of mineral, in g/cm^3;

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

When designing the sampling strategy, a value for the coefficient of variation σ_{rel} is selected. From Formula (F.8), the minimum sample mass ensuring that σ_{rel} does not exceed the selected value is shown in Formula (F.9):

$$M_{min.sample} = \frac{1}{2\sigma_{rel}^2} g \frac{1-w}{w} \rho_{mineral} \left(D_{95}\right)^3 \quad \text{(F.9)}$$

where

$M_{min.sample}$ is the minimum mass of the sample, expressed in g.

NOTE 1 In this clause, the variables in the formulae are expressed in CGS units for practical reasons (the formulae are also true in SI units).

NOTE 2 The coefficient of variation of the above formulae is that of the mass fraction of particles with a certain characteristic in the sample. When the characteristic is a fixed percentage of these particles (such as the concentration of a mineral particle in some chemical element), it is also the coefficient of variation of the average concentration of the sample.

NOTE 3 For a fixed sample mass, the magnitude of the "fundamental" error only depends on intrinsic characteristics of the material and cannot be reduced without a physical processing of the material (in practice, grinding), hence this denomination.

NOTE 4 Gy's formula (see References [28][29]) was derived for piles of particulate material. It can be applied at any stage of sampling, from samples to be taken in a stockpile up to the preparation of samples of several grams for chemical analysis. It can also be applied to auger or core samples taken in an industrial site; in such a case, it represents the variations in the grade or concentration of the sample when the location of the sample is slightly changed (e.g. by one or several decimetres).

NOTE 5 Gy's formula (see References [30][31]), as presented here, assumes contamination concentrations (or other properties) are the same for all particle sizes. Errors arise when there are marked disparity in concentrations in particles of different sizes. Experiments have shown that Gy's formula is not always accurate, notably in the case of gold deposits, where it can lead to the specification of unrealistically large samples. In contrast, other sources of error, such as a segregation of the particles of interest, are not quantified. Alternatives to Gy's formula exist.

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

F.4.1 General

Formula (F.9) is valid in ideal sampling conditions that are hardly met in practice. It depends on a certain number of parameters of the granular material that are imperfectly known. Formula (F.9) should therefore be considered as a rough estimate of the minimum sample size that should be used in order to prohibit inaccurate sampling results due to the fundamental heterogeneity of the material. It is however advisable to include duplicate samples in the sampling plan, in order to experimentally obtain the magnitude of the total measurement error, which also includes the pretreatment and analytical errors.

Hereafter, the main requirements for the use of Formula (F.9) are given, even though some of them apply only partially for soil sampling.

F.4.2 Particle size distribution, factors D_{05}, D_{95} and g

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}). Note that these percentages represent mass fractions.

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, moreover with a power of 3. In F.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 $g = 0{,}25$. Moreover, the maximum particle size (D_{95}) is for most soils 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.

F.4.3 Density of the particle

For the density $\rho_{mineral}$ of the "mineral" particles (those possessing the characteristic of interest) the density of the individual particles should be used, not the density of the sample or the (loosely) packed material.

F.4.4 Fraction of the particles with the characteristic to be determined, factor *w*

The actual value of *w*, the mass fraction of "mineral" particles (those possessing the characteristic of interest), depends on the soil to be sampled and the substances to be determined in that soil. If the mineral particles are pure metal (e.g. copper particles in a silica particle matrix), *w* coincides with the metal grade. If the constituent of interest is only a fixed part $\alpha < 1$ of the mineral particles, *w* is the ratio of the average concentration to α. Practical situations are usually more complex. As the information for a good estimation of *w* will often not be available, the value of 0,1 is most commonly used. For soil sampling, a "safer" choice of *w* is 0,02. Note the strong influence of a low value of *w* on Formula (G.9), because it occurs at the denominator of the formula.

If the concern is a contaminant occurring in a very low proportion of the particles (e.g. copper wires in a soil), Formula (G.9), applied for example with g = 0,25, $\rho_{mineral}$ = 2g/cm^3, and D_{95} = 1,6 cm, gives $M_{min.sample} = 1/\left(w\sigma_{rel}^2\right)$. If *w* is equal to 10^{-6} (1 ppm) and even accepting an uncertainty as large as σ_{rel} = 1 for the sample, $M_{min.sample}$ should be equal to 10^6 g, that is one tonne.

