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Soil quality — Pretreatment of samples for determination of organic contaminants

Qualité du sol — Prétraitement des échantillons pour la détermination des contaminants organiques

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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 14507 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

The properties of organic micropollutants can differ greatly according to chemical species:

- they can range from non-volatile to very volatile compounds (low to high vapour pressure);
- they can be labile or reactive at ambient or elevated temperatures;
- they can be biodegradable or UV-degradable;
- they can have considerably different solubilities in water;
- they require different analytical procedures.

Because of these differences, a general pretreatment procedure cannot be proposed. The goal of a pretreatment procedure is to prepare a test sample in which the concentration of the contaminant is equal to the concentration in the original soil, provided, however, that this procedure does not alter the chemical species to be analysed. For instance, if the sample contains only small particles and the contaminant is homogeneously distributed, it is not necessary to grind the sample. In this International Standard, the size 2 mm is used to distinguish between small and large soil particles.

Consistency among the following aspects is important:

- soil diversity;
- the aim of the analysis (including its accuracy);
- the nature of the chemical species to be analysed.

The particle size distribution of the sample in relation to the mass of sample taken for analysis is also important to pretreatment. For the analysis of organic contaminants, the sample mass taken in most cases is about 20 g.

With such a sample mass, and provided that the contaminant is homogeneously distributed and the particles in the sample are smaller than about 2 mm, further grinding of the sample is not necessary. If the sample contains large particles or if the contaminant is heterogeneously distributed (as occurs for instance with tar particles), it is not possible to take a representative test sample of about 20 g without grinding the sample. To improve the homogeneity, samples are ground to a size smaller than 1 mm. Prior to analysis, very often no information about the distribution of the contaminant in the soil is known.

Some analytical procedures start with a field-moist sample. Drying of the sample gives lower extraction results. If the sample is not dried, grinding is not possible.

In a situation in which accurate results are needed, the best available pretreatment procedure should be used. If it is necessary to establish whether the concentration is above a certain limit and it is already known that the soil is heavily polluted, the simplest pretreatment procedure may meet the need. In this case however, the result may not be presented as a representative value for the whole sample.

The choice depends above all on the volatility of the organic compounds under analysis. It also depends on the soil particle size distribution, the heterogeneity of the sample and the analytical procedure.

INTERNATIONAL STANDARD **ISO 14507:2003(E)**

Soil quality — Pretreatment of samples for determination of organic contaminants

1 Scope

This International Standard specifies three methods for the pretreatment of soil samples in the laboratory prior to the determination of organic contaminants:

- if volatile organic compounds are to be measured;
- if moderately volatile to non-volatile organic compounds are to be measured, if the result of the subsequent analysis must be accurate and reproducible, and if the sample contains particles larger than 2 mm and/or the contaminant is heterogeneously distributed;
- if non-volatile organic compounds are to be measured and the extraction procedure prescribes a field-moist sample, or if the largest particles of the sample are smaller than 2 mm and the contaminant is homogeneously distributed. This procedure is also applicable if reduced accuracy and repeatability are acceptable.

The pretreatment described in this International Standard is used in combination with an extraction procedure in which the contaminant is available for the extraction liquid.

NOTE For the pretreatment of soil samples for the purposes of determining non-volatile inorganic compounds and physico-chemical soil characteristics, see ISO 11464.

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 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 11074-2, *Soil quality — Vocabulary — Part 2: Terms and definitions relating to sampling*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 11074-2 and the following apply:

3.1
volatile compound
organic compound having a boiling point below 300 °C (at a pressure of 101 kPa)

NOTE 1 This includes volatile aromatic and volatile halogenated hydrocarbons as determined in accordance with ISO 15009. Some mono- and dichlorophenols, for instance, and naphthalene also belong to this group.

NOTE 2 The selection of the categories for volatile and moderately volatile compounds can be related in principle to the vapour pressure. However, as the vapour pressure of only a small number of compounds is known, and in view of the relationship between vapour pressure and boiling point, the boiling point has been chosen as the criterion for distinction. See Annex A.

3.2
moderately volatile compound
organic compound having a boiling point above 300 °C (at a pressure of 101 kPa)

NOTE 1 This definition includes:

a) mineral oil (see ISO/TR 11046);

b) most polycyclic aromatic hydrocarbons (PAH) (see ISO 13877);

c) polychlorobiphenyls (PCB) (see ISO 10382);

d) organochlorine pesticides (see ISO 10382).

