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Soil quality — Pretreatment of samples for physico-chemical analyses

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



Reference number
ISO 11464:1994(E)

Foreword

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

Annexes A and B of this International Standard are for information only.

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International Organization for Standardization

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Soil quality — Pretreatment of samples for physico-chemical analyses

1 Scope

This International Standard specifies the pretreatments required for soil samples that are to be subjected to physico-chemical analyses 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. In general, International Standards for analytical methods will state when it is necessary to adopt other procedures.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

3 Principle

Soil samples are dried in the air, or in an oven at a

temperature not exceeding 40 °C, or freeze-dried (see 5.2). If necessary, the soil sample is crushed while still damp and friable and again after drying (see 5.3). The soil is sieved (see 5.4) 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.

NOTES

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.

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

3 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 [1].

4 Special measures should usually be taken for samples from contaminated soils. It is important to avoid contact with the skin and special measures should be taken when drying such samples (ventilation, air removal, etc.). Samples may be hazardous because of the presence of chemical contaminants etc., fungal spores, or pathogens such as leptospirosis, and appropriate safety precautions should be taken.

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

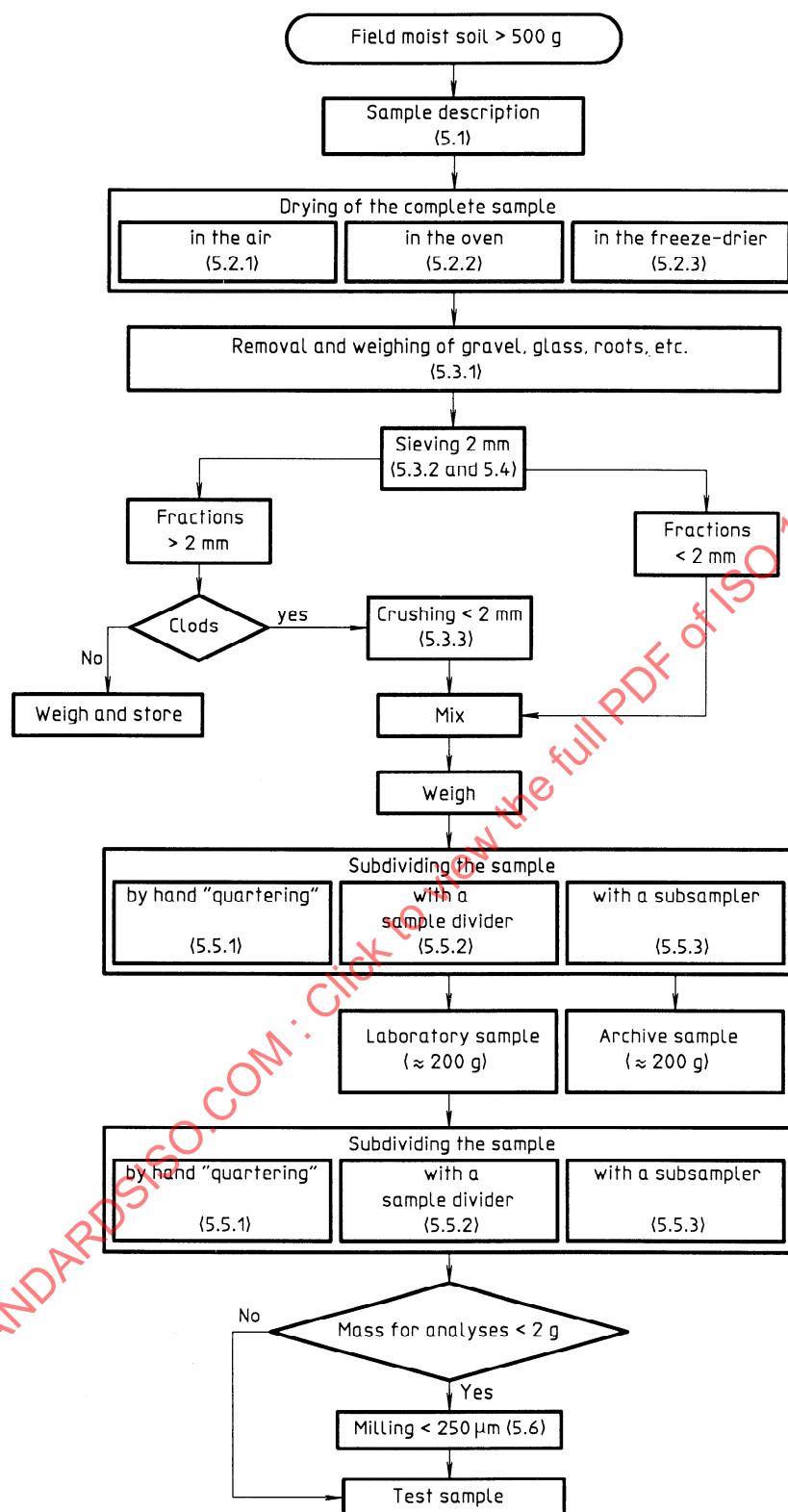


Figure 1 — Diagram for sample pretreatment

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 particular physico-chemical analysis, this should be mentioned in the relevant International Standards on analysis (see note 6).

4.1 Drying oven, thermostatically controlled, with forced ventilation, capable of maintaining a temperature of $40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

4.2 Freeze-drier, optional.

4.3 Crusher(s), mill(s), mortar and pestle, wooden or other soft-faced hammer (see note 6).

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 6).

4.7 Subsampler or sample divider, (see note 6).

4.8 Mesh sieve, complying with ISO 565, with apertures of $250\text{ }\mu\text{m}$ 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 6 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

The procedures for drying, fraction separation and size reduction are set out in 5.2 and 5.3. 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 (discharge of air, ventilation etc.).

NOTES

7 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).

8 It is recommended that pretreatment of soil material always be performed in a separate room used only for this purpose.