F.4.5 Coefficient of variation from the fundamental error, factor σ_{rel}

A choice has to be made about the amount of fundamental variation that is thought to be acceptable in relation to the quality of the sampling. Principally, this choice cannot 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, which would require a very large sample and 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.

There could be circumstances where a larger value for σ_{rel} could be accepted, for example when the objective is to show that the average concentration is several orders of magnitude below a given concentration threshold.

F.4.6 Commonly used assumptions

The following assumptions can be used for soils in common situations:

$\rho_{mineral}$ = 2,6 g/cm^3 (the particle density for silicon oxide);

D_{95} = 1,6 cm;

g = 0,25;

w = 0,02;

σ_{rel} = 0,1.

They lead to a minimum sample mass of 26 kg. Note the strong impact of moderate changes of D_{95} (because it arises with a power of 3) and *w* (because it is at the denominator). However, the assumptions may only be used after careful study of the backgrounds of the formula for the minimum sample size.

F.5 Determination of the maximum particle size, factor D_{95}

F.5.1 General

The minimum increment mass and minimum sample mass given by Formulae (F.2) and (F.9) depend heavily on D_{95} (which appears with a power of 3). It is therefore important to evaluate D_{95} as accurately as possible before starting the sampling itself. This is done with a unique sample solely dedicated to the determination of D_{95} (or more generally of the particle size distribution of the soil). The mass of this sample, which should be obtained with proper regard to the need for a sufficiently representative sample, is obviously larger than the minimum increment mass given by Formula (F.2). This is a two-step procedure, with possible iterations, as described hereafter.

F.5.2 Step 1: Sampling

Determine the mass of the necessary sample by applying Formula (F.10):

$$m = 150\left(D_{\text{eval.95}}\right)^3 \rho_{\text{bulk}} \tag{F.10}$$

where

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

ρ_{bulk} is the bulk density of the material, in g/cm^3;

$D_{\text{eval. 95}}$ is a preliminary evaluation of the maximum particle size, in cm.

Note that $D_{\text{eval. 95}}$ should be taken by excess in order to ensure that the sample is representative.

Take a sample of this mass from the stockpile or the domain of interest with a sampler that can accommodate all particle sizes or 1 000 g, whichever is the greater.

Weigh the sample (m_0).

F.5.3 Step 2: Sieving the sample

Transfer the entire sample on a sieve with a sieve aperture D equal or close to the estimated $D_{\text{eval. 95}}$.

Sieve the sample either manually or mechanically.

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.

Weigh the part of the sample that remains on the sieve (m_1).

The percentage of particles with a size less than D is calculated according to Formula (F.11):

$$p = 100\left(1 - \frac{m_1}{m_0}\right)\% \tag{F.11}$$

If:

— P < 95 %, the used sieve aperture D was too small. Use the fraction m_1 for a second sieving with a larger aperture;

— P = 95 %, the used aperture D was exactly right and is used as an estimation of D_{95};

— P > 95 %, the used aperture is too big. Use the sieved part of the sample to sieve it again with a smaller aperture. (Or use the aperture size D as a conservative approximation to D_{95}).

The re-sieving procedure should be repeated until two neighbouring apertures are found with p respectively smaller and larger than 95 %. The aperture leading to P = 95 % is then obtained by linear interpolation.

F.6 Calculation of the actual increment and sample size

F.6.1 Spot sampling

The sample amounts to a single increment. The actual sample size should be larger than the values given by Formulae (F.2), (F.5) and (F.6).

F.6.2 Composite sampling

Where composite sampling is to be undertaken, there is a possible conflict between:

a) the previously calculated values for increment size and sample size;

b) the planned number of increments, m (Annex G).

Such conflict should be resolved as follows:

— if m increments of minimum size amount to less than the minimum sample size, then the increment size should be increased so that m actual increments will produce an adequately large composite sample;

— conversely, if m increments of minimum size amount to more than the required minimum sample size, then this larger quantity defines the actual sample size.

In the latter case, the actual sample size will be larger than the minimum required, so that the resulting confidence will be better than asked for.

Annex G
(informative)

Statistical methods for estimating soil parameters

G.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 annex is divided into two main clauses:

— assuming that a certain number of samples are available, G.2 provides expressions for the estimate as well as the uncertainty associated with the estimate, for each of a number of commonly used statistical parameters;

— because the uncertainty about a statistical parameter of interest might be presently too large, G.3 gives methods for defining the total number of samples needed to achieve a desired precision for the estimation of this parameter.