NOTE 2 In ISO/TR 11046, mineral oil is defined as the group of hydrocarbons which, on chromatography, have retention times lying between that of n-decane ($C_{10}H_{22}$) and n-tetracontane ($C_{40}H_{82}$). As in this International Standard the limit for volatile compounds lies at a boiling point of 300 °C [approximately hexadecane (C_{16})], this means that mineral oil should be considered as a volatile compound, as part of the compounds to be determined fall within the boiling point range of volatile compounds. However for practical reasons, it has been decided that the pretreatment for moderately volatile compounds should be specified for the determination of mineral oil. As a result of cryogenic crushing, an improvement in the extraction yield occurs for compounds with a boiling point above 300 °C. The possible losses for the lower-boiling hydrocarbons (C_{10} to C_{16}) are assumed to be low, due to the retaining effect of the higher-boiling hydrocarbons present in mineral oil and to be compensated by the higher extraction yield of the other hydrocarbons present. As the total yield is used to determine the mineral oil as a group parameter, it is assumed that pretreatment using the method for moderately volatile compounds gives the best results at present.

4 Principle

4.1 Sampling

Sampling shall be carried out in accordance with ISO 10381-1. The samples taken shall be kept cool and processed as soon as possible. The method of pretreatment depends on the volatility of the organic compound(s) or group(s) of organic compounds to be determined.

When taking a subsample, the quantity of soil should be removed from the sample container in such a way that this quantity:

a) is a subsample over the full depth of the sample, and

b) can be taken quickly.

4.2 Volatile compounds

For determination of volatile organic compounds, core test samples are taken from the sample and extracted according to the relevant specific analytical procedure. If composite samples are required, extracts of individual samples are mixed. It is not possible to obtain composite samples without severe losses of volatiles. The procedure is described in 8.2.

4.3 Moderately volatile compounds

Samples are chemically dried at a low temperature (– 196 °C, liquid nitrogen). The dried samples are cooled with liquid nitrogen and ground with a cross-beater mill with a sieve of 1 mm mesh size (cryogenic crushing). After grinding, suitable test portions are processed according to the relevant specific analytical procedures. Composite samples can be prepared by mixing the ground samples. This procedure is described in 8.3.

If the extraction procedure requires a field-moist sample, drying and grinding are not possible.

If the original sample only contains a small fraction of particles greater than 2 mm and the distribution of contaminants is likely to be homogeneous, grinding may be omitted. In these two cases, suitable test portions are taken directly after mixing the sample. This procedure is described in 8.4.

NOTE 1 To distinguish the volatile organic compounds from the moderately volatile organic compounds, boiling points are used instead of the vapour pressure at ambient temperature. This is explained in Annex A, which also gives boiling points and vapour pressures of compounds regularly determined in soil investigations.

NOTE 2 For some specific components in the group of moderately volatile organic compounds, freeze-drying may give good results. In this International Standard freeze-drying is not described.

5 Reagents

Use only reagents of recognized analytical quality. Check samples of each batch of the reagents for the presence of contaminating compounds.

5.1 Sodium sulfate, anhydrous.

Heat the sodium sulfate before use for at least 6 h at about 550 °C to remove crystalline water and organic materials. After heating, allow to cool in a desiccator and store in a closed container.

5.2 Magnesium silicate (talcum powder).

5.3 Sand or **gravel.**

Before use, wash the sand or gravel at least twice with an equivalent mass of demineralized water and then heat for 6 h at about 550 °C to remove organic materials.

5.4 Liquid nitrogen.

For safety precautions, see warnings in 8.3.2.

6 Apparatus

Usual laboratory glassware and equipment, and in particular:

6.1 Glass containers with a volume of 750 ml to 1 000 ml, with a wide neck and screw cap with a polytetrafluoroethylene (PTFE) inlay.

6.2 Polyethylene containers with a volume of 750 ml to 1 000 ml, with a wide neck and screw cap. To prevent a significant headspace being formed, do not use larger containers.

6.3 Refrigerator, capable of maintaining a temperature below 10 °C.

6.4 Dewar vessel(s), capable of holding at least one polyethylene container of about 750 ml.

6.5 Cross-beater mill or mill with comparable qualities, with a sieve of mesh size 1 mm and accessories. A cross-beater mill as used in most soil laboratories is suitable for milling soil samples cooled with liquid nitrogen.

6.6 Gloves, suitable for working at low temperatures.

6.7 Oven, suitable for heating to about 550 °C.

6.8 Core cutter or similar instrument, for example apple corer. Depending on the type of soil (sand, clay), different instruments can be used.

