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Soil quality — Sampling —

Part 201: **Physical pretreatment in the field**

Qualité du sol — Échantillonnage —

Partie 201: Prétraitement physique sur le terrain

Reference number
ISO 18400-201:2017(E)

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Foreword

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

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 on 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 the following URL: www.iso.org/iso/foreword.html

This document was prepared by Technical Committee ISO/TC 190, *Soil Quality*, Subcommittee SC 2, *Sampling*.

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

Introduction

Pretreatment of samples is usually required before they are tested to determine chemical or other properties, although there are some situations when any pretreatment would be unacceptable because it would affect the results.

Sample pretreatment is to preferably take place in the laboratory, as sample integrity can be best controlled under laboratory conditions. However, under some circumstances, pretreatment may be started in the field directly after sampling, to obtain a representative laboratory sample from the material extracted from the ground, or to prepare a composite laboratory sample.

The representativeness of a sample depends on factors like sample size, particle size, particle shape, contaminant type and concentration, consistence of soil materials and sampling strategy (see ISO 18400-104[1)]).

When volatiles are present, the procedures described in ISO 22155 are to be used as appropriate if possible. No further pretreatment is allowed. Other specified pretreatment methods will result in a significant loss of volatiles.

Pretreatment comprises one or a combination of the following:

— homogenization;

— sample division: obtaining subsamples of smaller size than the original sample without reducing the particle size of the individual particles;

— particle size reduction: grinding and crushing the sample in order to reduce the particle size of the sample without reducing the sample size (mass);

— separation of fractions on the basis of particle sizes (sieving or screening) if only a separate size fraction of soil is of interest for investigation or on the basis of the physical nature of the materials (e.g. appearance);

— preparation of composite sample(s).

Several cycles of a number of these activities could be required to derive the test sample (e.g. analytical sample) from the material extracted from the ground. Except as noted above when pretreatment would affect the results of subsequent testing or analysis, subsampling is normally required in the laboratory because the amount of material in the laboratory sample (i.e. that sent from the field to the laboratory) is almost always larger than the amount of material necessary for the test or analysis.

There might be occasions when it is considered desirable to combine soil material in the field from, for example different locations into a composite sample. A suitable procedure for doing this is described in this document.

For reasons explained in Clause 4, only some of the pretreatment measures listed above can be carried out in the field.

This document is part of a series of sampling standards for soil. The role/position of the International Standards within the total investigation programme is shown in Figure 1.

NOTE This document is intended to complement ISO 23909 and ISO 22155.

1) Under preparation.

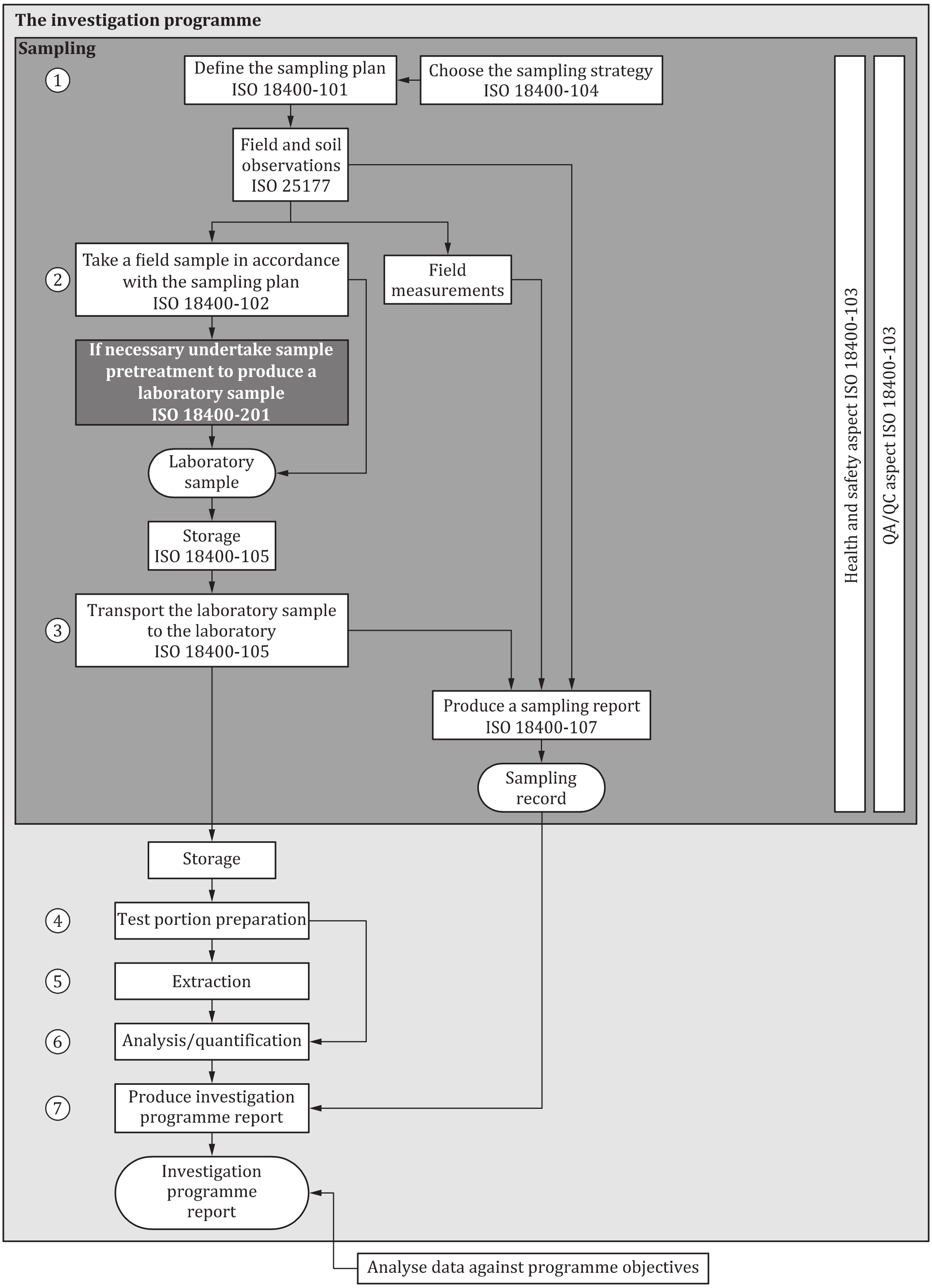


Figure 1 — Links between the essential elements of an investigation programme

NOTE 1 Numbers in circles define the key elements and steps of the investigation programme.

