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Soil quality — Preparation of laboratory samples from large samples

Qualité du sol — Préparation des échantillons de laboratoire à partir d'échantillons de grande taille

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Contents

Foreword

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

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

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

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

ISO 23909 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

The existing International Standards on pretreatment (ISO 11464, ISO 14507 and ISO 16720) are based on a laboratory sample of approximately 1 kg. Such a sample is considered representative if the maximum size of the particles is 8 mm (in accordance with Table A.1). The representivity also depends on other factors, like an adequate sampling strategy. The mentioned International Standards describe methods to mix and divide and reduce the particle-size, in order to provide a representative test sample. For volatiles, ISO 14507 provides a procedure for taking a test sample. The other specified pretreatment methods will result in a significant loss of the contaminant.

This International Standard connects samples taken in accordance with ISO 10381-8 and other methods where large samples are obtained with the International Standards concerning pretreatment ISO 11464, ISO 14507 and ISO 16720. This International Standard is defined for samples up to typically 25 kg, but is basically also applicable to considerably larger samples.

Soil quality — Preparation of laboratory samples from large samples

1 Scope

This International Standard specifies a method for the preparation of laboratory samples from large samples. In accordance with ISO 11464 and ISO 14507, the following analyses are considered:

- determination of physico-chemical parameters;
- determination of moderate volatile organic contaminants;
- determination of non-volatile contaminants.

This International Standard is not applicable when volatile organic compounds have to be analysed.

2 Normative references

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

ISO 10381-8:2006, *Soil quality — Sampling — Part 8: Guidance on sampling of stockpiles*

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

ISO 14507:2003, *Soil quality — Pretreatment of samples for determination of organic contaminants*

ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis*

3 Terms and definitions

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

3.1
analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

[ISO 11074:2005][3]

3.2
laboratory sample
sample intended for laboratory inspection or testing

3.3
moderately volatile compounds

sum of moderately volatile organic compounds and volatile inorganic compounds (e.g. mercury, arsenic cadmium, thallium) that can be lost during sample preparation (e.g. heating)

3.4
moderately volatile organic compounds

organic compound having a boiling point above 300 °C (at a pressure of 101 kPa)

NOTE This definition includes mineral oil, most polycyclic aromatic hydrocarbons (PAH) (see Reference [4]), polychlorobiphenyls (PCB) (see Reference [2]), organochlorine pesticides (see Reference [2]).

[ISO 14507:2003]

3.5
sample

subset of a population made up of one or more sampling units

NOTE Many different ways, random and not random, of selecting a sample can be envisaged. A collection of data obtained by biased sampling that is unavoidable in many areas (e.g. in human genetics, of families detected through abnormal children), is also a sample. In survey sampling, sampling units are often selected with a probability proportional to the size of a known variable, giving a biased sample.

[ISO 3534-2:2006][1]

3.6
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

[ISO 3534-2:2006][1]

3.7
subsample

selected part of a sample

NOTE The subsample can be selected by the same method as was used in selecting the original sample, but need not be so.

[ISO 3534-2:2006][1]

3.8
volatile organic compounds

organic compound having a boiling point below 300 °C (at a pressure of 101 kPa)

NOTE This includes volatile aromatic and volatile halogenated hydrocarbons, as determined in accordance with ISO 15009[5]. Some mono- and dichlorophenols, for instance, and naphthalene also belong to this group.

[ISO 14507:2003]

4 Principle

A laboratory sample is prepared from a large sample obtained in the field by manual or automated division. The maximum particle size defines the minimum mass of the subsamples and, if necessary, the degree of size reduction.

5 Apparatus

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

It is therefore preferable to choose a method of sample pretreatment that causes the minimum possible change in composition, particularly with respect to subsequent requirements of the material. For a description of the equipment used within this International Standard, see ISO 11464 and ISO 10381-8.

6 Procedure

6.1 General

Depending on the maximum size of soil aggregates, it is necessary to have a minimum amount of a sample that is representative of the site. This is described in Annex A (modified from ISO 10381-8). Following the procedure specified in ISO 10381-8, samples of several kilograms can be sent to the laboratory. To obtain the desired sample which is representative for analyses, it is necessary to mix and divide the sample as well as reducing the particle size, because methods of analysis are usually based on a test sample of less than 1 g and up to 50 g. Apart from large samples due to the maximum particle size of the soil material sampled, large samples can also occur due to the application of sampling strategies where large numbers of increments are put together in a composite sample.