These results are based on the assumption that the samples are independent (or at least uncorrelated). This is achieved when the property of interest displays no spatial correlation, or when a simple random sampling pattern is used. This is, however, not a reason for adopting a simple random sampling pattern when spatial correlations exist. Indeed, the estimates proposed hereafter will be more precise in the case of a stratified random pattern, and usually even more so in the case of a regular pattern. The formulae in G.2 for the uncertainty associated with these estimates should then be considered as upper bounds for the true uncertainty. Conversely, methods developed in G.3 for defining the number of samples are conservative: they should be considered as upper bounds for the required number of samples. Exact equations are given by the geostatistical methods described in Annex H.

G.2 Estimation of statistical parameters from *n* available samples

G.2.1 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;

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;

$\bar{x}$ is the sample mean;

σ is the population standard deviation;

s is the estimated standard deviation;

u_p is the standard normal deviate corresponding to cumulative probability p;

X_p^2 is the chi-squared deviate with n-1 degrees of freedom corresponding to cumulative probability p;

X_P is the population Pth percentile;

$\hat{X}_p$ is the estimated Pth percentile;

$e(z)$ is the standard error of the statistic z (in practice $\bar{x}$ or $\hat{X}_p$);

$b(r;n,p)$ 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;

$B(r;n,p)$ 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.

The mathematical expression of $b(r;n,p)$ is shown in Formula (G.1):

$$b(r;n,p)=\frac{n!}{(n-r)!r!}p^r(1-p)^{n-r} \quad \text{(G.1)}$$

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.

G.2.2 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 Formula (G.2):

$$\bar{x}=\frac{\sum x_i}{n} \quad \text{(G.2)}$$

The uncertainty is given by Formula (G.3):

$$e(\bar{x})=\frac{s}{\sqrt{n}} \quad \text{(G.3)}$$

G.2.3 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 Formula (G.4):

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

For normal populations, the uncertainty in s can be assessed using the chi-squared distribution with $n-1$ degrees of freedom. A C % confidence interval for σ given s can be calculated as Formula (G.5):

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

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

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 units 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.

G.2.4 Coefficient of variation

The variability of a population of non-negative values 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 Formula (G.6):

$$\text{CV} = \frac{s}{\bar{x}} \tag{G.6}$$

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.

G.2.5 Percentiles

G.2.5.1 General

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

EXAMPLE The 90th 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.

G.2.5.2 Percentiles assuming normality

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

where p is P/100.

Standard normal deviates, u_p, for various values of p are given in Table G.1:

Table G.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 The 95th percentile is $\mu + 1{,}645\sigma$, and the 1st-percentile is $\mu - 2{,}326\sigma$.

An (almost) unbiased estimate of the Pth percentile is given by Formula (G.7):

$$\hat{X}_{\mathrm{p}} = \bar{x} + u_p s \tag{G.7}$$

where

p is P/100;

$$e\left(X_p\right) = s\sqrt{\frac{1}{n} + \frac{{u_p}^2}{2(n-1)}}\,.$$

G.2.5.3 Percentiles assuming log-normality

Formula (G.8) applies equally to the case of log-normally distributed data, with the following adjustments:

— the mean $\bar{x}$ and the standard deviation s refer to the log-transformed data (it being immaterial whether base-10 or base-e is used);

— at the end of the calculation of $\hat{X}_{\mathrm{p}}$, the estimate of the Pth percentile should finally be antilogged to return to the unlogged domain.

G.2.5.4 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 Pth percentile is estimated as shown in Formula (G.8):

$$\hat{X}_{\mathrm{p}} = X\left(r\right) \tag{G.8}$$

where $r = \left(\frac{\mathrm{P}}{100}\right)(n+1)$

If r is not an exact integer, linear interpolation should be used as follows [see Formula (G.9)]:

$$\hat{X}_{\mathrm{p}} = (1-d)X(r') + dX(r'+1) \tag{G.9}$$

where

r' is the integer part of r;

d is $r - r'$.

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:

$$\{X(r_1) \text{ to } X(r_2)\} \quad \text{(G.10)}$$

where

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

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

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

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 80th percentile cadmium concentration from 39 random samples taken from a soil stockpile, together with a 90 % confidence interval.