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

Soil quality — Sampling —

Part 201:
Physical pretreatment in the field

1 Scope

This document specifies methods for the pretreatment of samples that can be applied "in the field" directly after sampling. Pretreatment methods in this document are limited to:

— sample division methods aimed at reducing the size/volume of the sample;

— the production of composite samples;

— the selection of a specific fraction of the sampled material.

This document

— does not apply to samples required for biological or microbiological examination,

— does not apply to soil materials sampled for the content of volatile components, and

NOTE 1 These soil materials are intended to be sampled according to ISO 22155.

— does not give instructions for particle size reduction.

NOTE 2 Guidance for particle size reduction is given in ISO 11464, ISO 14507 and ISO 23909.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 11074, *Soil quality — Vocabulary*

ISO 18400-101:2017, *Soil quality — Sampling — Framework for the preparation and application of a sampling plan*

ISO 18400-104[2)], *Soil quality — Sampling — Strategies*

ISO 18400-105, *Soil quality — Sampling — Packaging, transport, storage and preservation of samples*

ISO 18400-107, *Soil quality — Sampling — Recording and reporting*

ISO 22155, *Soil quality — Gas chromatographic determination of volatile aromatic and halogenated hydrocarbons and selected ethers — Static headspace method*

DIN 19747, *Investigation of solids — Pre-treatment, preparation and processing of samples for chemical, biological and physical investigations*

2) Under preparation. Stage at the time of publication: ISO/DIS 18400-104:2016.

3 Terms and definitions

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

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

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

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

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

[SOURCE: ISO 11074:2015, 4.1.3]

3.2
laboratory sample
sample intended for laboratory inspection or testing

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

Note 3 to entry: Several laboratory samples can be prepared and sent to different laboratories or to the same laboratory for different purposes.

[SOURCE: ISO 11074:2015, 4.3.7]

3.3
sample division
(bulk material) activity in sample preparation whereby a sample of bulk material is divided by such means as riffling, mechanical division, or quartering into separate parts, one or more of which is retained

[SOURCE: ISO 3534-2:2006, 5.3.8]

3.4
subsample
selected part of a sample

Note 1 to entry: The subsample can be selected by the same method as was used in selecting the original sample, but need not be so.

[SOURCE: ISO 3534-2:2006, 1.2.19]

3.5
selective subsampling
separation of part of a sample on the basis of grading (i.e. above or below a defined particle size), appearance or some other attribute

3.6
volatile organic compound
VOC
organic compound that is a gas under normal environmental/atmospheric conditions, although it can be found in the ground in the solid, liquid and dissolved phase form as well as in the gaseous phase

Note 1 to entry: The US Environmental Protection Agency uses a variety of definitions for VOCs in different contexts but the one most appropriate here is "an organic compound which has a boiling point below that of water and which can easily vaporize or volatilize".

Note 2 to entry: Examples include single-ring aromatic hydrocarbons and other low boiling halogenated hydrocarbons, which are used as solvents or fuels, and some degradation products.

4 Preliminary considerations

The intention when sampling in the field is almost always to obtain a sufficiently representative sample of the desired size that can be placed directly in a container for transport to the laboratory. However, under some circumstances, as described in this document, some pretreatment can be done in the field to reduce the size of a large field sample to a more manageable size for sending to the laboratory or to select a particular fraction to form the laboratory sample.

The direct selection of the material to form the laboratory sample from the material extracted from the ground when this forms an integral part of the sampling process is described in ISO 18400-102 on the selection and application of sampling techniques.

When the laboratory sample is received, pretreatment is usually required before testing to determine chemical or other properties, although there are some situations when any pretreatment would be unacceptable because it would affect the results (e.g. when volatile organic compounds are present). Pretreatment is normally required in the laboratory because the amount of material in the laboratory sample (i.e. that sent from the field to the laboratory) is almost always larger than the amount of material necessary for the test or analysis.

Pretreatment comprises one or a combination of the following:

— homogenization;

— preparation of a composite sample;

— sample division: obtaining subsamples of smaller size than the original sample without reducing the particle size of the individual particles;

— particle size reduction: grinding and crushing the sample in order to reduce the particle size of the sample without reducing the sample size (mass);

— selection of a fraction of a sample on the basis of particle sizes, appearance, or other physical characteristic.

Several cycles of a number of these activities could be required to derive the test sample (e.g. analytical sample) from the laboratory sample.

The International Standards on pretreatment (ISO 11464, ISO 14507 and ISO 16720) describe laboratory procedures for mixing (homogenization), dividing and particle size reduction, in order to provide a representative sample (e.g. analytical sample) assuming a laboratory sample (i.e. the material received in the laboratory for inspection or testing) of approximately 1 kg. When the sample received at the laboratory is larger than about 1 kg, the size of the sample can be reduced following the procedures described in ISO 23909 (this assumes a sample of about 25 kg is to be reduced in size but the procedures described are applicable to much larger samples).

