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Soil quality — Guidance on long and short term storage of soil samples

Qualité du sol — Lignes directrices relatives au stockage des échantillons de sol à long et à court termes

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Foreword

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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 18512 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 2, *Sampling*.

Introduction

Many soil investigation programmes require that soil samples be stored for future use. The choice of storage conditions may determine whether or not the samples will be suitable for the intended future use. This International Standard gives guidance on choosing conditions for storage of soil samples.

Soil quality — Guidance on long and short term storage of soil samples

1 Scope

This International Standard gives guidance on how to store and preserve soil samples for laboratory determinations and how to prepare them for analysis after storage. Special emphasis is given to maximum storage times as a function of different storage conditions.

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.

EN 15192:2006, *Characterisation of waste and soil — Determination of Chromium(VI) in solid material by alkaline digestion and ion chromatography with spectrophotometric detection*

ISO 10301, *Water quality — Determination of highly volatile halogenated hydrocarbons — Gas-chromatographic methods*

ISO 10381-6, *Soil quality — Sampling — Part 6: Guidance on the collection, handling and storage of soil under aerobic conditions for the assessment of microbiological processes, biomass and diversity in the laboratory*

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

ISO 10390, *Soil quality — Determination of pH*

ISO 10694, *Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis)*

ISO 11048, *Soil quality — Determination of water-soluble and acid-soluble sulfate*

ISO 11074, *Soil quality — Vocabulary*

ISO 11259, *Soil quality — Simplified soil description*

ISO 11261, *Soil quality — Determination of total nitrogen — Modified Kjeldahl method*

ISO 11263, *Soil quality — Determination of phosphorus — Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution*

ISO 11265, *Soil quality — Determination of the specific electrical conductivity*

ISO 11266, *Soil quality — Guidance on laboratory testing for biodegradation of organic chemicals in soil under aerobic conditions*

ISO 11267, *Soil quality — Inhibition of reproduction of Collembola (*Folsomia candida*) by soil pollutants*

ISO 11268-1, *Soil quality — Effects of pollutants on earthworms (*Eisenia fetida*) — Part 1: Determination of acute toxicity using artificial soil substrate*

ISO 11268-2, *Soil quality — Effects of pollutants on earthworms (*Eisenia fetida*) — Part 2: Determination of effects on reproduction*

ISO 11466, *Soil quality — Extraction of trace elements soluble in aqua regia*

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

ISO 13878, *Soil quality — Determination of total nitrogen content by dry combustion ("elemental analysis")*

ISO 14154, *Soil quality — Determination of some selected chlorophenols — Gas-chromatographic method with electron-capture detection*

ISO 14238, *Soil quality — Biological methods — Determination of nitrogen mineralization and nitrification in soils and the influence of chemicals on these processes*

ISO 14240-1, *Soil quality — Determination of soil microbial biomass — Part 1: Substrate-induced respiration method*

ISO 14240-2, *Soil quality — Determination of soil microbial biomass — Part 2: Fumigation-extraction method*

ISO 14255, *Soil quality — Determination of nitrate nitrogen, ammonium nitrogen and total soluble nitrogen in air-dry soils using calcium chloride solution as extractant*

ISO/TS 14256-1, *Soil quality — Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution — Part 1: Manual method*

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

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*

ISO 15473, *Soil quality — Guidance on laboratory testing for biodegradation of organic chemicals in soil under anaerobic conditions*

ISO 15685, *Soil quality — Determination of potential nitrification and inhibition of nitrification — Rapid test by ammonium oxidation*

ISO 15799, *Soil quality — Guidance on the ecotoxicological characterization of soils and soil materials*

ISO 15903, *Soil quality — Format for recording soil and site information*

ISO 15952, *Soil quality — Effects of pollutants on juvenile land snails (Helicidae) — Determination of the effects on growth by soil contamination*

ISO 16072, *Soil quality — Laboratory methods for determination of microbial soil respiration*

ISO 16387, *Soil quality — Effects of pollutants on Enchytraeidae (*Enchytraeus *sp.) — Determination of effects on reproduction and survival*

ISO 16703, *Soil quality — Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography*

ISO 17155, *Soil quality — Determination of abundance and activity of soil microflora using respiration curves*

ISO 20963, *Soil quality — Effects of pollutants on insect larvae (*Oxythyrea funesta*) — Determination of acute toxicity*

ISO 22030, *Soil quality — Biological methods — Chronic toxicity in higher plants*

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

ISO 23753-1, *Soil quality — Determination of dehydrogenase activity in soils — Part 1: Method using triphenyltetrazolium chloride (TTC)*

ISO 23753-2, *Soil quality — Determination of dehydrogenase activity in soils — Part 2: Method using iodotetrazolium chloride (INT)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074 and ISO 11259 apply.

In this International Standard, the term “refrigeration” refers to a temperature of 4 °C ± 2 °C. The term “freezing” refers to a temperature lower than –18 °C.

4 General comments on soil storage

Many studies involve the collection of soil samples in the field, followed by laboratory determination of various properties of the collected samples. In general, the samples are taken at the site being investigated, mixed or otherwise treated at the site, packed in containers and then transported to the laboratory. Upon arrival at the laboratory, the samples may again be treated before being sent for analysis. Some samples may be stored directly for later analysis. After analysis, the remaining part of the samples may be discarded or stored. The samples are stored when there is a need for further analysis, either because there is a need for checking parameters already determined or there is a need for making additional determinations in the future.

In practice, there are two main situations in which sample storage is relevant.

— Routine testing of soil samples, e.g. by environmental laboratories, where soil samples typically are stored for a few weeks after sampling in order to carry out some additional tests, or in order to confirm results found earlier.

