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

Part 3: Dissolution with hydrofluoric, hydrochloric and nitric acids using pressurised microwave technique

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

Partie 3: Mise en solution par l'acide fluorhydrique, l'acide chlorhydrique et l'acide nitrique à l'aide de la technique de micro-ondes pressurisées

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

A list of all parts in the ISO 14869 series can be found on the ISO website.

Introduction

This document is a module for analysis of inorganic parameters in soil and soil material. This document concerns the complete dissolution for the subsequent analysis of elements.

An acid mixture based on nitric acid, hydrofluoric acid and hydrochloric acid will be used to totally dissolve most soils and similar materials. The resulting solutions can be determined separately or after combination with atomic absorption spectrometry (AAS) techniques, inductively coupled plasma optical emission spectrometry (ICP-OES) or inductively coupled plasma mass spectrometry (ICP-MS) determination methods.

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

Part 3:
Dissolution with hydrofluoric, hydrochloric and nitric acids using pressurised microwave technique

1 Scope

This document specifies a method for microwave-assisted dissolution of soil samples for determination of total element contents of

Al, As, Ba, Ca, Cd, Co, Cr, Cs, Cu, Fe, Hg, K, Li, Mg, Mn, Na, Ni, P, Pb, S, Se, Sb, Sr, Tl, V, Zn

using an acid mixture of nitric acid (HNO_3), hydrofluoric acid (HF) and hydrochloric acid (HCl). This method is applicable to all types of soil and soil material.

The main field of application is geological and pedological survey.

The acid mixture is suitable for total dissolution of element contents in soil (major, minor and trace), but some refractory compounds such as SiO_2, TiO_2, spinel, Al_2O_3 or other compounds may remain as a residue. In such a case, the use of alkaline fusion, following ISO 14869-2, is recommended to determine the true total element content.

NOTE 1 In environmental studies, usually, aqua regia extraction is applied using ISO 12914 or ISO 11466.

Solutions produced by the microwave method are suitable for analysis, for example, by using atomic absorption spectrometry (FAAS, HGAAS, CVAAS, GFAAS), inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS).

NOTE 2 Due to the presence of chloride in the digestion solution, limitations for the application of some analytical techniques can occur.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 11074, *Soil quality — Vocabulary*

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

ISO 11465, *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 given in ISO 11074, ISO 11464 and ISO 11465 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Safety remarks

All this work has to be performed by trained persons.

The reagents used within this document are strongly corrosive and potentially very harmful. Safety precautions are absolutely necessary due to strong corrosive reagents, high temperature and high pressure.

All procedures have to be performed in a hood or in closed force-ventilated equipment. By the use of strong oxidising reagents, the formation of explosive organic intermediates is possible, especially when dealing with samples with a high-organic content. Do not open pressurised vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

DANGER — Hydrofluoric acid is dangerous if inhaled as vapour or by direct contact with skin and mucosa. It should be noted that the effects of exposure to HF may not be apparent for several hours, particularly for skin, after which effective treatment can be difficult. Users of this document 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. Observe safety precaution for handling hydrofluoric acid.

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

5 Principle

The laboratory sample shall be treated in accordance with the principles of ISO 11464 in order to produce a homogeneous test sample from which a representative test portion can be sub-sampled and completely dissolved in an acid mixture by microwave-assisted heating.

6 Interferences and sources of errors

During trace element determinations, contaminations shall be avoided. The container in which the sample is delivered and stored can be a source of errors. The container's material shall be chosen such that it does not absorb the elements to be determined (e.g. elemental Hg can penetrate polyethylene walls very fast in both directions).

Grinding or milling samples includes a risk of contamination of the sample by the environment (e.g. air, dust, wear of milling equipment). Due to the volatility of some compounds, it is important to take care that the sample is not heated before the dissolution and that the volatile reaction products which might be formed during the dissolution are not allowed to escape.

For the determination of elements forming volatile compounds (e.g. Hg, As, Cr, Se), special care shall be taken during sample pre-treatment and pressurized digestions.

High acid and high dissolved matrix element concentrations in the dissolution solution may cause interferences in the analytical method.

Depending on the concentration of the elements of interest, particular attention needs to be paid to the cleaning of the laboratory equipment. It is recommended to thoroughly clean all laboratory equipment and as a minimum, leave the equipment standing over night in 5 % nitric acid.

Some elements of interest can be lost due to precipitation with ions present in the digestion solution, e.g. low-solubility chloride, fluoride or sulfate compounds. Before starting filtration, boric acid should be added to bind excess hydrofluoric acid and re-dissolve insoluble fluorides. During filtration, avoid introduction of contaminants.

7 Reagents

Use only reagents of recognized analytical grade.

7.1 Water.

Deionised or distilled water used shall conform at least to grade 2 of ISO 3696.

It is recommended that the same batch of water be used throughout a given batch of determinations. Blank tests are carried out in each series of sample determination.

7.2 Hydrochloric acid, $c(HCl)$ = 12 mol/l; ρ = 1,18 g/ml; $w(HCl)$ = 36 %.

7.3 Nitric acid, $c(HNO_3)$ = 14,3 mol/l; ρ = 1,4 g/ml; not less than $w(HNO_3)$ = 65 %.

7.4 Nitric acid, diluted, $c(HNO_3)$ = 0,5 mol/l, dilute 35 ml nitric acid (7.3) to 1 l with water (7.1).

7.5 Hydrofluoric acid, $c(HF)$ = 22,6 mol/l, ρ = 1,13 g/ml, $w(HF)$ = 40 %.

7.6 Antifoaming agent.

For example, *n*-dodecane ($C_{12}H_{26}$) or polyethyleneglycol *p*-(1,1,3,3-tetramethylbutyl)-phenyl ether ($C_{14}H_{22}O(C_2H_4O)_n$) is suitable.

7.7 Boric acid solution, $w(H_3BO_3)$ approximately 4 %.

Dissolve 20 g boric acid (H_3BO_3) in 450 ml of water and dilute to 500 ml with water. Store in a polyethylene bottle. Solubility limit of H_3BO_3 is 49 g in 1 000 ml of water.

8 Apparatus

All glassware and plasticware shall be adequately cleaned and stored in order to avoid any contamination.

8.1 Microwave-assisted dissolution system.

8.1.1 Microwave apparatus requirements, microwave apparatus for pressurised digestions — preferably a temperature-regulated device, in combination with pressure measurement for laboratory use only.

A laboratory-grade microwave unit cavity shall be well ventilated and corrosion resistant. All electronics shall be protected against corrosion for safe operation. The temperature performance requirements necessitate that the microwave decomposition system is able to sense the temperature with an accuracy of ±2,5 °C and automatically adjust the microwave field output power within 2 s of sensing. Temperature sensors should be accurate to ±2 °C [including at the final reaction temperature of (175 ± 5) °C]. Temperature feedback control provides the primary performance mechanism for the method. Due to the variability in sample matrices and microwave equipment (i.e. different vessel types and microwave designs), control of the temperature during the dissolution process is important for ensuring reproducible microwave heating for different units.