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Soil quality — Pretreatment of samples for physico-chemical analysis

Qualité du sol — Prétraitement des échantillons pour analyses physico-chimiques

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Foreword

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

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

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

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

ISO 11464 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

This second edition cancels and replaces the first edition (ISO 11464:1994), which has been technically revised.

Soil quality — Pretreatment of samples for physico-chemical analysis

1 Scope

This International Standard specifies the pretreatments required for soil samples that are to be subjected to physico-chemical analyses of stable and non-volatile parameters and describes the following five types of pretreatment of samples: drying, crushing, sieving, dividing and milling.

The pretreatment procedures described in this International Standard are not applicable if they affect the results of the determinations to be made. This International Standard is also not applicable to samples when volatile compounds are measured. In general, International Standards for analytical methods will state when it is necessary to adopt other procedures.

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

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

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

3 Principle

Soil samples are dried in air, or in an oven at temperature not exceeding 40 °C, or freeze-dried (see 5.3). If necessary, the soil sample is crushed while still damp and friable, and again after drying (see 5.4). The soil is sieved and the fraction smaller than 2 mm is divided into portions mechanically, or by hand, to enable representative subsampling for analysis (see 5.5). If small subsamples (< 2 g) are required for analysis, the size of the particles of the fraction smaller than 2 mm is further decreased (see 5.6). The necessary procedures are given in the flow diagram in Figure 1.

A drying temperature of 40 °C in an oven is preferable to air drying at room temperature because the increased speed of the drying limits changes due to microbial activity.

It should be noted that every type of pretreatment will have an influence on several soil properties.

The sieve aperture size of 2 mm is generally used. However, before the pretreatment is started, it should be checked if any of the analytical methods to be applied later require other sieve sizes.

NOTE Storing soil samples, including samples that are as received, air dried, refrigerated or stored in the absence of light, for a long time may have an influence on a number of soil parameters, especially solubilities of both inorganic and organic fractions. See Reference [1].

Special measures should usually be taken for samples from contaminated soils. It is important to avoid contact with the skin and special provisions should be taken when drying such samples (air discharge, ventilation, etc.).