Estimating the 80th percentile cadmium concentration by the Weibull method is shown as Formula (G.11):

$$\hat{X}_{80} = X(r) \quad \text{(G.11)}$$

where r is (80/100)(39 + 1) = 32.

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

With a 90 percent confidence level:

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

— $B(r_1 - 1; 39, 0,8) \leq 0,05$;

— $B(r_2 - 1; 39, 0,8) \geq 0,95$.

Using appropriate software, experimentation shows that:

— $B(26; 39, 0,8) = 0,036$;

— $B(35; 39, 0,8) = 0,967$.

Thus, the interval $X(27)$ to $X(35)$, i.e. the interval from the 13th biggest to the 5th biggest sample value, provides a conservative 90 % confidence interval for the true 80th percentile cadmium concentration. The actual confidence coefficient is 0,967 − 0,036 = 0,931, or 93 %.

G.2.6 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 objective is concerned with "worst case" values, the recommended approach is to recast the objective in terms of a suitably high percentile – say the 99th percentile. The methods described in G.2.5 can then be applied.

G.2.7 Percentage compliance with a given limit

G.2.7.1 General

The primary sampling objective 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 objectives, both parametric and non-parametric approaches can be taken. To contrast the two approaches, imagine that the limit L should be complied with for P % of the time or better.

G.2.7.2 Percentage compliance — Parametric approach

Using the parametric approach, the Pth 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 $e(\hat{X}_p)$.

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.

G.2.7.3 Percentage compliance — Non-parametric approach

By the non-parametric approach, the quantity r (the number of sample values ≤ 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 Formula (G.12)

$$[100p_{LO} \text{ to } 100p_{UP}] \tag{G.12}$$

where

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

p_{UP} is chosen so that $B(r; p_{UP}, n) = (1 - C/100)/2$.

$B(r; n, p)$ 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 document, 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) 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 % of the population at the defined scale, rather than 100 %, should comply with the limit.

G.3 Calculating the number of samples required to achieve a desired precision

G.3.1 Symbols and abbreviated terms

To complement those of G.2, this subclause uses the following symbols and abbreviated terms.

m is the number of increments per composite sample;

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 $\left(=\sqrt{\sigma_w^2+\sigma_b^2}\right)$;

σ_a is the standard deviation of analytical error;

C is the desired confidence level (%);

t is the cumulative probability related to the desired confidence level;

d_{prec} is the desired precision.

G.3.2 Estimating a mean concentration

G.3.2.1 Using composite sampling

The standard error of the mean is given by Formula (G.13):

$$e\left(\bar{x}\right)=\sqrt{\left[\frac{\left(\frac{\sigma_w^2}{m}+\sigma_b^2+\sigma_a^2\right)}{n}\right]} \tag{G.13}$$

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 Formula (G.14):

$$n=\left(\frac{u_t}{d_{prec}}\right)^2\times\left(\frac{\sigma_w^2}{m}+\sigma_b^2+\sigma_a^2\right) \tag{G.14}$$

where t is $1-(1-C/100)/2$.

Alternatively, Formula (G.13) 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:

$$m = \frac{\sigma_w^2}{\left[n\left(\frac{d_{prec}}{u_t} \right)^2 - \sigma_b^2 - \sigma_a^2 \right]} \quad \text{(G.15)}$$

NOTE It might be desirable to plan to take only a single composite sample. Provided $\sigma_b^2 + \sigma_a^2$ is sufficiently small, this can be achieved by setting n equal to 1 in Formula (G.15).

In practice, the true standard deviations are unknown and so estimates should be used. In some cases, it could 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 t for a 90-percent confidence level:

For C = 90, t = 1 − (1 − 90/100) / 2 = 0,95, and so u_t = 1,65.

Calculating n from Formula (G.14),

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

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.

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:

$$T_c = (Am + B)n \quad \text{(G.16)}$$

Thus, using Formula (G.14) with various trial values of m, it is possible to find the combination of m and n which minimizes T_c.