7 Preservation and storage

Do not preserve the samples, but analyse them as soon as possible. Between sampling in the field and analysis store the samples below 10 °C, if possible in a refrigerator (6.3). The maximum storage time for a sample kept in the dark below 10 °C depends on the parameter(s) to be determined, but can also depend on the material used for the container. Details can be obtained from the relevant specific analytical standards.

The storage time also depends on possibilities of volatization and biological degradation. If these processes are likely to occur, then the storage time should not exceed four days. In cases with no volatization, poorly biodegradable compounds present and biologically less active soil, longer storage times are possible.

If rapid microbial decomposition of the compounds to be determined can be expected, the sample should be pretreated immediately after receipt at the laboratory. For samples containing volatile organic compounds in particular, the storage time should preferably be as short as possible and analysis carried out as soon as possible, e.g. within 1 day to 2 days.

NOTE Chemically dried and ground samples are generally stable for longer periods if stored in a cool, dark location. Freezing samples could extend the useful storage time.

8 Procedure

8.1 General

The pretreatment method depends on the volatility of the substance(s) or group(s) of substances to be determined. Two categories are distinguished here:

a) Volatile compounds: boiling point < 300 °C.

b) Moderately volatile organic compounds: boiling point > 300 °C:

   1) grinding necessary;

   2) grinding not possible or not necessary.

The method for pretreating samples containing volatile organic compounds is described in 8.2.

The method for pretreating samples containing moderately volatile organic compounds using grinding is described in 8.3. If grinding is not necessary or the required result is only qualitative, the method described in 8.4 is used.

If the pretreatment methods differ for various parameters to be determined, divide the sample before pretreatment into subsamples which are as large as possible.

If it is known in advance that both volatile organic compounds and other parameters are to be determined in a sample, it is essential that a separate sample be taken in the field in accordance with the appropriate International Standard.

For the purposes of calculation of the content of volatile and moderately volatile organic compounds on the basis of dry matter, the content of dry matter shall be determined in accordance with ISO 11465 on a subsample of the original (moist) sample.

8.2 Volatile compounds (boiling point < 300 °C)

8.2.1 General

For samples in which volatile compounds are to be determined, no sample pretreatment is carried out. Take test samples from the sample as soon as possible after sampling, to avoid losses.

Test samples can be taken and extracted in the field. Precautions should be taken to prevent contamination of the extraction liquid. This should be verified using field blanks, which are subject to the same procedures as the samples. Otherwise the sample should be covered with the extraction solution, the container tightly closed and transported to the laboratory under cool conditions, to perform the extraction.

8.2.2 Individual samples

Store the samples in the refrigerator (6.3) until further processing. Using a corer (6.8), take one or more soil cores from the container such that the combined mass of the cores corresponds to the required mass of the test sample (see Clause 2 and Bibliography for the respective International Standards).

If several cores of soil are taken and/or if several test samples are taken from the same container, take the cores from different points in the container, if possible.

8.2.3 Composite samples

It is not possible to form composite samples for the analysis of volatile organic compounds. If the analysis method involves a liquid extract, a composite extract can be prepared by mixing equivalent volumes of the extracts from the different samples.

8.3 Moderately volatile organic compounds (boiling point > 300 °C) — Grinding necessary

8.3.1 General

Remove parts that are not representative of the soil, e.g. all visible metallic content.

A note shall be made to this effect in the analytical report.

8.3.2 Individual samples

8.3.2.1 Chemical drying

For each sample to be analysed, add approximately 200 g of sodium sulfate (5.1) and approximately 50 g of magnesium silicate (5.2) to a glass container (6.1). Determine the total mass of these substances to an accuracy of 0,1 g. After sealing the container, mix the two substances by shaking and cool to a temperature below 10 °C.

For each sample, add approximately 250 g of soil (weighed to 1 decimal point) to the glass container with the sodium sulfate and magnesium silicate.

Close the container and mix the soil and the additives by shaking. Place the container in a refrigerator (6.3) to stand cold for 12 h to 16 h. Shake the container vigorously every hour for the first 4 h to avoid clod formation. Before the end of the drying time, again shake the samples vigorously.

If large clods, e.g. larger than 3 cm, are formed in the initial phase, these can be crushed manually by cutting with a spatula in the container. This may particularly be necessary with heavy clay soil.

If the moisture content is greater than 60 %, add extra sodium sulfate instead of reducing the amount of sample. If a sample has a low moisture content and no clods are formed, it is possible to dry in less than 12 h. An adequate drying time should be ensured.