6.2 Methods of sample division

In accordance with ISO 11464 and ISO 14507, the following analyses are considered:

- determination of physico-chemical parameters;
- determination of moderate volatile organic contaminants.

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

The location for sample pretreatment shall be chosen and the location shall be made fit for use by cleaning it of all materials that can influence the integrity of the (sub)sample(s). When all preparations are ready, the sample pretreatment shall be carried out using the chosen technique.

Remove particles not relevant for the analyses, weigh and describe them. If the largest particle size within the laboratory sample is too big, a stone crusher or another crushing device can be used to reduce the maximum particle size before further reduction to 2 mm. Direct reduction of the particle size to less than 2 mm is only allowed if this does not influence the results. For moderate organic contaminants, losses will occur due to development of heat in such a step.

If it is not possible to reduce the maximum particle size to a value which can be processed by one of the described techniques, this shall be reported with the remark that it was not possible to obtain a representative subsample and the results shall be considered as indicative.

Use one of the following procedures to obtain a sample of 1 kg:

- coning and quartering;
- riffling;
- Tyler divider;
- rotating divider.

NOTE These procedures are also described in ISO 10381-8.

Pretreat the obtained laboratory sample further, in accordance with ISO 11464, ISO 14507 or ISO 16720.

The equipment should not contaminate the sample with substances to be analysed.

It can be desirable to dry the sample for a better performance of the stone crusher. This is allowed for physico-chemical analysis and non-degradable organic contaminants. If degradable contaminants are present, the fresh sample should always be used and pretreated as quickly as possible.

Having obtained the subsample, it shall be stored directly in a suitable sample container.

6.3 Reduction of sample size

6.3.1 General

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

6.3.2 Coning and quartering

This procedure is suitable for all samples down to approximately 1 kg.

a) Use a hard surface large enough to allow ease of access around the whole sample when spread on the surface.

b) Use a cleaned surface or place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface.

c) Spread the soil sample into a conical pile on the surface. When the entire soil sample is on the surface, 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.

d) Flatten the cone so that the height is less than or equal to the height of the shovel, spade or spatula used.

e) Divide the pile into quarters along two lines intersecting at 90° to each other, using one of the following methods:

1) Method 1
    - Place the centre of a sheet-metal cross, made with four blades joined together at the centre at 90° to each other, at the centre of the flattened cone, and press the lower edges of the metal cross through the soil sample. The height and length of the blades forming the cross should be greater than that of the flattened cone.
    - With the metal cross left in position, discard opposite diagonal quarters and brush clean the space that they occupied.
    - Remove the metal cross and mix together the remaining two quarters.
    - Cone and quarter again using the previous stages, until the volume of remaining soil is equal to the desired size of the subsample (but no less than the minimum size of the subsample in accordance with Table A.1).
2) Method 2
    - Quarter the flattened cone along two diagonals intersecting at right angles, using a shovel inserted vertically into the soil.
    - Discard one pair of opposite quarters and shovel the remainder into a stockpile.
    - Check if the mass of the discarded material is equal to half the mass of the (sub)sample before subdivision, allowing a variation of ± 10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, whereafter the subdivision can continue.
    - Repeat the process of mixing and quartering until the volume of remaining soil is equal to the desired size of the subsample (1 kg).
    - Transfer the subsample to an appropriate sample container in accordance to ISO 10381-8.

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).

6.3.3 Riffling

The use of a riffle box is possible when the soil is dry enough to allow free flow of the soil particles through the riffle box. Division of the sample with a riffle box is most often only practical for samples less than approximately 100 kg (but depending on the size of the riffle box).

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

a) Use a hard surface large enough to allow ease of access around the whole sample when spread on the surface.

b) Use a cleaned surface or place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface.

c) Spread the soil sample into a conical pile on the surface. When the entire soil sample is on the surface, 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.

d) Check that the slot widths of the riffle box are at least three times larger than the maximum particle size of the soil to be subsampled (24 mm).

e) Using a shovel or container, pour the material into the riffle box. It is essential that the soil be poured evenly over the whole riffle, in order to prohibit biased subsampling.

f) Remove one subsample as the reduced sample, discarding the remaining material.