ISO 11464, ISO 14507, ISO 16720 and ISO 23909 shall only be used for pretreatment of materials within their respective scopes and having regard to the need to preserve sample integrity. Inappropriate use of these International Standards, including ISO 14507, will result in unacceptable loss of volatile organic compounds (VOCs) (3.6) and other volatiles.

When volatiles are present, the procedures described in DIN 19747 and ISO 22155 shall be used as appropriate.

NOTE DIN 19747 covers chemical, physical and biological investigations.

Uncertainty about whether a compound should be regarded as volatile or not should trigger a specific quality scheme to ensure that sample preparation does not introduce bias, cross contamination or other forms of unacceptable errors. Guidance on quality control is given in ISO 18400-106.

Sample pretreatment should preferably take place in the laboratory, as sample integrity can be best controlled under laboratory conditions. Among other things, the laboratory should have a range of equipment available that can be selected on the basis of the size and nature of the sample to be processed. However, under some circumstances, pretreatment of the material extracted from the ground may be started in the field directly after sampling. For example, the size of sampling equipment might be such that more soil material is extracted from the ground than needed. Sample pretreatment "in the field" is then necessary in order to limit the amount of material to be transported to the laboratory.

The procedures described can be used in the field to limit the amount of material to be transported to the laboratory. They can be used to produce a laboratory sample of about 1 kg (or larger if required) which can then be subjected as appropriate to the pretreatment procedures described in ISO 11464, ISO 14507, ISO 16720 or DIN 19747 or produce a larger sample that can then be subjected in the laboratory to the procedures described in ISO 23909 to further reduce the size of the sample.

Size reduction, other than the manual crushing of clods and/or macro-aggregates as described in 9.4, is seldom practical in the field because it requires powered equipment and appropriate laboratory conditions. Particle size reduction involves a substantial risk of (cross) contamination, loss of components and loss of soil material. These risks can be properly controlled under laboratory conditions. Particle size reduction should therefore only be carried out under laboratory conditions.

Effective homogenization can be difficult in the field because it often requires powered equipment and appropriate laboratory conditions, but can be done provided proper care and equipment is used (see Clause 8).

Depending on the objective of the investigation programme, it might be that there is only an interest in part of the soil or soil-like material. For example when "non-soil materials" are present (e.g. bricks, stones). This might imply that it is desirable to obtain only a specific size fraction of the material, either through removing the large elements from the sample, or, the other way around, through specifically selecting the larger parts that are of interest. Sometimes both fractions could be of interest.

Selective subsampling of materials of a particular grading (e.g. below a defined particle size) could be possible in the field if the material sampled is suitably dry (see 9.6). Sieving or screening is regularly practised in horticulture and when old mineral waste deposits are being processed on a small-scale to recover previously discarded materials of value. However, it might not be desirable in a particular case as it will usually be necessary to record the type and amount of both over-sized particles and under-sized particles to provide a full characterization of the material being sampled, and it could be difficult to avoid losses, especially fine materials, while processing the sample. Such processes are best carried out under laboratory conditions where a range of manual and powered equipment should be available.

As described in this standard (see 9.6.3), a fraction of the field sample may also be formed in the field (or the laboratory) by "hand-picking" of material from the bulk sample on the basis of particle size, appearance (e.g. colour), or nature (e.g. wood fragments, coal, organic/vegetable material, asbestos cement materials). As for sieving, the mass of the material removed should be weighed and recorded as should the mass of the bulk sample from which it is removed.

The preparation of composite samples is usually an integral part of the sampling process (see ISO 18400-102), e.g. in cluster sampling numerous small incremental samples roughly equal in size taken from a small area are placed in the sample container to form the laboratory sample which is then homogenized in the laboratory as part of the pretreatment process.

In spatial (i.e. area-wide) composite sampling, incremental samples roughly equal in size taken on a defined sampling pattern across the area of interest (e.g. a field) are placed in the (largish) sample container to form the laboratory sample which is then homogenized and subsampled in the laboratory as part of the pretreatment process.

However, there could be occasions when it is considered desirable to combine soil material in the field from, for example, different locations, into a composite sample. A suitable procedure for doing this is described in this standard (see Clause 8).

5 Incorporation in the sampling plan

The pretreatment method(s) to be used in the field (if pretreatment is necessary) and the necessary equipment shall be prescribed in the sampling plan according to ISO 18400-101.

When the circumstances in the field deviate too much from the assumed situation in the sampling plan, the requirements concerning pretreatment in the plan should be changed. In general, minor changes that have no effects on the test results may be made in the field by the sampler. If effects on the test results are to be expected or when in doubt, the sampler shall consult the project manager. This includes seeking advice on how to proceed if circumstances in the field or weather conditions deviate too much from the assumed situation in the sampling plan.

The project manager should always be consulted (see Note 1), when

— there is a change in the necessity for pretreatment,

— there is a change in the practicality of pretreatment.

Any changes made to the sampling plan should, like the original requirements in the plan, be in conformance with this document. The guidance in ISO 18400-101:2017, Clause 6 on the procedure when changes to the sampling plan are needed during sampling should be followed.

NOTE 1 ISO 18400-101:2017, Clause 6 distinguishes between changes that will not affect the achievement of the objective of the investigation and those which might affect the achievement of the objective of the investigation.

NOTE 2 The necessity of pretreatment might change for example when pretreatment was not planned, but appears to be necessary in light of the coarse soil material to be sampled (the particle size distribution of the soil material was not adequately identified by the project manager when defining the sampling plan).

NOTE 3 The practicality of pretreatment might change for example due the absence of a clean and unused surface at the sampling site or due to weather conditions that do not allow sample pretreatment of sufficient quality.

NOTE 4 As the potential effect of changes in the sample pretreatment will depend on the nature of the necessary changes, the specific sampling situation and the test to be performed, no further guidance to these changes is provided in this document.

