SLOVENSKI STANDARD
oSIST prEN ISO 54321:2019

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Tla, obdelani biološki odpadki, blato in odpadki - Razklop frakcij elementov, topnih v zlatotopki (ISO/DIS 54321:2019)

Soil, treated biowaste, sludge and waste - Digestion of aqua regia soluble fractions of elements (ISO/DIS 54321:2019)

Boden, behandelter Bioabfall, Schlamm und Abfall - Aufschluss von mit Königswasser löslichen Anteilen von Elementen (ISO/DIS 54321:2019)

Sols, biodéchets traités, boues et déchets - Digestion des éléments solubles dans l'eau régale (ISO/DIS 54321:2019)

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Soil, treated biowaste, sludge and waste — Digestion of aqua regia soluble fractions of elements

ICS: ISO ics

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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 of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

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

This document combines text elements from EN 13657:2002, EN 16174:2012, ISO 11466:1995 and ISO 12914:2012 and will replace these standards.

Regarding the comparability of the procedure described in this document with those of the other standards mentioned above the next remarks can be made:

— This document describes, like the four other standards, the digestion of solid samples with aqua regia.

— Differences in the procedures of the different standards are small. An important difference between the reflux procedures as described in ISO 11466 and EN 13657 and EN 16174 concerns the waiting time after addition of the acid to the sample, before the digestion starts. ISO 11466 specifies a waiting time of 16 h, both European standards state that the digestion can start after the first strong reactions have ceased. In validation work it was proven that the difference between 2 h and 16 h of waiting was negligible, therefore this document follows the approach of EN 13657 and EN 16174.

— The heating block procedure was added to the reflux and microwave digestion procedures. The procedure was adopted from the Dutch standard NEN 6961, which specifies a boiling time of 2 h to 4 h. This document specifies a boiling time of 2 h.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The methods specified in this document are providing multi-element aqua regia digestion techniques for soil, treated biowaste, sludge and waste prior to analysis. It is known that the digestion of environmental samples with aqua regia will not necessarily lead to complete element recoveries, and that the extract from a test sample may not reflect the total concentrations of the target analytes. However, for most environmental applications the result obtained based upon digestion methods specified in this document are considered to be fit for the intended purpose.

This document is validated for several types of matrices as indicated in Table 1.

Table 1 — Matrices for which EN 54321 is validated

Matrix	Materials used in the validation test
Sludge	Municipal sludge
	Industrial sludge
	Sludge from electronic industry
	Ink waste sludge
	Sewage sludge
Biowaste (Method A)	Compost
	Composted sludge
Soil	Agricultural soil
	Sludge amended soils
Waste	City waste incineration fly ash ("oxidised" matrix)
	City waste incineration bottom ash ("silicate" matrix)
	Ink waste sludge (organic matrix)
	Electronic industry sludge ("metallic" matrix)
	BCR 146R (sewage sludge)
	BCR 176 (city waste incineration ash)

WARNING — Persons using this document should be familiar with usual laboratory practice. Some of the reagents used in this document are highly corrosive and very toxic. Safety precautions are absolutely necessary, not only due to the strong corrosive reagents, but also to the high temperature and high pressure.

The use of laboratory-grade microwave equipment with isolated and corrosion resistant safety devices is required. Domestic (kitchen) type microwave ovens shall not be used, as corrosion by acid vapours may compromise the function of the safety devices and prevent the microwave magnetron from shutting off when the door is open, which could result in operator exposure to hazardous levels of microwave energy.

All procedures shall be performed in a fume 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 pressurized vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

Soil, treated biowaste, sludge and waste — Digestion of aqua regia soluble fractions of elements

1 Scope

This document specifies two methods for digestion of soil, treated biowaste, sludge and waste by the use of aqua regia as digestion solution.

Digestion with aqua regia will not necessarily accomplish total decomposition of the sample. The extracted analyte concentrations may not necessarily reflect the total content in the sample but represent the aqua regia soluble metals under the condition of this test procedure. It is generally agreed that for environmental analysis purposes, the results are fit for the intended purpose to protect the environment.

This document is applicable for the following elements:

Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), boron (B), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), nickel (Ni), phosphorus (P), potassium (K), selenium (Se), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), thallium (Tl), tin (Sn), titanium (Ti), , vanadium (V), and zinc (Zn),.

