INTERNATIONAL STANDARD

ISO 14869-2

First edition
2002-08-01

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

Part 2: Dissolution by alkaline fusion

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

Partie 2: Mise en solution par fusion alcaline

Reference number
ISO 14869-2:2002(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

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 14869-2 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 2:
Dissolution by alkaline fusion

1 Scope

This part of ISO 14869 specifies a method for the dissolution of total contents for the following elements in soils:

Na, K, Mg, Ca, Ti, Mn, Fe, Al, Si.

This list is not exhaustive, and other elements are applicable for determination, provided

— they are not lost during the fusion process,

— $w > (3d \cdot V/m)$

where

w is the mass content of the element, expressed in milligrams per kilogram soil,

d is the detection limit, in milligrams per litre, for the element and analytical method considered,

3 is a conventional factor,

V is the adjusted volume, in litres, of the final solution containing the dissolved sample, and

m is the mass of the test portion, in kilograms,

— the determination is not adversely affected by the high salt concentration in the resultant solution.

The flux proposed in this method is suitable for a wide range of materials, among which soil samples are relatively easy to deal with.

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 analyses*

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

3 Principle

To avoid reduction of metallic oxides to metals, the dried and ground sample is first ignited at 450 °C and afterwards fused with a mixture of dilithium tetraborate (one part) and lithium metaborate (four parts). While still liquid, the melt is poured quantitatively into dilute nitric acid. The suspension is then stirred until the solid phase dissolves completely.

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 Dilithium tetraborate, $Li_2B_4O_7$, apparent density when packed = 1.

4.3 Lithium metaborate, $LiBO_2$, apparent density when packed = 0,8.

4.4 Nitric acid, $c(HNO_3) = 15{,}2$ mol/l, $\rho = 1{,}41$ g/ml.

4.5 Nitric acid, $c(HNO_3) = 0{,}5$ mol/l.

Dilute 32 ml of nitric acid (4.4) with water (4.1) to make 1 l of solution.

5 Apparatus

5.1 Grinding mill, capable of grinding dried soils without contamination by the elements of interest.

5.2 Drying oven and dessicator, for the determination of dry matter in accordance with ISO 11465.

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

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

5.5 Heating device, capable of heating the proposed flux above its melting point.

Several devices are allowed:

— inductively heated furnace;

— muffle furnace;

— air-propane Meker burner.

5.6 Crucible, of about 30 ml capacity, preferably made of platinum:gold (95:5) alloy or vitreous carbon.

Molten borates wet some metals and their alloys. For this reason, the use of the above type of crucible is strongly recommended. Failure to do so can result in loss of melt, and the possibility of carry-over between subsequent dissolutions.

5.7 Usual laboratory glassware, including beakers of 800 ml capacity.

5.8 Magnetic stirrer, with polytetrafluoroethylene (PTFE)-coated follower.

6 Procedure

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 that milled sample to determine the water content in accordance with ISO 11465.

Weigh precisely by means of the balance (5.3) about 0,200 g of the milled sample, and transfer to a crucible (5.6). Place the crucible in the furnace (5.4) and allow the temperature to reach 450 °C progressively during 1 h. Maintain this temperature for 3 h. Allow the crucible to cool to room temperature.

Weigh 0,200 g ± 0,002 g of dilithium tetraborate (4.2) and 0,800 g ± 0,005 g of lithium metaborate (4.3), transfer to the crucible containing the ignited sample and mix thoroughly with a plastics spatula.

Heat the mixture by means of the heating device (5.5) to between 1 000 °C and 1 100 °C until the boron salts melt and the sample dissolves completely. Depending on the type of heating device, dissolution is generally achieved within 10 min to 30 min. With unknown samples, it may be necessary to swirl the mixture at least once to verify that dissolution is complete.

WARNING — Handle the hot crucible with tongs while wearing protective gloves and eye/face protection.

Before the melt solidifies, carefully pour it quantitatively into a beaker (5.7) containing 200 ml of dilute nitric acid (4.5).

Stir the solution by means of a magnetic stirrer (5.8) until the solid phase is dissolved; generally 15 min to 20 min is sufficient.

Transfer this solution to a volumetric flask of capacity 250 ml, 500 ml or 1 000 ml depending on the levels of concentration needed by the method of measurement for the elements of interest.

Rinse the beaker with dilute acid (4.5), add these washings to the flask and adjust the volume to the mark still using acid (4.5). If this solution is turbid, discard it and perform a new fusion with a smaller test portion.

Using the same procedure without the sample, perform at least one blank test within each batch of fusions.

Determinations should be undertaken within three days following the fusion. After a longer period of time, some compounds may hydrolyse or polymerize and precipitate, rendering the solution unusable.

If Pt-alloy or vitreous carbon crucibles (5.6) are not available, melt adhesion is likely to occur. In this case, the crucible plus adhering melt should be placed in the beaker with 200 ml nitric acid and the solid melt allowed to dissolve. This can take several hours. Care should be taken to ensure that the outside of the crucible is free of material containing elements of interest before it is immersed in the nitric acid, to avoid contamination of the sample solution. When the quantity of adhering melt is small enough, its dissolution can also be achieved by adding a few millilitres of nitric acid (4.5) to the crucible. In that case, slightly heating the solution on a hot plate may be necessary to assist dissolution.

NOTE 1 Experience has shown that dissolution of sample can be considerably prolonged if the material is not ground below 250 μm.

NOTE 2 Very hot platinum alloy crucibles are best handled with platinum-tipped tongs to avoid problems of contamination from other metals.

7 Test report

Submit the test report either separately or in conjunction with the test report of the subsequent analytical measurement.

The test report shall contain the following information:

a) a reference to this part of ISO 14869;

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.

ICS 13.080.10

Price based on 3 pages