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Soil quality — Dissolution for the determination of total element content —

Part 1:
Dissolution with hydrofluoric and perchloric acids

Qualité du sol — Mise en solution pour la détermination des teneurs élémentaires totales —

Partie 1: Mise en solution par l'acide fluorhydrique et l'acide perchlorique

Reference number
ISO 14869-1:2001(E)

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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 3.

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 part of ISO 14869 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14869-1 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

ISO 14869 consists of the following parts, under the general title *Soil quality — Dissolution for the determination of total element content*:

Part 1: Dissolution with hydrofluoric and perchloric acids

Part 2: Dissolution by alkaline fusion

Soil quality — Dissolution for the determination of total element content —

Part 1:
Dissolution with hydrofluoric and perchloric acids

WARNING — Several stages in the procedure of this part of ISO 14869 are potentially hazardous, especially those involving hydrofluoric and perchloric acids. Users should familiarize themselves with the necessary safety precautions and, where appropriate, any legal requirements for their use. If in doubt, seek professional advice.

1 Scope

This part of ISO 14869 specifies a method for the complete dissolution, using hydrofluoric and perchloric acids, of the following elements in soils:

— Al, Ba, Cd, Ca, Cs, Cr, Co, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, Pb, Sr, V, Zn.

This procedure may be appropriate for the subsequent determination of other elements provided their concentrations are high enough relative to the sensitivity of the methods of measurement.

The low acid concentration of the final solution allows the use of a large range of analytical devices and the volatilization of silicon simplifies analytical procedures.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14869. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14869 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analysis.*

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

ISO 14869-2:—[1], *Soil quality — Dissolution for the determination of total element content — Part 2: Dissolution by alkaline fusion.*

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[1] To be published.

3 Principle

The dried and ground sample is pre-treated to destroy organic matter, and then digested with a mixture of hydrofluoric and perchloric acids. After evaporation to near dryness, the residue is dissolved in dilute hydrochloric or nitric acid.

Hydrofluoric acid decomposes silicates by the reaction of F with Si to form volatile SiF_4. As it evaporates last, perchloric acid forms readily-soluble perchlorate salts.

To minimize the danger of acid ejection due to violent oxidation of organic matter by perchloric acid, two alternative procedures have been adopted to destroy organic matter prior to digestion:

- dry ashing at 450 °C (see 6.1.2);
- pretreatment with nitric acid (see 6.1.3).

NOTE The method using dry ashing for the destruction of organic matter has been applied to reference soils and compared with other methods for the determination of total content. No significant loss of elements has been detected but, when the presence of volatile organometallic compounds is suspected, only nitric acid pre-treatment should be used.

4 Reagents

The reagents used shall meet the purity requirements of the subsequent analysis.

4.1 Water, complying with grade 2 of ISO 3696.

4.2 Hydrofluoric acid, $c(\mathrm{HF}) = 27{,}8\ \mathrm{mol \cdot l^{-1}}$, $\rho = 1{,}16\ \mathrm{g \cdot ml^{-1}}$

4.3 Perchloric acid, $c(\mathrm{HClO_4}) = 11{,}6\ \mathrm{mol \cdot l^{-1}}$, $\rho = 1{,}67\ \mathrm{g \cdot ml^{-1}}$

4.4 Hydrochloric acid, $c(\mathrm{HCl}) = 12{,}1\ \mathrm{mol \cdot l^{-1}}$, $\rho = 1{,}19\ \mathrm{g \cdot ml^{-1}}$

4.5 Nitric acid, $c(\mathrm{HNO_3}) = 15{,}2\ \mathrm{mol \cdot l^{-1}}$, $\rho = 1{,}41\ \mathrm{g \cdot ml^{-1}}$

4.6 Nitric acid, $c(\mathrm{HNO_3}) = 1{,}5\ \mathrm{mol \cdot l^{-1}}$, dilute 100 ml of nitric acid (4.5) with water to make 1 l.

5 Apparatus

5.1 Mill, capable of grinding dried soils without contamination by elements of interest.

5.2 Drying oven and desiccator, for the determination of dry matter conforming to ISO 11465.

5.3 Analytical balance, capable of weighing accurately to 0,000 1 g.

5.4 Crucible, of fused silica or platinum with an approximate capacity of 10 ml. Although more expensive, platinum crucibles of a larger capacity (30 at least) can be used through several consecutive stages of the procedure (see 6.1.2).

5.5 Furnace, which can reach a temperature of 450 °C ± 25 °C within 1 h.

5.6 Evaporating dishes, made from polytetrafluoroethylene (PTFE), with an approximate capacity of 30 ml and a diameter of about 5 cm. After use, the dishes shall be soaked in dilute nitric acid (4.6) overnight and rinsed with water (4.1). The dishes shall be replaced regularly to avoid carry-over due to increased absorption and desorption of elements on the ageing surface. The onset of carry-over shall be monitored by carrying out blank digestions in dishes recently used for the digestion of soil samples.

5.7 Hot plate, set at 150 °C ± 10 °C.

5.8 Fume hood, for hydrofluoric and perchloric acid and with an appropriate washdown facility.

5.9 Volumetric flask, of polypropylene and with a capacity of 50 ml.

6 Procedure

WARNING — Hydrofluoric acid is dangerous if inhaled as vapour or by direct contact with skin and mucosa. The effects of exposure to HF may not be apparent for several hours, particularly for skin, after which effective treatment can be difficult. Users should familiarize themselves with the necessary precautions, taking professional and medical advice where appropriate. The use of an efficient exhaust hood, rubber gloves, glasses or face protection and protected pipetting devices is essential.