This document can also be applied for the digestion of other elements, provided the user has verified the applicability.

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.

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

3 Terms and definitions

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

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

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

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

3.1
aqua regia
digestion solution obtained by mixing 1 volume of nitric acid (65 % *m*/*m* to 70 % *m*/*m*) and 3 volumes of hydrochloric acid (35 % *m*/*m* to 37 % *m*/*m*)

Note 1 to entry: These mass percentages agree with the concentrations of 6.2 and 6.3.

3.2
digestion
mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacting with a reagent mixture

3.3
dry residue
dry matter expressed as a percentage by mass after drying at 105 °C ± 5 °C to the constancy of weight

3.4
laboratory sample
sample intended for laboratory inspection of testing

[SOURCE: ISO 11074:2015]

3.5
sample
portion of material selected from a larger quantity of material

[SOURCE: ISO 11074:2015]

3.6
test portion
analytical portion
quantity of material of proper size for measurement of the concentration or other properties of interest, removed from the test sample

Note 1 to entry: The test portion may be taken 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 to entry: A unit or increment of proper homogeneity, size and fineness, needing no further preparation, may be a test portion.

[SOURCE: ISO 11074:2015]

3.7
test sample
analytical 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

[SOURCE: ISO 11074:2015]

4 Principle

A test portion is digested with aqua regia according to one of the following heating procedures:

— Method A: procedure under atmospheric conditions

  — A1: reflux for 2 h, followed by filtration/centrifugation;

  — A2: heating block at (105 ± 5) °C for 2 h, followed by filtration/centrifugation.

— Method B: microwave digestion

  — B1: Temperature controlled procedure: at (175 ± 5) °C for (10 ± 1) min in a closed vessel followed by filtration/centrifugation.

5 Interferences and sources of errors

The container in which the sample is delivered and stored can be a source of errors. Its material shall be chosen according to the elements to be determined (e.g. elemental Hg can penetrate polyethylene walls very fast in both directions. Glass can contaminate samples with its major elements: e.g. B, Na, K, Si and Al).

Grinding or milling samples includes a risk of contamination of the sample by the environment (air, dust, wear of milling equipment). Due to elevated temperature losses of volatile compounds are possible.

For the determination of elements forming volatile compounds (e.g. Hg, As) special care has to be taken at sample pre-treatment.

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

In the case of filtration of the digested solution it is necessary to take care that the filtration procedure does not introduce contaminants.

Ensure that all of the test portion is brought into contact with the acid mixture in the digestion vessel.

Some elements of interest can be lost due to precipitation with ions present in the final digest solution, e.g. low soluble chlorides, fluorides and sulfates.

6 Reagents

Use only acids and reagents of recognized analytical grade to avoid high blank values for subsequent analytical measurements. Use a test blank solution throughout the procedure applying all steps with the same amount of acids, but without a sample.

6.1 Water, e.g. deionized.

6.2 Hydrochloric acid, $c(HCl) \approx 12$ mol/l.

6.3 Nitric acid, $c(HNO_3) \approx 15$ mol/l.

6.4 Nitric acid, $c(HNO_3) \approx 0{,}5$ mol/l.

Dilute 35 ml nitric acid (6.3) to 1 l with water (6.1).

6.5 Antifoaming agent, e.g. *n*-dodecane ($C_{12}H_{26}$) or *n*-octanol ($C_8H_{18}O$) are suitable.

7 Apparatus

7.1 General

Usual laboratory apparatus. All glassware and plastics ware shall be adequately cleaned and stored in order to avoid any contamination.

Depending on the concentration of the element of interest, a particular caution to the cleaning of the vessels shall be taken.

7.2 Method A, Apparatus for boiling under atmospheric conditions

7.2.1 Method A1, reflux

7.2.1.1 Digestion vessel, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution, for example a quartz vessel. The digestion vessel shall have a volume of at least 5 times of the volume of the aqua regia used. The inner wall of the vessel shall be inert and shall not release substances to the digest in excess of the purity requirements of the subsequent analysis.

NOTE 1 Silica or borosilicate glass vessels can be used instead of quartz vessels.

NOTE 2 It may be necessary to periodically clean the digestion vessels with a suitable surfactant to remove persistent deposits.

