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Soil quality - Pretreatment of samples by freeze-drying for subsequent analysis

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Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis

Qualité du sol — Prétraitement des échantillons par lyophilisation pour analyse subséquente

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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 16720 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis

1 Scope

This International Standard specifies a method for pretreatment of soil samples by freeze-drying for subsequent analysis.

This International Standard is applicable to soil samples for subsequent determination of elements or organic compounds recognized as non-volatile under freeze-drying conditions. Generally, this International Standard can also be applied to samples from sludges and sediments.

This method is also applicable as a first step for the determination of dry matter (or water) content, for instance in the case of samples with high water content.

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

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

3 Principle and general requirements

Before freeze-drying, samples shall be cooled below their eutectic points. These are rarely known, but freezing the samples below −40 °C is usually sufficient.

During freeze-drying, water is removed from the sample under vacuum conditions by direct conversion from ice to vapour (sublimation) which is collected in a condenser where it releases its heat energy and turns again into ice. Generally a temperature below −50 °C inside the condenser is suitable for usual applications.

Sublimation requires heat in order to take place within an acceptable range of time. When the frozen sample containers are connected to an external inlet manifold (see 5.1.2), heat energy is supplied by the laboratory atmosphere. In case of a drying chamber equipped with temperature-controlled carriers (see 5.1.1), this energy is provided by heating the carriers.

Vacuum conditions shall allow the generation of vapour pressure difference needed for sublimation and the continuous evacuation of water vapour from the sample. Throughout the drying process, the pressure inside the apparatus and the temperature of the sample(s) shall be such as to avoid sample thawing and loss of any compound of interest. This last point may be difficult to achieve due to the large range of possible energy levels of the bonds between compounds of interest and the solid phase. For compounds known as non-volatile, a final temperature of the sample between −20 °C to −25 °C is suitable. Other final temperatures may be needed for compounds recognized as more volatile. In all cases, the final step shall be as short as possible.

A sample in thin layer form is required for suitable freeze-drying. Whatever the kind of sample container, the thickness of the sample should not exceed 2 cm. If the representative amount exceeds a thickness of 2 cm, multiple drying may be applied.

The quantity of sample treated depends on the representativeness, the dry matter content and the number of subsequent determinations. Sample containers for freeze-drying are selected accordingly.

NOTE Soil samples often need to be dried before analysis. In the case of very humid or clayey soils, the technique of freeze-drying compared with air or oven drying at temperatures < 40 °C in accordance with ISO 11465 has the advantage of a quicker drying process and provision of a dried sample that can be reduced in size more easily.

4 Interferences

Substances may volatilize during freeze-drying and may partly be released to the ice condensate or be found in the gas released to the vacuum pump. If compounds lost due to valatilization are amongst those included in the list of parameters to be determined subsequently, analysis results are then directly affected. In contrast, mass losses only affect the dry matter content and this effect is generally negligible.

NOTE For example, ammonia nitrogen is lost during freeze-drying of sludges, the intensity of loss depending on the dry matter content. Therefore determination of ammonia nitrogen content should be avoided after freeze-drying, at least for liquid sludges, but in such a case the dry matter content is not significantly affected.

In the final step of freeze-drying, samples become pulverulent and turbulences induced by the vacuum pump may carry away smaller solid particles. Samples can be protected by means of a wire mesh.

Achieving total dryness is difficult and may consume too much time, especially when several samples are treated at the same time. In these case, a level of dryness should be achieved which merely allows the samples to be handled, ground and homogenized, and residual water content determined afterwards on a sub-sample.

If not free, water may interact more or less strongly with the solid phase, leading to residual water contents depending on the selected drying method. For a standard determination of dry matter or water content, drying at 105 °C in accordance with ISO 11465, is required after freeze-drying.

5 Apparatus

5.1 Freeze-drying apparatus, composed of a drying device (5.1.1 or 5.1.2) connected to a condensing chamber (condenser), a vacuum system, a cooling system and the appropriate controls.

5.1.1 Thermally isolated drying chamber equipped with temperature controlled carriers.

The temperature of the samples, measured by inserted probes, is controlled by heating or cooling the carriers during freeze-drying. Generally, samples can be frozen on the carriers prior to freeze-drying.

5.1.2 External inlet manifold to which the frozen sample containers (generally flasks, but a few apparatus allow the use of trays instead of flasks) are connected.

Heat energy is supplied by exchange with the laboratory atmosphere.

5.2 Suitable sample containers for freeze-drying, trays or flasks, forms and sizes are depending on the type of apparatus selected or available and on the quantities of sample treated.

5.3 Freezer for pre-cooling of samples, if possible with contact freezing facilities, minimum temperature −35 °C, explosion-protected.

Instructions of the manufacturer shall be followed.