6 General requirements

There are potential disadvantages in carrying out pretreatment in the field (see Note 1). Before deciding to do this, the sampling plan should be reviewed to determine whether the necessity to do so can be avoided by changing the sampling techniques to be employed, e.g. to select techniques that will provide samples suitable in size for direct transport to the laboratory without pretreatment.

Whatever sample pretreatment is carried out, the defined objectives of the sampling exercise and the need for samples to be “representative” to avoid bias (or to acknowledge unavoidable or designed bias) should govern what is done.

When it is considered that pretreatment in the field is unavoidable, consideration should be given to the establishment of a temporary on-site pretreatment laboratory. This may be a specially constructed facility or an area within in an existing building adapted for the purpose.

When establishment of a full on-site laboratory is not practical, provision of at least temporary protection from the weather should be considered (e.g. a canopy over the treatment area).

NOTE 1 Sample pretreatment, either being sample division or the preparation of a composite sample, can result in significant changes in the composition of the soil material when no or inadequate precautions are taken. The risk of significant changes depends on the components or characteristics to be tested, the nature of the soil material, the pretreatment method selected and the (weather) conditions under which the pretreatment is performed (see also Note 3).

NOTE 2 An on-site pretreatment facility can provide reasonably controlled conditions free of the vagaries of the weather that can be kept properly clean and, if necessary, provided with powered equipment in a safe environment.

As field conditions are rarely comparable to laboratory conditions, the types of sample pretreatment that should be carried out in the field are limited to sample division and the production of composite samples and/or the selective subsampling of a specific part of the sampled material that is of interest. Only when laboratory conditions are available on site (there is a sample pretreatment laboratory/facility present) can the full range of sample pretreatment activities—thus also including particle size reduction—be carried out directly after sampling.

Integrity of the soil material should always be ensured. Therefore a method, time and place for sample pretreatment should be chosen that causes the minimum possible risk of changes in the characteristics of the soil material.

When the aim of the sample pretreatment is to perform selective subsampling, the composition of the soil material will obviously change, however, care should be taken to ensure that only the desired changes occur.

Suitable methods for subsampling might be limited by the requirements set by the components to be analysed or the test to be performed. Expert advice should be sought from the laboratory.

NOTE 3 Some examples of problems that might arise are:

— loss of moisture;

— loss of volatile components;

— aggregate formation of soil material while subsampling due to its moisture content;

— oxidation of newly exposed material following crushing of soil macro-aggregates, etc.;

— soil material sticking to the equipment (e.g. clay);

— loss of fine particles due to air entrainment;

— addition of water due to rain;

— loss of fine particles due to heavy rain;

— presence of oily materials that might adhere selectively to sheeting and tools;

— separation of components including mineral oils due to density differences or other differences in physical characteristics;

— biodegradation of organic materials and substances.

7 Safety

Pretreatment in the field should be carried out in accordance with the guidance on safety in ISO 18400-103 and with regard to any other relevant factors.

8 Homogenization

Effective homogenization can be difficult in the field because it often requires powered equipment and appropriate laboratory conditions, but can be done provided proper care and equipment is used. The chances of success depend on a variety of factors including in particular on the heterogeneity of the soil and the property of interest (e.g. how a contaminant is distributed within and on particles).

The procedures described in 9.5 are all intended to ensure as far as practical that the subsample is sufficiently representative of the larger field sample that is to be reduced in size for the purposes of the investigation. For example, the first steps in the long pile and alternate shovel method (9.5.2) are intended to homogenize the sample.

Mixing to obtain bulk sample homogeneity can be achieved by passing and merging the sample three, four or more times through a mechanical sample splitter (e.g. riffle box, Tyler divider or rotating divider).[18]

9 Subsampling

9.1 General

The characteristics of the soil material to be subsampled should be checked against the methods described in the sampling plan to see whether the prescribed methods are appropriate. The maximum size of the particles is important, as is the moisture content. The latter is related to the inclination to macro-aggregate formation and cohesive behaviour. If the prescribed methods are not considered appropriate, the sampling plan should be amended in accordance with Clause 5.

In the case of very coarse soils, it can be necessary to reduce the particle size of the larger particles in the field in order to be able to send a representative sample of an acceptable size to the laboratory. When grinding or crushing "in the field" is truly necessary, measures have to be taken in order to prevent contamination and/or loss of both components and soil material. Particle sizes in the sampled material should therefore only be reduced as little as possible, just allowing the laboratory sample to be of acceptable size.

Everything put into the size reduction process should be reduced in size to pass a predetermined sieve to avoid selectivity.

The location for subsampling should be chosen carefully and made fit for use, by ensuring a fully flat surface, cleaning it of all materials that could influence the integrity of the (sub)sample(s) and by covering with an appropriate material (see below). When all preparations are ready, the sample pretreatment should be carried out using the prescribed method(s).

Heavy duty plastic sheeting will be suitable in many situations when the work is undertaken using manual means but will not withstand trafficking, etc. by mechanical equipment. When this is to be used, canvas, wood, or metal sheeting should be used.

9.2 Equipment for subsampling

All materials and equipment that come in contact with the (sub)sample(s) should be such that they will not contaminate the (sub)sample(s).

Prior to subsampling, the apparatus and tools used shall be cleaned in order to prevent cross-contamination. This means that they should be cleaned between processing different samples. The protective covering to the working area should be cleaned or replaced between processing different samples.

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

— large heavy-duty plastic sheeting or other protective covering such as wood or metal sheeting (see 9.1);

- stainless steel spade or shovel;
- mechanical shovel;
- tools for reducing the particle size (e.g. sledge hammer);
- riffle box;
- tools for subsampling (e.g. cross divider, Tyler divider);
- mechanized turntable/rotating dividers.