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

5.1 Sample description

Examine the sample as received and record the description according to a nationally, or preferably internationally, accepted terminology, including details of extraneous matter, remains of vegetation, and other noticeable or relevant features.

5.2 Drying

Dry the complete sample in air or in a ventilated drying oven from which the moist air has been removed or in a freeze dryer. Depending on the chosen method of drying, follow the procedure set out in 5.2.1, 5.2.2 or 5.2.3. Dry until the loss in mass of the soil sample is not greater than 5 % (m/m) per 24 h.

To accelerate the drying process, break down the size of larger clods (greater 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. 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 greater than 2 mm.

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

5.2.1 In air

Spread all the material, in a layer not thicker than 15 mm, 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.

NOTE 10 Direct sunlight could create large temperature differences in the sample, especially between the partly or completely dried top layer and the lower layers which are still wet.

5.2.2 In a drying oven

Spread all the material, in a layer not thicker than 15 mm, on a tray which does not absorb any moisture from the soil and which does not cause contamination. Put the tray in the drying oven (4.1) and dry at a temperature that is not higher than 40 °C.

5.2.3 Freeze-drying

Dry all the material in the freeze-drier (4.2) according to the procedures recommended by the manufacturer of the apparatus.

NOTE 11 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 normally 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 Crushing and removal of coarse materials

5.3.1 Separation of stones, etc.

If soil samples have dried into clods, crushing is necessary. Before crushing, remove stones, fragments of glass and rubbish etc., which are larger than 2 mm by sieving (5.4) and hand picking (see note 12). Take care to minimize the amount of fine material adhering to the separated stones, etc. Determine and record the total mass of the dried sample, and the mass of any material removed at this stage.

NOTE 12 If the material under examination is a contaminated soil or waste, the analyst may wish to grind the complete sample, including for example pieces of slag, to pass the 2 mm mesh sieve.

5.3.2 Separation of material "naturally" less than 2 mm

After removal of extraneous matter, either

- a) sieve out the material smaller than 2 mm, recording the masses greater and smaller than 2 mm, crush the material greater than 2 mm (see 5.3.3) and recombine the fractions using a mechanical mixer (4.5); or

- b) crush the entire sample (see 5.3.3).

5.3.3 Size reduction of material greater than 2 mm

Crush the dried soil into particles no larger than 2 mm using suitable apparatus (4.3). The necessary apparatus shall be adjusted or used in such a way that crushing of the original particles (concretions and conglomerates) is minimized.