EXAMPLE 2 Continuing with the earlier example, suppose that:

— values of m ranging from 1 to 20 are considered; and

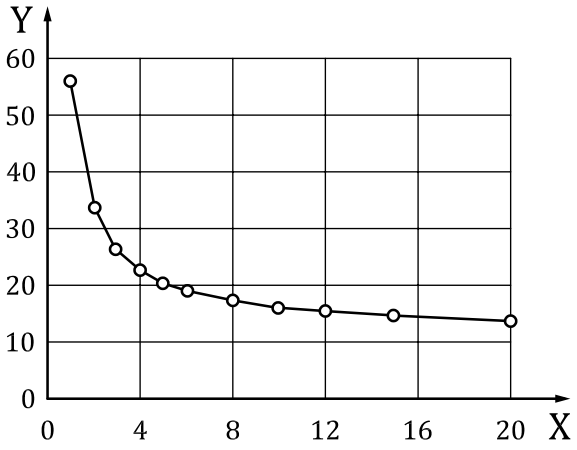
— B/A = 30 – that is, a sample analysis is 30 times more expensive than the cost of a sample increment.

Getting the n and T_c values:

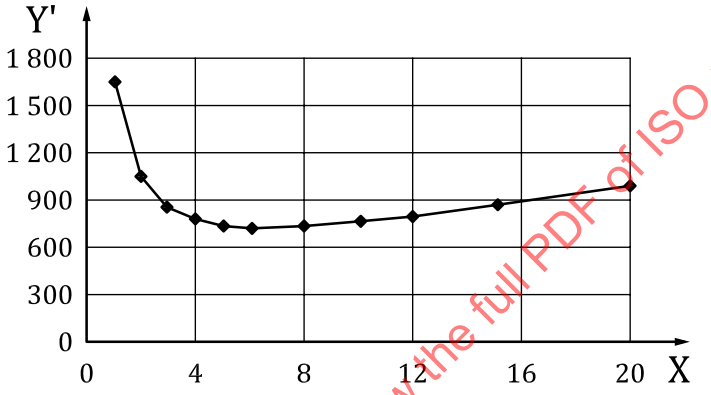
The upper panel of Figure G.1 shows the n value given by Formula (G.14) for each trial value of m.

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 G.1 — Illustration of the relationships between m, n and T_c

G.3.2.2 Using individual samples

The standard error of the mean is given by Formula (G.17):

$$e(\bar{x}) = \sqrt{\frac{\sigma_s^2 + \sigma_a^2}{n}} \qquad \text{(G.17)}$$

Thus, the number of samples required to achieve the desired precision and confidence is given approximately by Formula (G.20):

$$n = \left(\frac{u_t}{d_{\text{prec}}} \right)^2 \times \left(\sigma_s^2 + \sigma_a^2 \right) \qquad \text{(G.18)}$$

where t is 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 $\sigma_w^2 + \sigma_b^2$ by σ_s^2.

In practice, the true standard deviations are unknown and so estimates should be used. In some cases, it could 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_a are 4,5 mg/kg and 0,5 mg/kg; and

— the mean is required to be estimated to a precision of d_{prec} = 2 mg/kg with 90 % confidence.

Getting u_t:

For C = 90, t = 1 − (1 − 90/100) / 2 = 0,95, and so u_t = 1,65.

Getting the n value:

From Formula (G.20), $n = (0{,}825)^2\,(20{,}25 + 0{,}25) = 13{,}9$.

Thus:

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

G.3.3 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.

When the true dispersion variance σ^2 of the population is estimated by the sample variance s^2 defined as Formula (G.19):

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2 \tag{G.19}$$

where x_i, i = 1, …, n are the values of the property of interest for the n samples and $\bar{x}$ is the sample mean.

The ratio s^2/σ^2 has the statistical distribution of a random variable $Y/(n - 1)$ where Y follows a χ^2 distribution with $n - 1$ degrees of freedom. Confidence intervals for σ can be calculated using tables of quantiles of the χ^2 distribution, which can be found in most statistical books. 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 G.2 can be developed.

Table G.2 — 90 % confidence limits for σ /s for various numbers of samples

Number of samples n	Lower 90 % confidence limit for σ/s $\sqrt{[(n-1)/\chi^2]}$ (P = 0,95)	Upper 90 % confidence limit for σ/s $\sqrt{[(n-1)/\chi^2]}$ (P = 0,05)
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

Table G.2 *(continued)*

Number of samples n	Lower 90 % confidence limit for σ/s $\sqrt{[(n-1)/\chi^2]}$ (P = 0,95)	Upper 90 % confidence limit for σ/s $\sqrt{[(n-1)/\chi^2]}$ (P = 0,05)
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.