— Situations in which samples have to be stored for a long period, sometimes over decades, e.g. monitoring programs, reference materials, or research programs in which degradability is tested.

Both these situations fall within the scope of this International Standard.

The conditions for storage should be selected carefully at all stages, from the point of taking the sample onwards. As an unexpected delay in transport may occur, this guidance should be applied even if the planned transportation time is short. Examples of storage conditions to be considered are light, temperature, humidity, accessibility, duration of storage, type of container and amount of sample to be stored. Documentation of the samples and the storage conditions is also important. Risk and security problems should be considered. Well-designed storage conditions are particularly important in large-scale studies, such as monitoring, where the number of samples may become quite large over the years. Incorrectly chosen storage conditions may lead to high costs and may render the samples unfit for future use.

The effect of storage on biodiversity has been considered only with respect to microbiological diversity.

Radioactive change caused by loss or gain of radioactive matter should be considered in connection with the respective compounds. Radioactive decay is generally not affected by storage and is not treated in this International Standard.

5 Change in soil properties during storage

It is helpful to consider the principal biological, chemical and physical phenomena that may cause changes in the samples:

— change in water content;

— biological activity;

— evaporation or precipitation of volatile substances;

— chemical reactions with the atmosphere;

— reactions with the sample container.

Unacceptable changes in soil parameters may occur if these phenomena are not controlled by a proper choice of storage conditions. However, controlling all these phenomena in all samples for a long period of time may turn out to be very costly or impossible. It is therefore important to design the storage conditions to fit the objectives of the study.

It is worth noting that some parameters, for example, the contents of some volatile substances, may not be measurable after storage, regardless of storage conditions. In such cases, serious consideration for the future need for data on such parameters should be given at the outset, and the analysis program adapted accordingly.

6 Storage conditions

6.1 General

This clause contains a list of storage conditions that shall be determined when designing the storage programme.

6.2 Light

Light conditions affect the content of some substances, particularly organics. This should be considered and taken care of, e.g. by using brown glass bottles or keeping the samples in total darkness.

6.3 Temperature

The choice of temperature is always very important as the temperature affects the biological activity in the samples. Temperature is therefore a major factor in the design of a storage facility. In some cases, room temperature will be appropriate but, in many cases, refrigeration or freezing may be required to reduce the biological activity. In very special cases, the temperature of liquid nitrogen will be required.

The need for storage of a few samples at – 80 °C or a lower temperature should be considered, e.g. storage of higher quality reference samples at – 80 °C or at a lower temperature, in order to demonstrate whether or not samples stored at low temperatures are stable.

6.4 Humidity

Moisture will induce microbiological activity or chemical changes in soil samples unless the temperature is very low. The control of humidity is therefore important.

When the samples are not kept in airtight containers, the humidity of the storage facility shall be kept low all year round.

If airtight containers are used, the sample humidity will not change during storage. In this case, it is necessary to ascertain that the original humidity of the samples is low enough to prevent microbiological activity.

6.5 Accessibility, security, documentation and quality control

If the samples are to be analysed urgently, or repeatedly, the storage facility should be easily accessible from the laboratory. This will reduce the time and the risk for quality deterioration during transport to the laboratory.

Security issues, such as fire, theft and destruction, are also important, particularly for samples of great value.

Documentation (see ISO 15903), proper labelling and elimination of cross-contamination are other safety issues to be addressed.

Samples from contaminated land should always be regarded as hazardous and handled accordingly.

A relevant quality control (QC) programme should be introduced. A (certified) reference sample may be used, or a prior analysis on one or more of the freshly taken samples.

6.6 Duration of storage

The required duration of storage is an important element in the storage conditions. As mentioned in Clause 4, some samples are stored only for a few weeks (e.g. for routine environmental testing), other samples for a long period of time. Well-documented long-term “reference” soil samples collected at regular intervals, over many years, could be used to determine the magnitude of any changes in important soil properties. There may also be legal requirements on the duration of storage.

The need for a long duration of storage should always be evaluated versus the cost of storage and documentation.

6.7 Containers and quantity of sample stored

The containers should be carefully chosen regarding the construction material, type of sealing and size. Relevant functions should be validated, e.g. the protection from contamination and the ability to keep the sample protected from light or air. Appropriate cleaning or sterilizing procedures shall be followed.

Many plastic containers will become brittle after five to ten years and glass containers are preferred. However, if the samples contain a high content of water, as many clay samples do, the glass may crack on freezing. The risk of cracking on freezing can be reduced by partial filling of the bottles.

The amount of sample to be stored should be considered. The amount required depends on the planned determinations and may be difficult to calculate. Unless the material is very costly or the need for reanalysis is very unlikely, it is wise to store enough material for at least five determinations of the parameter requiring the largest sample size. In addition, storage of at least 50 g is recommended in order to allow homogeneity.

Once a soil is frozen, it is very difficult to sub-sample for a repeat analysis. Thus, it is wise to freeze a number of smaller sub-samples. Care should be taken to guarantee the homogeneity when sub-samples are prepared.

6.8 Preparing the samples after storage

Appropriate procedures for preparing the samples after storage will depend on the storage conditions and the determinations. It is not possible to give a general specification. Existing standards (e. g. ISO 11464) should be considered.

When a non-frozen soil sample is stored for a long period of time, a vertical redistribution of particles may occur. Remixing in a suitable mixer is advisable. For large samples, remixing in a mixer may not be sufficient. Mixing by spreading the sample in a thin layer on a plastic foil, then repeatedly folding the layer and spreading it out again, is recommended.