5.4 Sieving

Sieve the dried and crushed sample by hand or using a mechanical shaker. Remove and weigh stones and fresh plant fragments, glass, etc. from the fraction remaining on the sieve (see 5.3.1). Crush any clods left behind on the sieve separately and return them to the sample. Collect all or part of the material left on the sieve and treat it separately if required (see note 12). Take care to minimize the amount of fine material adhering to the separated stones, etc.

5.5 Subsampling

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 5.5.1 or 5.5.2 or another suitable procedure. 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.

NOTE 13 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 5.5.1 and 5.5.2 may be used to produce subsamples/test portions of the materials less than 2 mm and not less than 2 g in mass.

Select the method of subsampling (5.5.1, 5.5.2 or 5.5.3) according to the nature of the sample, the requirements of the subsequent determinations and the equipment available.

5.5.1 Subsampling by hand (quartering)

Mix the soil sample thoroughly using a suitable mechanical mixer (4.5) and spread it into a thin layer on a tray of a type which will not influence the composition of the sample. Separate the soil into four equal portions (quadrants). Combine two of the four portions diagonally, rejecting the other two. Repeat

this procedure until the desired amount of soil is obtained.

5.5.2 Use of the sample divider

A suitable example of a sample divider of the multiple-slot type (riffle box) is shown in figure A.2. This splits the sample into two equal parts.

NOTE 14 The dimensions of the equipment should be chosen in such a way as to suit the amount and particle size of the materials to be divided (see figure A.2 and table A.1).

5.5.3 Mechanical subsampling

A variety of appropriate equipment for subsampling is available, often manufactured according to national standards. These may be used for subsampling in accordance with the appropriate national standard and the manufacturer's instructions.

An example of mechanical subsampling equipment is illustrated in figure A.3. This operates according to the following procedure.

Pour the soil sample into the funnel of the subsampler (figure A.3) and screw the sample bottles into place. Start the subsampler. After subsampling, pour the contents of the bottles into other sample containers. Repeat this procedure, if necessary, with the contents of one of the containers until the desired amount of soil is obtained. The material should be rehomogenized between each stage of subsampling. The contents of more than one container may be thoroughly mixed and used for subsequent phases of the subsampling routine.

5.6 Milling

If a test sample of less than 2 g is to be taken for the analysis, it is essential to further comminute the fraction less than 2 mm.

Mill a representative subsample (see 5.5) of the dried, pulverized and sieved soil until the complete subsample just passes through a sieve of 250 μm or a size otherwise specified in the test method (see 4.8).

If more than one analysis is to be made, sufficient material should be ground to the smallest particle size specified, to enable all the analyses to be made on this one subsample.

NOTES

15 For the determination of some parameters based on chemical extractions, milling is not permitted because it increases the specific surface and thus the reactivity of the sample.

16 If required, the fraction greater than 2 mm can be milled and mixed with the fraction less than 2 mm before chemical analysis is performed.

6 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) which processes, procedures and apparatus were used, including the drying temperature;
- c) a complete identification and description of the sample, including the presence (and if necessary relative masses) of stones, fragments of glass, detritus, etc., odour (if any) and colour;
- d) any details not specified in this International Standard or which are optional, and any other factors which may have affected the results.

Annex A

(informative)

Examples of apparatus

Examples of apparatus given in clause 4 are illustrated in figures A.1 to A.4 .

The design of a mechanical sample divider illustrated in figure A.2 has been found satisfactory but alternative designs may be employed, provided that the essential requirements are fulfilled.