6.1 Pretreatment

6.1.1 Sample preparation

Mill a representative portion of the dried sample (prepared in accordance with ISO 11464) as fine as possible in order to obtain a subsample of approximately 20 g. Use a portion of this milled sample to determine the water content in accordance with ISO 11465.

Pretreatments designed to oxidize organic matter (6.2) should be followed strictly in order to avoid explosions during the digestion procedure.

NOTE Experience has shown that dissolution of the sample can be considerably prolonged if the material is not ground below 250 µm.

6.1.2 Dry ashing

Weigh precisely 0,250 g of the milled sample by means of the balance (5.3) and transfer to a crucible (5.4). Place the crucible in the furnace (5.5) and allow the temperature to reach 450 °C, progressively over 1 h. Maintain this temperature for 3 h. Allow the furnace to cool to room temperature and transfer the ash quantitatively to a PTFE evaporating dish (5.6) with a minimum amount of water (4.1). Using a platinum crucible of about 30 ml avoids ash being transferred to a PTFE dish and allows digestion to be performed in the same container as dry ashing.

6.1.3 Nitric acid pre-treatment

Weigh precisely 0,250 g of the milled sample by means of the balance (5.3), transfer to an evaporating dish (5.6) and add 5 ml of nitric acid (4.5). Place the dish on the hot plate (5.7) at 150 °C and evaporate until approximately 1 ml of nitric acid remains. Note that several successive additions of nitric acid may be necessary until the emission of nitrous vapours ceases to remove all the organic matter. In such cases, remove the dish from the hot plate and cool to room temperature before adding the next portion of nitric acid.

After the last addition of nitric acid, remove the dish from the hot plate and cool to room temperature before undertaking the digestion.

6.2 Digestion

6.2.1 Add 5 ml of hydrofluoric acid (4.2) and 1,5 ml of perchloric acid (4.3) to the pretreated test portion (see 6.1.1) in the PTFE dish (5.6) or the 30 ml platinum crucible (5.4). Heat this mixture on the hot plate (5.7) until the dense fumes of perchloric acid and silicon tetrafluoride cease. Do not allow the mixture to evaporate to complete dryness. Remove the dish from the hot plate, allow to cool, add 1 ml of hydrochloric acid (4.4) or 1 ml of nitric acid (4.5) and approximately 5 ml of water (4.1) to dissolve the residue. Warm the dish briefly on the hot plate to assist dissolution. Transfer this solution quantitatively to the 50 ml volumetric flask (5.9), fill to the mark and mix well.

A solid phase remaining in the resultant solution indicates incomplete dissolution. It may be of no importance with respect to the elements of interest, especially when pure silica constitutes the solid phase, but in that case, the procedure shall be completed by one of the following stages.

NOTE With regard to the dissolution of the residue, the efficiencies of hydrochloric and nitric acids are the same. The choice between them depends on the analytical methods used afterwards. With ICP-MS, nitric acid is preferred, as chloride may generate various interferences. However, its use shall be avoided in the colorimetric determination of phosphorus by means of sulfomolybdic reagent. In addition, some elements, which are not listed in clause 1, may precipitate when hydrochloric acid is used (e.g. Ag).

6.2.2 The procedure is stopped at this point and failure of total dissolution with a possible effect on the determination of total contents is noted in the test report.

6.2.3 The procedure is adjusted to improve the dissolution. One or a combination of the three following treatments is carried out.

a) The procedure is started again with a new test portion but a further dose of 5 ml of hydrofluoric acid (4.2) and 1,5 ml of perchloric acid (4.3) is added after evaporation of the first one to near dryness. The second dose is also evaporated as above and the procedure is carried on as described in 6.2.1.

b) The procedure is started again with a new test portion but after the addition of hydrofluoric and perchloric acids the mixture is left overnight at room temperature before being heated as described in 6.2.1.

c) The whole procedure is not changed but the mass of the test portion is reduced.

If a solid phase remains in spite of these further treatments, then failure of total dissolution is mentioned in the test report.

6.2.4 Acid digestion is completed by lithium borate fusion in accordance with ISO 14869-2 and the following pre-treatment.

Decant the content of the volumetric flask (5.9) on to an ashless filter paper. Collect the filtrate. Rinse the flask with water in order to transfer quantitatively the residue on the filter. Thoroughly wash this with water and allow it to dry.

The residue and the filter are considered as a test portion and then treated according to the procedure described in ISO 14869-2. Depending on the quantity of remaining residue, the mass of lithium borate and the final volume can be reduced.

Adding the quantities found for a given element after the two dissolution steps described above leads to its total content in the sample.

6.3 Blank test

Use the same procedure, without the sample, to perform at least one blank test within each batch of digestions.

7 Test report

The test report can be made separately or in conjunction with the test report of the subsequent analytical measurement.

The test report shall contain the following information:

a) reference to this part of ISO 14869, i.e. ISO 14869-1;

b) complete identification of the sample;

c) any details not specified in this part of ISO 14869, or which are optional, as well as any factor which may have affected the results, especially information about insoluble residues.

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