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

h) Repeat the process of riffling until the volume of remaining soil is equal to the desired size of the subsample (1 kg).

i) Transfer the subsample to an appropriate sample container in accordance with ISO 10381-8.

6.3.4 Application of Tyler divider

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

NOTE If the sample is smaller than 17 kg, it can be necessary to combine some of the obtained subsamples to make up a sample of 1 kg.

The mechanical feed should be set at a constant rate suitable for the material being sampled and as identified in the sampling plan. This implies the requirement for the hopper width to be equal to that of the sloping plate and a gate of variable height.

- Check that the slot width of the Tyler divider is at least three times larger than the maximum particle size (24 mm).
- Start the division process by pouring the sample into the divider with a constant rate and catch the subsample(s) in (an) appropriate sample container(s).
- When necessary, repeat the process of subsampling by using one or more of the resulting subsamples, until a subsample of the required size is obtained (but is no less than the minimum size: 1 kg).
- Transfer the subsample to an appropriate sample container, in accordance with ISO 10381-8.

6.3.5 Application of rotating divider (mechanized turntable)

The rotating divider comprises a number of prismatic containers, of equal size, mounted round the periphery of a circle which pass under the falling stream of the sample fed from a hopper mounted above the turntable, and off-set from the centre.

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

- Check that the slot width of the turntable is at least 24 mm.
- Transfer the soil with a constant speed into the turntable. The speed should be relatively low, in order to allow all particles to fall freely into the slot of the turntable, and it will take a large number of rotations of the turntable before the full amount of soil is transferred into the slot.
- After completion of the division process, one or more of the subsample(s) 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.
- One subsample obtained is (if necessary) divided again, until a subsample of the required size is obtained, or until the minimum sample size (1 kg) is reached.
- Transfer the subsample to an appropriate sample container, in accordance with ISO 10381-8.

7 Test report

The test report may be prepared along with the related pretreatment and/or analytical determination or separately, and shall contain at least the following information:

a) a reference to this International Standard, i.e. ISO 23909;

b) a complete identification of the sample;

c) a date of receipt of the sample;

d) details on the applied division technique;

e) the weight and a description of the 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 details not specified in this International Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

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

For a granular material of which each particle possesses a certain characteristic to the same degree, no minimum sample size is required in order to determine this characteristic. In soil samples, it is often the case that only a fraction of the particles contains a characteristic. In that case, the sample which is to be analysed should have a minimum size which is regarded as sufficiently characteristic for the whole sample. The minimal sample size can be calculated using Equation (A.1). For more details and the assumptions made, see ISO 10381-8:2006, Annex B.

$$m_{\text{min. s}} = \frac{1}{6} \pi \left(D_{95}\right)^3 \rho_{\text{p}}\, c\, \frac{\left(1-p\right)}{\left(\text{CV}_{\text{f e}}\right)^2 w_{\text{p}}} \qquad \text{(A.1)}$$

where

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

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

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

c is the correction factor for the use of D_{95} instead of the maximum particle size of the material to be sampled;

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

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

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

For the sampling of soil, in most cases the following estimates of the factors in Equation (A.1) are used:

$\rho_{\text{p}} = 2{,}6$ g/cm^3;

$c = 0{,}25$;

$w_{\text{p}} = 0{,}02$;

$\text{CV}_{\text{f e}} = 0{,}1$.

Using Equation (A.1) and the given assumptions, the minimum samples in Table A.1 can be calculated.

Table A.1 — Minimum size of subsamples as a function of the maximum size of macro-aggregates or particles present in the sample

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

Bibliography

[1] ISO 3534-2:2006, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

[2] ISO 10382, *Soil quality — Determination of organochlorine pesticides and polychorinated biphenyls — Gas-chromatographic method with electron capture detection*

[3] ISO 11074:2005, *Soil quality — Vocabulary*

[4] ISO 13877, *Soil quality — Determination of polynuclear aromatic hydrocarbons — Method using high-performance liquid chromatography*

[5] ISO 15009, *Soil quality — Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons — Purge-and-trap method with thermal desorption*

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