All equipment used shall comply with relevant International Standards (e.g. ISO 565 for sieves).

NOTE 1 For a description of the equipment used in this document, see ISO 11464.

NOTE 2 A Tyler divider is a device that cuts a representative 1/16th from feed material by systematically rejecting segments of material flowing down an adjustable 45° or 60° incline (see Figure A.1). It can be used “batch-wise” or with a continuous feed.

9.3 Minimum size of the subsample

Decisions about the size of subsample required should be based on the guidance provided in ISO 18400-104. This states that a number of factors govern the size of the subsample to be sent to off-site laboratories (i.e. the laboratory sample) including:

- the range of pedological, chemical, physical and or biological examinations and tests that are to be carried out;
- the specific requirements of the laboratory/laboratories carrying out the examinations and tests; and
- the need for samples to be sufficiently representative, taking into account the particle size distribution and the concentration distribution of the material to be sampled;

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

The first two of the above points will usually be defined by following an established procedure and/or discussion with the laboratory/laboratories.

As discussed in ISO 18400-104, the relationship between particle size distribution and the size of sample or increment required to obtain reliable analytical results for a granular material in which the properties of interest are inherent within the particles is amenable to theoretical determination. However, this is not possible 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 phases. 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. A minimum sample size of 500 g to 1 000 g often will be required. However it should be noted 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).

NOTE The guidance provided in DIN 18123 regarding the relationship between minimum sample size and maximum particle size might help when making a decision about the size of sample required. When the sample contains macro-aggregates, the maximum size of the macro-aggregates determines the minimum size of the subsamples whenever the macro-aggregates behave like individual particles during subsampling, i.e. when macro-aggregates are not cut into pieces by the subsampling equipment used (see 9.4 for macro-aggregate size reduction).

Table 1 — Minimum sample size and maximum particle size

DIN 18123	
Maximum particle size mm	Minimum sample size g
10	700
20	2 000
30	4 000

9.4 Procedure for macro-aggregate reduction by hand

Soil macro-aggregates should be regarded as individual "particles" when the method of sampling and sample pretreatment is not able to sample part of a macro-aggregate.

NOTE When subsampling under field conditions, macro-aggregates behave as individual particles for instance when a riffle box is used for dividing a moist or clay-like soil. As the particle size is one determinant of the minimum size of laboratory sample required, it will be preferable to reduce the size of macro-aggregates if possible during or prior to subsampling so as to reduce the size of laboratory sample required.

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

Macroaggregates should be reduced in size in accordance with the following procedure:

— identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread evenly on the surface;

— place a clean protective floor covering to protect the sample from contamination by the surface and loss of material (9.1 and 9.2);

— place the sample on the protective covering sheeting and spread evenly to identify all macro-aggregates within the sample;

— use the base of a spade or the head on a sledge hammer (see 9.2) gently to reduce the size of the macro-aggregates until all oversized material is less than or equal to the required particle size.

If available, suitable machinery may also be used.

9.5 Subsampling methods

9.5.1 General

When subsampling in the field, where materials are likely to be moist and to behave cohesively, sample division by manual means is preferable (mechanical division is often impossible or will result in biased subsamples).

Drying before sample reduction may be carried out if this does not influence the sample integrity (e.g. due to volatilization, biodegradation).

NOTE Subsampling can be performed either mechanically or manually. However, this is only true when the material is dry and particles can move in a stream of particles on an individual basis. When this is the case, i.e. the materials are dry and free flowing, it is preferable to use a mechanical system since this will result in more representative subsamples. If the particles in the sample behave cohesively, mechanical division is often impossible due to cohesion of soil in the system and subsequent blockage of the divider. And even when the mechanical division is still possible, mechanical subsampling devices will probably function incorrectly, and therefore will result in biased subsamples.

If the material is suitably dry, determine the minimum size of the subsample(s) required according to 9.3 and ISO 18400-104[3]. When the minimum size of the subsamples is larger than desired and the maximum particle size is related to the size of macro-aggregates, the macro-aggregate size can be reduced according to 9.4. The subsampling process shall be stopped when the size of the subsample is equal to or larger than the size of sample required as determined by reference to 9.3 and ISO 18400-104[3] (see also Table 1).

Based on the boundary conditions as given in the subsequent paragraphs and practical experience, one of the following subsampling methods should be chosen:

— long pile and alternate shovel method (see 9.5.2);

— coning and quartering (see 9.5.3);

— riffling (see 9.5.4);

— application of Tyler divider (see 9.5.5);

— application of mechanized turntable (rotating divider) (see 9.5.6).

9.5.2 Long pile and alternate shovel method

This subsampling method is suitable for samples in excess of approximately 100 kg. The procedure is as follows:

— identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface;

— place a clean protective floor covering to protect the sample from contamination by the surface and loss of material;

— shovel the soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples in excess of approximately 500 kg, the use of a mechanical shovel is preferable to manual handling. Manual handling is preferred for samples smaller than 500 kg;

— when the entire soil sample is on the floor, work around the cone systematically depositing shovelfuls from the base to the apex of the cone so that the centre of the cone is not displaced. Repeat the process twice;

— form the cone into a long pile as follows:

  — taking a shovelful from the base of the cone, spread the material into a ribbon having an initial width equal to that of a shovel and a length of 1,5 m to 3,0 m;

  — take the next shovelful from a different point at the base of the cone, and spread it directly over the previous shovelful, but in the opposite direction;

  — repeat the above step until one long pile is formed;

— discard half the soil sample in the following manner:

  — take a shovelful from the bottom of one end of the pile and set it aside;

  — take the next shovelful immediately adjacent to the first by advancing along the side of a pile a distance equal to the width of the shovel and discard this shovelful;

  — again, advancing in the same direction a distance of one shovel width, take the third shovelful and add it to the first;

3) Under preparation. Stage at the time of publication: ISO/DIS 18400-104:2016.