7.2.1.2 Reflux condenser adaptable to the digestion vessel (7.2.1.1).

7.2.1.3 Absorption vessel, volatile species trap, in an open digestion system capable of trapping one or more volatile measurement species, adaptable to the reflux condenser (7.2.1.2).

7.2.1.4 Heating device, for example a heating mantle, thermostatic controlled, or an aluminium block thermostat.

7.2.2 Method A2, heating block

7.2.2.1 Digestion tube, 50 ml propylene tube with a screw cap from polypropylene.

NOTE The part of the tube not being heated and the screw cap function as a condenser, but are not really a reflux system. The material of the tube and screw cap have to be tested in order to be sure that release of elements of interest does not take place. Other materials and vessels with other volumes than mentioned above are allowed to be used if suitability has been proven.

7.2.2.2 Temperature controlled heating block, heating block able to heat the tube(s) to a temperature of (105 ± 5) °C.

7.3 Method B, Microwave digestion, temperature controlled, closed vessels

7.3.1 Digestion vessel, for pressurized microwave digestion, typically 100 ml volume, reagent-, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution. The vessel shall be suitable for the safe application in the temperature and pressure range applied, capable of withstanding pressures of at least 3 000 kPa.

Digestion vessels made of perfluoro alkoxyl alkane (PFA), modified polytetrafluoroethene (PTFE) or quartz, and equipped with a safety pressure releasing system to avoid explosion of the vessel, shall be used. The inner wall of the vessel shall be inert and shall not release contaminations to the digest solution.

NOTE It may be necessary to periodically clean the digestion vessels with a suitable surfactant to remove persistent deposits.

7.3.2 Microwave digestion system, corrosion resistant and well ventilated. All electronics shall be protected against corrosion for safe operation.

Use a laboratory-grade microwave oven with temperature feedback control mechanisms.

The microwave digestion system should be able to control the temperature with an accuracy of ±5 °C and automatically adjust the microwave field output power within 2 s of sensing. Temperature sensors shall be accurate to ±2 °C, including 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 matrix types and microwave digestion equipment (i.e. different vessel types and microwave designs), control of the temperature during digestion is important for reproducible microwave heating and comparable data.

The accuracy of the temperature measurement system should be periodically tested at an elevated temperature according to the manufactures instructions. If the temperature deviates by more than 2 °C from the temperature measured by an external, calibrated temperature measurement system, the microwave temperature measurement system should be re-calibrated.

7.4 Sample containers, plastics and glass containers are both suitable.

7.5 Filter paper, usually with a pore size of 0,45 µm and resistant to the diluted aqua regia final digestion solution.

7.6 Volumetric flasks, usually of nominal capacity of 50 ml or 100 ml.

7.7 Analytical balance, with an accuracy of 1 mg or better.

7.8 Boiling aids, anti-bumping granules or glass beads, diameter 2 mm to 3 mm, acid washed.

8 Procedure

8.1 General

Pretreat, if not otherwise specified, soil, sludge and biowaste samples according to EN 16179 or ISO 11464 and waste samples according to EN 15002.

Determine the dry matter content, depending on the matrix of the sample, e.g. according to EN 15934.

For waste samples the next remarks apply:

— Pretreatment should include drying or grain size reduction below a particle size of 250 µm for solid waste or homogenizing by use of a high speed mixer or sonification for liquid waste samples.

— The mass of test portion for a single digestion has to be selected in a way, that:

  — it is representative for the laboratory sample;

  — it complies with the specifications of manufacturer of the digestion unit.

NOTE If the representative test portion exceeds the manufacturers' specifications the test portion should be divided into smaller quantities and digested separately. The individual digests should be combined prior to analysis.

— For representability reasons a mass above 200 mg is to be preferred for the test portion. Follow, for safety reasons, the manufacturer's instructions regarding the maximum amount of organic carbon in the sample.

8.2 Blank test

Carry out a reagent blank test digestion in parallel with the determination, using the same procedure and the same quantities of all the reagents as in the determination, but omitting the test portion. The laboratory shall define acceptable limits.

NOTE The measurement of a blank is introduced to determine the contribution of the extracting solution, glassware, digestion tube and filter paper used to the measured value.