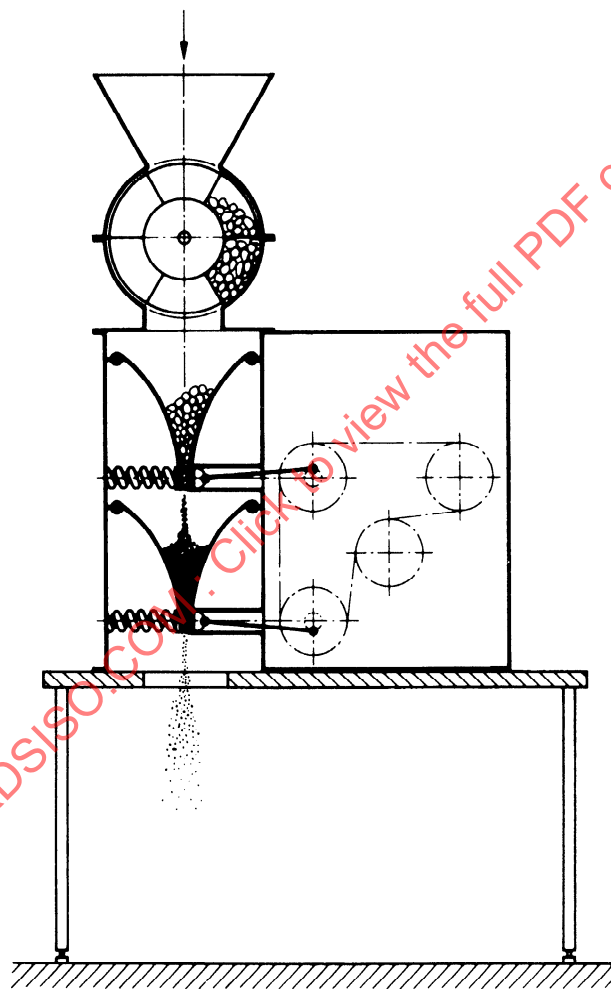


Figure A.1 — Example of a mechanical soil crusher

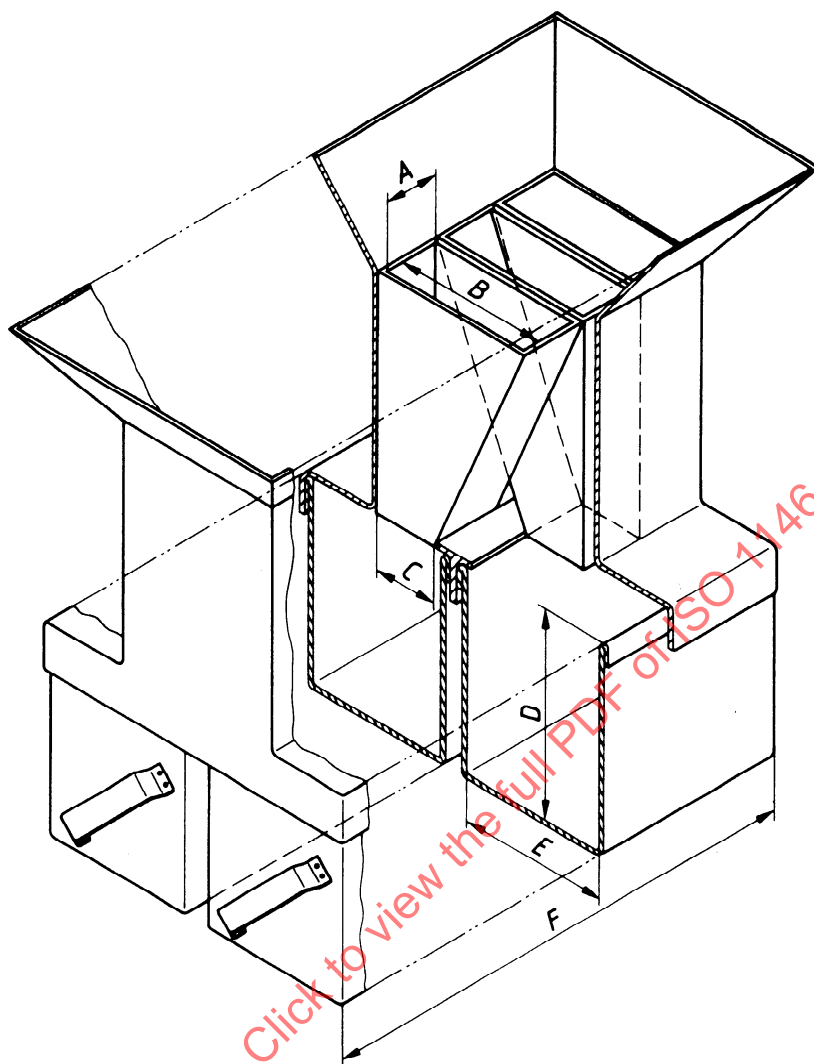


Figure A.2 — Example of a mechanical sample divider

Table A.1 — Dimensions of a mechanical sample divider

All dimensions, except A, are approximate only.

Maximum size of sample mm	Number of slots	Internal dimensions			Internal dimensions of the boxes (three required)		
		A mm	B mm	C mm	D mm	E mm	F mm
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