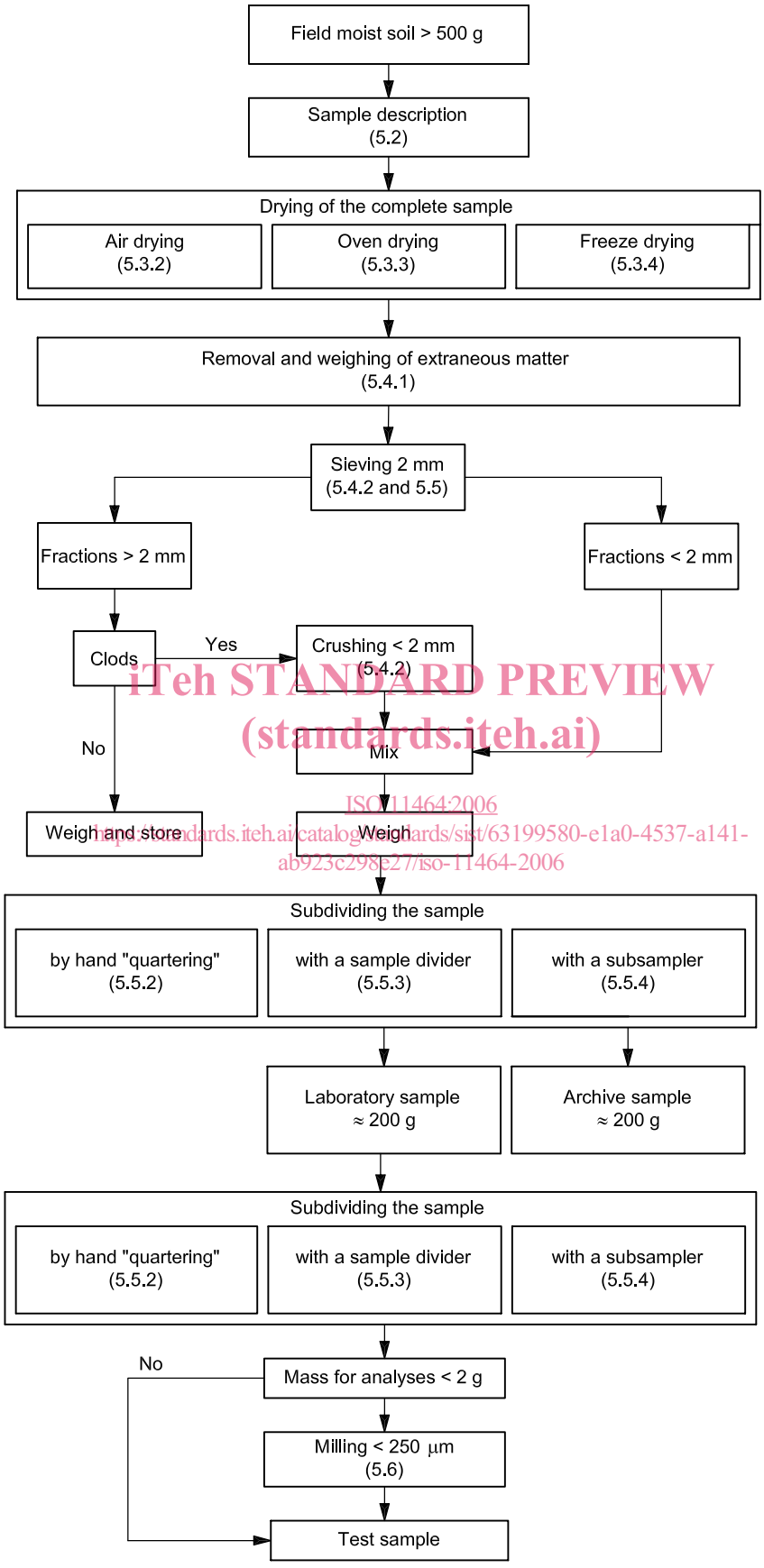

Figure 1 — Diagram for sample pretreatment

Samples may be hazardous because of the presence of chemical contaminants, fungal spores, or pathogens such as leptospirosis, and appropriate safety precautions should be taken.

In this International Standard, it is generally assumed that at least 500 g of fresh soil is available.

Keeping an archive sample (see Figure 1) is optional and should be clearly stated in the overall description of the investigation programme.

4 Apparatus

It is essential that the apparatus used does not add or remove any of the substances under investigation (e.g. heavy metals). If the use of certain equipment and/or materials is not permitted in pretreatment of samples required for a particular physico-chemical analysis, this shall be mentioned in the relevant International Standards on analysis (see Note).

4.1 Drying oven, thermostatically controlled, with forced ventilation and capable of maintaining a temperature not exceeding 40 °C.

4.2 Freeze-drier, optional.

4.3 Crusher(s), mill(s), mortar and pestle, wooden or other soft-faced hammer (see Note).

4.4 Plate sieve, complying with ISO 565, with an aperture of 2 mm.

4.5 Mechanical mixer(s).

4.6 Mechanical sieve shaker, optional (see Note).

4.7 Subsampler or **sample divider** (see Note).

4.8 Mesh sieve, complying with ISO 565, with apertures of 250 µm or of the size specified in the relevant test method.

4.9 Analytical balance, readable and accurate to 0,1 g.

4.10 Balance, readable and accurate to 1 g.

NOTE The apparatus to be used is not specified in detail, although drawings of some suitable items of equipment are provided in Figures A.1 to A.4. Most comparable national standards contain detailed equipment specifications and these may be used, provided they meet the basic performance requirements indicated in this International Standard.

5 Procedure

5.1 General

The procedures for drying, fraction separation and size reduction are set out in 5.3 and 5.4. At several stages in the procedure, the analyst will be required to make decisions, referring in particular to whether size fractions are to be combined or treated separately: this will depend on the nature of the soil and the objectives of the analytical programme.

The sample shall be rehomogenized after any separation, sieving, crushing or milling operation (that may have resulted in segregation of different sized particles) has been carried out.

WARNING — Take special precautions with samples from potentially hazardous soil. Avoid any contact with the skin and make special provisions concerning drying (air discharge, ventilation, etc.).

Care should be taken to avoid contamination of the sample via the air or by dust (e.g. from the ambient laboratory atmosphere or between samples stored or processed close to one another).

It is recommended that pretreatment of soil material always be performed in a room used only for this purpose and remote from locations where analytical measurements are made.

NOTE If the sample has a dust-like consistency, part of it may be lost, and this may alter its physico-chemical properties.

5.2 Sample description

Examine the sample as received and record the description including details of extraneous matter, remains of vegetation, and other noticeable or relevant features.