G.3.4 Estimating a percentile

G.3.4.1 Assuming normality

The standard error of the Pth percentile $\hat{X}_p$ is given by Formula (G.20):

$$e_s(\hat{X}_p) = \sigma\sqrt{\frac{1}{n} + \frac{u_p^{\,2}}{2(n-1)}} \tag{G.20}$$

where

p is P/100;

σ is $\sqrt{(\sigma_s^2 + \sigma_a^2)}$.

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

$$n = \left(\frac{u_t s}{d_{\text{prec}}}\right)^2 \times \left(\frac{1+u_p^2}{2}\right) \tag{G.21}$$

where

t 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 95th percentile is required to be estimated to a precision of d_{prec} = 1,46 mg/kg with 90 % confidence.

Finding u_p and u_t:

— for the 95$^{\text{th}}$ percentile, p = 0,95 and so u_p = 1,65;

— for C = 90, $t = 1 - (1 - 90/100) / 2$ = 0,95, and so u_t = 1,65.

Finding the n value:

— thus from Formula (G.21), $n = (1{,}65 \times 3{,}5/1{,}46)^2 \times (1 + 1{,}65^2/2) = 36{,}9$.

Result:

— thus, about 37 samples would be needed for the 95th percentile to be estimated to the required reliability.

G.3.4.2 Non-parametric approach

For determining the precision achievable by a non-parametric approach, there is no direct expression available corresponding to Formula (G.21) for the normal case. As a rough approximation, however, Formula (G.21) 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 G.3.5 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, see Reference [40].

EXAMPLE

The 80th percentile cadmium concentration from a particular soil stockpile is required to be estimated to a precision of d_{prec} = 15 mg/kg with 90 % confidence.

Select n = 39 as the trial number of samples.

From G.3.4, a conservative 90 % confidence interval is provided by the interval $x(27)$ to $x(35)$.

From a set of 39 cadmium concentrations taken at random from prior comparable soil stockpiles, the 15 highest values are: 12 mg/kg, 12 mg/kg, 15 mg/kg, 17 mg/kg, 20 mg/kg, 20 mg/kg, 25 mg/kg, 26 mg/kg, 31 mg/kg, 31 mg/kg, 35 mg/kg, 36 mg/kg, 40 mg/kg, 44 mg/kg, and 55 mg/kg.

— Thus, the 27th and 35th 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 G.3.4, a conservative 90 % confidence interval is provided by the interval by the interval $x(20)$ to $x(27)$;

— From a set of 29 values taken at random from the historical data, the 12 highest values are: 10 mg/kg, 12 mg/kg, 15 mg/kg, 20 mg/kg, 20 mg/kg, 25 mg/kg, 26 mg/kg, 31 mg/kg, 35 mg/kg, 40 mg/kg, 44 mg/kg, and 55 mg/kg.

— Thus, the 20th and 27th 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 80th percentile to be estimated to the required reliability.

G.3.5 Estimating a percentage compliance with a given limit

The approach here is similar to that described in G.3.4. 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 G.2.7. 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 is to be estimated to a precision of 12 % 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 G.2.7.3, calculate a 90 % confidence interval for the true compliance percentage.

— This is 59,9 % to 92,9 %, giving a precision of about 17 %.

— 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 G.2.7.3, calculate a 90 % confidence interval for the true compliance percentage.

— This is 66,8 % to 89,6 %, giving a precision of about 11,5 %.

— 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 H
(informative)

Geostatistical methods for sampling design and evaluation of soil quality

H.1 General

Contaminated sites usually show complex contamination patterns. Variables such as the concentration of contaminants, the porosity and the permeability of the medium, or the thickness of a contaminated layer exhibit spatial variations with a complexity of detail that precludes a description by deterministic models. Such variables are named "regionalized variables" to stress both their structured and random nature. Furthermore, for economic reasons, these variables are sampled very sparsely. As a consequence, decision making is always based on incomplete information and, because of spatial variability, comes with some uncertainty. To cope with that uncertainty, probabilistic methods are used.