  - continue along the pile following the above procedure, discarding alternate shovelfuls so that the pile is decreased gradually and uniformly;
- repeat the above procedure (from forming the coning to halving the pile) until the retained amount of material is equal to or greater than the required size of the subsample;
- transfer the subsample to an appropriate sample container in accordance with Clause 11.

9.5.3 Coning and quartering

NOTE Coning and quartering are known to be subject to bias. This bias is partly caused by the tendency of larger particles to roll down the side of the cone and to collect at the base. This results in segregation of particles from the top to the bottom of the cone. The same problem arises when taking subsamples when the areas to be subsampled are not previously separated (for instance, by the metal cross as described in the first method of quartering).

This procedure is suitable for all samples down to approximately 1 kg. The procedure is as follows:

- identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface;
- place a clean protective floor covering to protect the sample from contamination by the surface and loss of material (see 9.2 and 9.3);
- shovel the soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples in excess of approximately 500 kg, the use of a mechanical shovel is preferable to manual handling. Manual handling is preferred for samples smaller than 100 kg;
- when the entire soil sample is on the floor circumvent the cone systematically taking shovelfuls from the base and forming a second cone with all the material from the first cone transferred to the apex of the second cone. Repeat the process twice;
- flatten the cone so that the height is less than or equal to the height of the shovel or spade used;
- divide the pile into quarters along two lines intersecting at 90° to each other, using one of the following methods:

  Method 1:

  - place the centre of a sheet metal cross, made with four blades joined together at the centre at 90° to each other, at the centre of the flattened cone and press the lower edges of the metal cross through the soil sample (the height and length of the blades forming the cross should be greater than that of the flattened cone);
  - discard one pair of opposite quarters with the metal cross still in place and mix together the remaining two quarters forming another stockpile;
  - check if the mass of the discarded material is equal to half the mass of the (sub)sample before subdivision, allowing a variation of ±10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, where after the subdivision can continue;
  - cone and quarter again, using the previous stages, until the mass of remaining soil is equal to or greater than the required size of the subsample;

  Method 2:

  - quarter the flattened cone along two diagonals intersecting at right angles, using a shovel inserted vertically into the soil;
  - discard one pair of opposite quarters and shovel the remainder into a stockpile;

  - check if the mass of the discarded material is equal to half the mass of the (sub)sample before subdivision, allowing a variation of ±10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, where after the subdivision can continue;

- repeat the process of mixing and quartering until the mass of remaining soil is equal to or greater than the required minimum size of the subsample (but no less than the minimum size of the subsample in accordance with Table 1);

- transfer the subsample to an appropriate sample container in accordance with Clause 11.

9.5.4 Riffling

The use of a riffle box is possible when the soil is dry enough to allow free flow of the soil particles through the riffle box (see Figure A.2). The maximum quantity of material that can be treated depends on the size of the riffle box. It is usually practical to treat a maximum of about 100 kg in a single riffle operation.

Division of the sample with a riffle box will result in a reduction to one half or one quarter (depending on the riffle) at each operation:

- identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface;

- place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface and loss of material (see 9.2 and 9.3);

- shovel the soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one;

- when the entire soil sample is on the floor circumvent the cone systematically taking shovelfuls from the base and forming a second cone with all the material from the first cone transferred to the apex of the second cone. Repeat the process twice;

- check before starting that the slot widths of the riffle box are at least three times larger than the maximum particle size of the soil to be subsampled;

- using a shovel or container, pour the material into the riffle box. It is essential that the soil is poured evenly over the whole riffle in order to avoid biased subsampling;

- remove one subsample as the reduced sample, discarding the remaining material,

- check if the mass of the discarded material is equal to half (or three quarters of) the mass of the (sub)sample before subdivision, allowing a variation of ±10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, where after the subdivision can continue;

- repeat the process of riffling until the mass of remaining soil is equal to or greater than the minimum required size of the subsample;

- transfer the subsample to an appropriate sample container in accordance with Clause 11.

9.5.5 Application of Tyler divider

The sloping plate of the Tyler divider (see Figure A.1) provides a reduction ratio of 16:1. Material flows over the plate and is reduced successively in steps at each station down the plate by means of slots or holes placed in the plate. Each reduction is to one half of the amount passing the station and a means for re-mixing after each stage is incorporated in the plate. An essential requirement in applying a Tyler divider is that the soil is dry enough to allow free flow of the soil particles.

If a mechanical feed is used, it should be set at a constant rate suitable for the material being provided to the Tyler divider. This implies the requirement for the hopper width to be equal to that of the sloping plate and a gate of variable height.

The procedure is as follows:

— identify the maximum particle size of the sample;

— check that the slot width of the Tyler divider is at least three times larger than the maximum particle size;

— determine the minimum size of the subsample(s) required and calculate if the reduction ratio of the divider will result in a subsample that is equal to or larger than the minimum size of the subsample. If not, this type of divider shall not be used;

— start the division process by pouring the sample into the divider with a constant rate and catch the subsamples(s) in (an) appropriate sample container(s);

— when necessary repeat the process of subsampling using one or more of the resulting subsamples until a subsample of at least the minimum required size is obtained;

— transfer the subsample to an appropriate sample container in accordance with Clause 11.

9.5.6 Application of mechanized turntable (rotating divider)

The mechanised turntable comprises a number of prismatic containers, of equal size, mounted round the periphery of a circle which pass under the falling stream of the sample fed from a hopper mounted above the turntable, and off-set from the centre (see Figure A.3).

The turntable should operate at a constant speed of rotation that should not change (significantly) while sample material is coming into the turntable.

