SLOVENSKI STANDARD
SIST ISO 12914:2019

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Kakovost tal - Določevanje elementov v frakciji, topni v zlatotopki, po ekstrakciji z mikrovalovi

Soil quality - Microwave-assisted extraction of the aqua regia soluble fraction for the determination of elements

Qualité du sol - Extraction assistée par micro-ondes de la fraction soluble dans l'eau régale pour la détermination des éléments

Ta slovenski standard je istoveten z: **ISO 12914:2012**

ICS:

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Soil quality — Microwave-assisted extraction of the aqua regia soluble fraction for the determination of elements

Qualité du sol — Extraction assistée par micro-ondes de la fraction soluble dans l'eau régale pour la détermination des éléments

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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 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 12914 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

This International Standard is a module for analysis of inorganic parameters in soil and soil material. This International Standard concerns the extraction with *aqua regia* for the subsequent analysis of elements.

Aqua regia will not totally dissolve most soils and similar materials, and the efficiency of the extraction for particular elements differs from element to element. Such efficiency might also differ for the same element in different matrices. Users of this International Standard should carry out a program of analysis using reference materials to ensure that the method given here is appropriate for their needs. Elements extractable in *aqua regia* cannot, therefore, be regarded as 'totals'; conversely, they cannot be regarded as the 'bio available' fraction, as the extraction procedure is too vigorous to represent any biological process.

Soil quality — Microwave-assisted extraction of the aqua regia soluble fraction for the determination of elements

1 Scope

This International Standard specifies a method for microwave-assisted extraction of elements from samples using *aqua regia* as the extraction solution for the determination of elements. This method is applicable to all types of soil and soil material.

Aqua regia extraction is suitable for the release of trace and major element fractions in soil. *Aqua regia* is not suitable for the extraction of elements from refractory compounds, such as SiO_2, TiO_2 and Al_2O_3.

The extraction with *aqua regia* is operationally defined and will not necessarily release all elements completely.

The microwave method is generic and can be implemented using a wide variety of equipment, provided:

a) the extraction mixture ratio is unchanged;

b) the extraction temperature is known.

Solutions produced by the microwave method are suitable for analysis, for example, by using atomic absorption spectrometry (flame: FAAS, hydride generation: HGAAS, cold vapour: CVAAS, graphite furnace; GFAAS), inductively coupled plasma emission spectrometry (ICP/OES) and inductively coupled plasma mass spectrometry (ICP/MS).

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

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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

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

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

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
aqua regia
extraction solution obtained by mixing 1 volume of nitric acid, $w(HNO_3)$ = 65 % to 70 %, and 3 volumes of hydrochloric acid, $w(HCl)$ = 35 % to 37 %

3.2

extraction

mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacting with the reagent mixture

3.3

sample

portion of soil material selected from a larger quantity of material

[ISO 11074:2005]

3.4

laboratory sample

sample intended for laboratory inspection of testing

[ISO 11074:2005]

3.5

test sample

portion of material resulting from the laboratory sample by means of an appropriate method of sample pretreatment and having the size (volume/mass) necessary for the desired testing or analysis

[ISO 11074:2005]

3.6

test portion;

analytical portion

quantity of material, of proper size, for measurement of the concentration or other property of interest removed from the test sample

NOTE 1 The test portion may be taken from the primary sample or from the laboratory sample directly if no preparation of sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

NOTE 2 A unit or increment of proper homogeneity, size and fineness, needing no further preparation, may be a test portion.

[ISO 11074:2005]

3.7

dry matter

mass fraction of a sample weight after the specified drying process and the weight of the moist sample

NOTE It is expressed in percent.

[ISO 11465:1993]

4 Safety remarks

All of the work has to be performed by trained persons.

The reagents used within this International Standard are strongly corrosive and potentially very harmful. Safety precautions are absolutely necessary due to toxic fumes and 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 oxidizing reagents, the formation of explosive organic intermediates is possible, especially when dealing with samples with a high organic content. Do not open pressurized vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

5 Principle

The laboratory sample should be treated in accordance with the principles of ISO 11464, in order to produce a homogeneous test sample from which a test portion can be subsampled and digested by *aqua regia* to the following heating procedure.

Microwave extraction at 175 °C ± 5 °C for 10 min ± 1 min in a closed vessel, followed by filtration and adjustment of the volume in a volumetric flask.

6 Interferences and sources of errors

Due to the volatility of some compounds, it is important to take care that the sample is not heated before the extraction, and that the volatile reaction products which might be formed during the extraction are not allowed to escape. Grinding or milling samples includes a risk of contamination of the sample by the environment (e. g. air, dust, wear of milling equipment). Losses of volatile compounds are possible due to elevated temperatures (e.g. a drying temperature over 40 °C may result in losses of mercury).

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 and glass can contaminate samples with its constituent elements such as B, Na, K, Al).

For the determination of elements forming volatile compounds (e.g. Hg, As), special care has to be taken during sample pretreatment.

The use of the described extraction procedures may leave large parts of the sample undissolved.

High-acid and high-dissolved-matrix concentrations in the extract may cause interferences in measurement steps.

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 overnight in 1 % to 5 % nitric acid.

Care shall be taken to ensure that the test portion is in contact with the acid mixture in the reaction vessel. Boiling aids like glass beads should be used, provided they do not contain constituents contaminating the sample.

Some elements of interest can be lost due to precipitation with ions present in the extract solution, e. g. low soluble compounds of chlorides. During filtration of the extracted solution, it is necessary that the filtration procedure does not introduce contaminants.

7 Reagents

Use only reagents of recognized analytical grade.

7.1 **Water**: deionized or distilled water used shall conform at least to grade 2 of ISO 3696.

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

7.2 **Hydrochloric acid**, $c(\mathrm{HCl}) = 12$ mol/l; $\rho = 1{,}18$ g/ml; $w(\mathrm{HCl}) = 36$ %.

7.3 **Nitric acid**, $c(\mathrm{HNO_3}) = 14{,}3$ mol/l; $\rho = 1{,}4$ g/ml; not less than $w(\mathrm{HNO_3}) = 65$ %.

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

7.5 **Anti-foaming agent**, for example, *n*-dodecane ($C_{12}H_{26}$) or polyethyleneglycol *p*-(1,1,2,3-tetramethylbuthyl)-phenyl ether [$C_{14}H_{22}O(C_2H_4O)_n$] is suitable.