The quantification of spatial uncertainty requires a model specifying the mechanism by which spatial randomness is generated. The simplest approach is to treat the regionalized variable as deterministic and the positions of the samples as random, assuming for example that they are selected uniformly and independently over a reference area, in which case, standard statistical rules for independent random variables apply, such as that for the variance of the mean. If the samples are collected on a regular grid, they are not independent and things become more complicated, but a theory is possible by randomizing the grid origin.

Geostatistics takes the bold step of associating randomness with the regionalized variable itself. When a die is thrown, the future result is random, in that case 1, 2, 3, 4, 5 or 6 with equal probability. But once the die has stopped, the observed result is for example 5, which is no longer random; it is called a realization of the random variable representing the die. In the case considered here, the analogue of the die is the variable of interest and a *stochastic model* is used in which the regionalized variable is regarded as one among many possible *realizations* of a random function. Normally, many realizations are needed to infer the statistical parameters of the *random function*. When a single realization is available, as is the case when considering a specific contaminated site, repeatability is achieved by introducing an assumption of stationarity: spatial variations in some part of the study domain (study area or volume) are assumed to be statistically similar to spatial variations in any other part of the domain.

The advantage of this conceptual site model is that geostatistical methods do not require randomly located samples; but they require a preliminary characterization of the spatial variability of the variable of interest. This characterization is a very useful step because it is based on an exploratory data analysis (EDA) which can show most problems affecting the data. Nevertheless, this EDA requires already having data. Otherwise, geostatistics can only be used by assuming characteristics identified for another site deemed similar to the site of interest. This is the reason why geostatistics is usually not used to design an initial or exploratory exploration, even if some general results of geostatistics give guidelines about sensible sampling patterns. Geostatistics are useful once a certain number of data are available, that is, at the stages of the main and supplementary investigations.

To answer the problem of the definition of a sampling plan at these stages, the first requirement is to know which data are already available and to make a point on how far the objectives are met with these data. Then, the questions how many supplementary samples are needed, at which locations, and with which sample characteristics to achieve the objectives, can be considered.

The specification of the sampling plan should then take place after a study of the contamination from the data already available. If this study has not been carried out at the end of the preceding stage, it should be done now. The latter situation is considered here, because it offers the opportunity to present the geostatistical tools before presenting their use for rationalizing sampling.

This presentation therefore comprises the following:

— characterization of spatial variability;

— geostatistical estimation and simulation methods;

— rationalizing a sampling plan.

Once the new sampling survey has been completed, the first two steps have to be updated or developed.

It is important to recognize before seeking to apply geostatistics the limitations to its use, such as:

— the characterization of spatial variability requires a certain number of samples: about 30 samples is usually a minimum;

— some stationarity of the behaviour of the contamination or other property of interest should be assumed. This assumption can be weakened (presence of a trend, local stationarity), but in that case, more samples are needed;

— geostatistical techniques are not applicable in the case of preferential sampling (e.g. data specially located in areas supposed to be of high contaminant concentration);

— the contamination is linked with auxiliary variables such as lithology or past use of the site and these variables have not been carefully recorded (e.g. there are layers with very different properties and the layer from which an auger sample, for example, has been taken has not been recorded);

— geostatistics provides no improvement over standard statistics if the spatial distribution of the contamination is purely random (no, or very poor, spatial correlation) – but the absence of spatial correlation can be shown by the exploratory data analysis;

— a sound use of geostatistical techniques requires proper training and more effort than a conventional study, as well as a collaboration with the specialists of the other disciplines.

H.2 Characterization of spatial variability

H.2.1 General

The characterization of spatial variability is based on an exploratory data analysis (EDA), which ends with the calculation of sample variograms and their fitting by variogram models. The objective is to understand, characterize and model spatial variability. It is also to detect and delineate subdomains that have distinct spatial characteristics and need to be studied separately. A by-product of the EDA is the detection of anomalous data (wrong and true) and thus the possibility of a validation of the data.

The variogram will be used in the subsequent steps, for example to provide a map of the contamination from past surveys (estimate and precision) and define a complementary survey adapted to the spatial characteristics of the contamination.

H.2.2 Exploratory data analysis (EDA)

H.2.2.1 General

The aim of the EDA is to understand and represent (in particular, graphically) how the contamination is structured in the site: trends, similar or contrasted values at neighbouring points, variation of concentration with depth, dependence on soil type, etc. The method consists in an interactive query of the data.