The procedure is as follows:

— check that the slot width of the turntable is at least three times larger than the maximum particle size;

— transfer the soil with a constant speed into the turntable. The speed should be relatively low in order to allow all particles to fall freely into the slot of the turntable (it will take a large number of rotations of the turntable before the full amount of soil is transferred into the slot);

— after completion of the division process, one or more of the subsamples is(are) collected;

— check the mass of one of the subsamples. If the mass is not equal to the product of the total mass and the inverse number of subsamples in the rotating divider, allowing a variation of ±10 % (mass fraction), all subsamples shall be added and the subsampling step shall be repeated;

— the subsamples obtained are (if necessary) divided again, until a subsample of at least the minimum required size is obtained;

— transfer the subsample to an appropriate sample container in accordance with Clause 11.

9.6 Selective subsampling based on the particle size

9.6.1 General

Selective subsampling should only be used when this is explicitly required in the sampling plan.

NOTE 1 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.

Two methods for selective subsampling are described:

— sieving (see 9.6.2);

— hand picking (see 9.6.3).

Both methods can be applied, independent of whether the interest is in the fraction with the smaller or the larger particles. In addition, hand picking can also be applied when other distinctive features of the material are a basis for the selection.

Sieving is to be 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.

The nature and amount of any material discarded should be reported, together with the mass of the material retained.

NOTE 2 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.

9.6.2 Sieving

The type and size of the equipment for sieving depends on the size of the sample and can vary between heavy mechanical equipment and hand-held screens.

NOTE As a side effect, the amount of energy used for sieving has some influence on the size reduction of macro aggregates, but as such it is not a method for macro aggregate size reduction; see 9.4.

Depending on the particle size distribution, it can be efficient to use screens of different mesh size, either in repetitive steps or on top of each other.

The procedure is as follows:

— place the sample on top of the screen and move the screen for such a time that all smaller particles have passed the screen;

— after completion of the sieving process, the fraction of interest is collected as the subsample;

— transfer the subsample to an appropriate sample container in accordance with Clause 11.

9.6.3 Hand picking

Hand picking is based on visually distinguishing the larger particles or the particles with specific characteristics and either selecting these as the sample or discarding them.

The procedure is as follows:

— identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface;

— place a clean protective floor covering to protect the sample from contamination by the surface and loss of material (see 9.1 and 9.2);

— transfer the sample to the floor covering;

— flatten the sample so that the height is less than or equal to the height of the particles that are to be handpicked;

When the handpicking is based on a distinctive feature other than the size of the particles, the layer of soil should be so to allow for visible distinction of all particles.

— carry out the hand picking using tweezers or gloves in a manner that avoids cross contamination and that is protective of the safety of the person carrying out the operation;

— put the handpicked material aside of the rest of the sample;

— after completion of the handpicking, transfer the subsample to an appropriate sample container in accordance with Clause 11.

NOTE This can either be the handpicked particles, the remaining material or both fractions, depending on the objective of sampling.

10 Forming composite samples

10.1 General

The preparation of composite samples is usually an integral part of the sampling process (see ISO 18400-102), for example:

— in cluster sampling, numerous small incremental samples roughly equal in size taken from a small area are placed in the sample container to form the laboratory sample which is then homogenized in the laboratory as part of the pretreatment process;

— in spatial (i.e. area-wide) composite sampling, incremental samples roughly equal in size taken on a defined sampling pattern across the area of interest (e.g. a field) are placed in the (largish) sample container to form the laboratory sample which is then homogenized and subsampled in the laboratory as part of the pretreatment process.

However, there could be occasions when it is considered desirable to combine soil material in the field from, for example, different locations, into a composite sample.

Two situations for the composition of composite samples can be distinguished:

— the individual increments are completely used in formation of the composite sample;

— parts of the individual samples are used in formation of the composite sample.

When only part of the individual samples is used for the composition of a composite sample, the process of sub-sampling to obtain the soil material to be used for the composite sample shall be performed according to 9.5.

The characteristics of the soil material to be combined in a composite sample should be checked against the method described in the sampling plan. Basically, the maximum size of the particles in the original soil samples is important, as is the moisture content. The latter is related to the inclination to macro-aggregate formation, cohesive behaviour and potential loss of water and/or components in the composition process.

Composite samples should contain equivalent quantities (mass fraction) of the individual increments or samples. For incremental sampling, this is realized by using a constant volume (as a consequence of the applied sampling apparatus) and assuming the water content between the increments to be constant. When forming a composite sample from individual samples, care should be taken to use the same quantity of material from each individual sample. When a significant deviation in dry matter content is expected, the increments or samples should be put together in the laboratory after determining the dry matter content of each of the individual increments or samples.

Effective homogenization of the composite samples is difficult in the field and should preferably be carried out in the laboratory on receipt of the sample. However, techniques are available that should be effective if the soil is in an appropriate condition (e.g. dry and free flowing). See Clause 8.

The size of the composite sample should only be reduced in the laboratory and by using an appropriate sub-sampling procedure (see Clause 4).

Equivalent quantity on a mass basis implies that account should be taken of the variation in water content for the individual increments or samples, for which it would be necessary to know the dry matter content. This is generally impossible under field conditions and, consequently, will usually have to be assumed although field instruments for measurement of moisture contents are available.

NOTE 1 For incremental sampling, the volume of the increments will vary in practice, but in general, when the increments are obtained correctly, these variations can be considered sufficiently small not to affect the measurement results significantly.

The water content will depend on the origin of the increments or samples. Consequently, composite samples should not be formed in the field from the increments or samples originating from different soil layers, different soil types and the like.

When parts of individual samples are used for the composite sample, the method of subsampling should be such that the same quantity of material from each sample is transferred to the composite sample. This means that preferably a small scoop or similar is used with a constant volume and that the number of scoops taken from each individual sample is constant. The risk of error in the composition process is larger than in incremental sampling.