5.3 Drying

5.3.1 General

Dry the complete sample in air or in a ventilated drying oven from which the moist air is removed or in a freeze dryer. Depending on the chosen method of drying, follow the procedure set out in 5.3.2, 5.3.3 or 5.3.4. Dry until the loss in mass of the soil sample is not larger than 5 % (mass fraction) per 24 h. After the drying process has been completed, determine and record the total mass of the dried sample.

To accelerate the drying process, break down the size of larger aggregates (larger than 15 mm) during the process. When samples are dried in air, crush them lightly by hand using a wooden hammer or a mortar and pestle, taking great care to avoid contamination. When samples are dried in an oven, remove them temporarily from the oven and treat them in the same way. This procedure also makes it easier to separate the particles larger than 2 mm.

Freeze-drying has the advantage that the sample to be dried rarely dries into clods; it usually breaks up into parts.

The drying time depends on the type of material, the thickness of the layer, the initial moisture content of the material and of the air, and on the rate of ventilation. In a drying oven, the drying time for sandy soils is usually not more than 24 h and for clay soils more than 48 h. For soils containing a large proportion of fresh organic matter (e.g. plant roots, etc.), 72 h to 96 h may be required.

5.3.2 Air drying

Spread all the material, in a layer not thicker than 5 cm, on a tray which does not absorb any moisture from the soil and which does not cause contamination.

It is essential that direct sunlight be avoided and the temperature does not exceed 40 °C.

NOTE Direct sunlight could create large temperature differences in the sample, especially between the partly or completely dried top layer and the lower layers.

5.3.3 Oven drying

Spread all the material, in a layer not thicker than 5 cm, on a tray made of material which does not absorb any moisture from the soil and which does not cause contamination. Put the tray in the drying oven (4.2) and dry at a temperature that does not exceed 40 °C.

5.3.4 Freeze-drying

Freeze-drying shall be performed according to ISO 16720.

5.4 Crushing and removal of coarse materials

5.4.1 Separation of stones, etc.

Before crushing, which isnecessary if soil samples have dried into large aggregates, extraneous matter, such as stones, fragments of glass and rubbish, should be removed from the dried sample. This process may be facilitated by the use of a 2 mm sieve (4.5) and by hand picking (see the following paragraph). Care should be taken to minimize the amount of fine material adhering to the extraneous matter removed. Determine and record the mass of any matter removed at this stage.

If the material under examination is a contaminated soil or waste, the analyst may wish to crush the complete sample, including, for example, pieces of slag, to pass the 2 mm sieve.

5.4.2 Crushing

If a 2 mm sieve has been used to facilitate removal of extraneous matter, any large dried particles remaining on the 2 mm sieve should be crushed (using suitable apparatus) to smaller than 2 mm. The apparatus used should be adjusted so that any crushing of the large particles is minimized to enable crushed particles to pass through the 2 mm sieve.

If a 2 mm sieve has not been used to facilitate removal of extraneous matter, then the dried sample should be sieved through a 2 mm sieve. Any large dried particles remaining on the 2 mm sieve should be crushed (using suitable apparatus) to smaller than 2 mm. The apparatus used shall be adjusted in such a way that complete crushing of particles larger than 2 mm before drying is minimized.

The whole sample passing through the 2 mm sieve should be weighed and well mixed.

If the fraction of aggregates larger than 2 mm is low, it may be more efficient to sieve out particles smaller than 2 mm prior to crushing.

In special cases, the entire sample may be crushed.

Fractions may be recombined after crushing using a mechanical mixer (4.6).

5.5 Subsampling

5.5.1 General

Subsampling is necessary when the sample cannot be stored (laboratory sample and archive sample) or used (test sample) completely, because of its size. For the preparation of a laboratory sample, divide the dried, crushed and sieved sample (now $<$ 2 mm) into representative portions of 200 g to 300 g according to 5.5.2 or 5.5.3. For the preparation of a test sample, split up the laboratory sample into representative portions until the required sizes of samples are obtained. Avoid the production of dust as much as possible.

In the case of larger sample masses, subsampling methods according to ISO 10381-8 shall be used to reduce the initial sample size.

It may be necessary to mill the material (5.6) between subsampling stages, to ensure homogeneity as the mass of the subsample is decreased. The procedures described in 5.5.2 and 5.5.3 may be used to produce subsamples/test portions of the materials smaller than 2 mm, and not smaller than 2 g in mass.

When replicate analyses are required, it shall be clarified in the overall investigation plan at which stage of subsampling replicates must be separated. The most representative stage would be a very early one.

Select the method of subsampling (5.5.2, 5.5.3 or 5.5.4) according to the nature of the sample, the requirements of the subsequent determinations and the equipment available.