NOTE 2 In practice, it is often assumed that increments can actually be mixed, for instance by stirring. Effective mixing of particulate materials is however very difficult, especially when the increments have a different particle size distribution, moisture content or soil type. Appropriate mixing is best realized by specific sample pretreatment methods under laboratory conditions. However, techniques are available that can be effective if the soil is in an appropriate condition (e.g. dry and free flowing). See Clause 8.

10.2 Minimum size of increments or subsamples

For the minimum size of either the increments or subsamples, 9.3 applies.

10.3 Production of composite samples

10.3.1 Composite sample based on incremental sampling

The following procedure should be followed:

— transfer the complete increment obtained by sampling the soil material to the sample container;

— record the number of increments forming the composite sample.

10.3.2 Composite sample based on parts of individual samples

The following procedure should be followed:

— place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface and loss of material;

— take a subsample of the appropriate size in accordance to 9.5;

— transfer the complete subsample obtained by sampling the soil material to the sample container;

— record the number and identity of the samples joined in the composite sample.

11 Packaging and storage

Packaging, storage, etc. shall be in accordance with ISO 18400-105.

12 Reporting

Reporting shall be in accordance with ISO 18400-107.

The report should contain at least the following information:

a) a reference to this document, i.e. ISO 18400-201;

b) a complete identification of the sample;

c) the date of subsampling or other pretreatment;

d) details of the division technique applied;

e) the weight and a description of any removed particles;

f) the whole sequence and operating conditions (procedures and equipment used) actually applied to the sample for preparation of (a) subsample(s);

g) any relevant details not specified in this document or which are optional, as well as any factor which might have affected the results.

Annex A
(informative)

Illustrations of apparatus

Figure A.1 — Tyler divider

Dimensions in millimetres

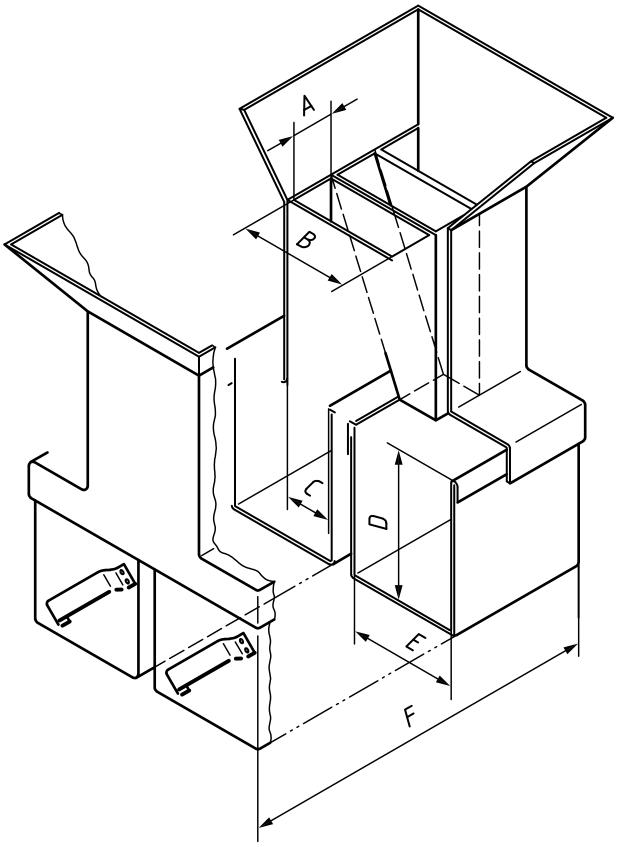


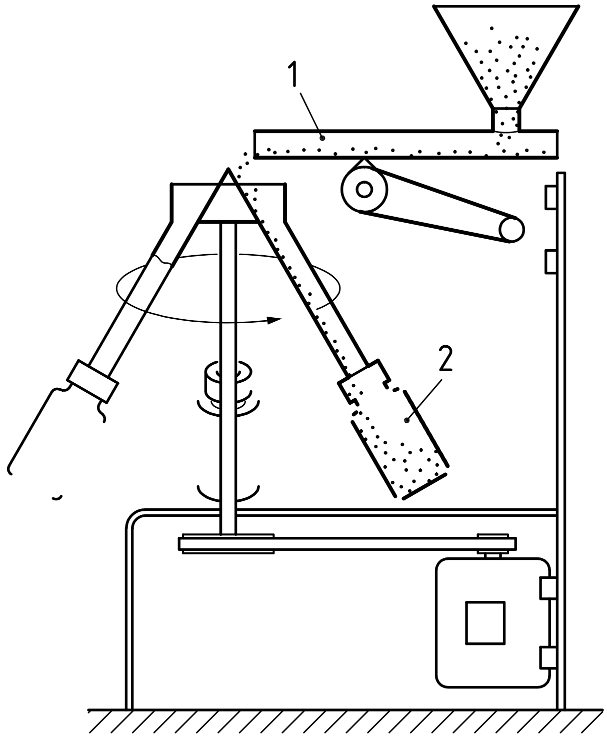
Figure A.2 — Riffle sampler divider/splitter (riffle box)

Table A.1 — Dimensions of a sample divider

Dimensions in millimetres

Maximum size of sample	Number of slots	Internal dimensions[a]			Internal dimensions of the boxes (two required)[a]		
		A	B	C	D	E	F
40	8	50	150	70	230	150	400
20	10	30	130	40	150	100	300
10	12	15	80	30	120	90	200
5	12	7	20	15	50	50	90
2	12	5	20	15	50	50	90

[a] All dimensions, except A, are approximate.



Key

1 vibrating tube

2 sample bottle

Figure A.3 — Example of a rotating sample divider

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