



# SLOVENSKI STANDARD SIST EN ISO 54321:2021

01-april-2021

---

**Tla, obdelani biološki odpadki, blato in odpadki - Razklop frakcij elementov, topnih v zlatotopki (ISO 54321:2020)**

Soil, treated biowaste, sludge and waste - Digestion of aqua regia soluble fractions of elements (ISO 54321:2020)

Boden, behandelter Bioabfall, Schlamm und Abfall - Aufschluss von mit Königswasser löslichen Anteilen von Elementen (ISO 54321:2020)

Sols, biodéchets traités, boues et déchets - Digestion des éléments solubles dans l'eau régale (ISO 54321:2020)

[SIST EN ISO 54321:2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-e15a4e9b2178/sist-en-iso-54321-2021)

[https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-e15a4e9b2178/sist-en-iso-54321-2021)

**Ta slovenski standard je istoveten z: EN ISO 54321:2021**

---

**ICS:**

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

**SIST EN ISO 54321:2021**

**en,fr,de**

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST EN ISO 54321:2021](#)

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

EUROPEAN STANDARD

EN ISO 54321

NORME EUROPÉENNE

EUROPÄISCHE NORM

January 2021

ICS 13.030.20; 13.080.10

English Version

## Soil, treated biowaste, sludge and waste - Digestion of aqua regia soluble fractions of elements (ISO 54321:2020)

Sols, biodéchets traités, boues et déchets - Digestion des éléments solubles dans l'eau régale (ISO 54321:2020)

Boden, behandelter Bioabfall, Schlamm und Abfall - Aufschluss von mit Königswasser löslichen Anteilen von Elementen (ISO 54321:2020)

This European Standard was approved by CEN on 18 July 2020.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents	Page
European foreword.....	3

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST EN ISO 54321:2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021)  
<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

## European foreword

This document (EN ISO 54321:2021) has been prepared by Technical Committee ISO/TC 190 "Soil quality" in collaboration with Technical Committee CEN/TC 444 "Environmental characterization of solid matrices" the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2021, and conflicting national standards shall be withdrawn at the latest by July 2021.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

### Endorsement notice

**iTeh STANDARD PREVIEW**  
(standards.iteh.ai)

The text of ISO 54321:2020 has been approved by CEN as EN ISO 54321:2021 without any modification.

[SIST EN ISO 54321:2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021)

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST EN ISO 54321:2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021)

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

INTERNATIONAL  
STANDARD

ISO  
54321

First edition  
2020-08

---

---

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

*Sols, biodéchets traités, boues et déchets — Digestion des éléments  
solubles dans l'eau régale*

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST EN ISO 54321:2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021)

[https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-  
f45a4e9b2178/sist-en-iso-54321-2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021)



Reference number  
ISO 54321:2020(E)

© ISO 2020

## iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 54321:2021

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>



### **COPYRIGHT PROTECTED DOCUMENT**

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

Published in Switzerland



<b>Contents</b>	<b>Page</b>
<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Interferences and sources of errors</b> .....	<b>2</b>
<b>6 Reagents</b> .....	<b>3</b>
<b>7 Apparatus</b> .....	<b>3</b>
7.1 General.....	3
7.2 Method A — Apparatus for thermal heating under atmospheric conditions.....	3
7.2.1 Method A1 — Thermal heating under reflux conditions.....	3
7.2.2 Method A2 — Thermal heating with a heating block with containers.....	4
7.3 Method B — Microwave digestion with temperature control, closed vessels.....	4
<b>8 Procedure</b> .....	<b>5</b>
8.1 General.....	5
8.2 Blank test.....	5
8.3 Method A — Thermal heating under atmospheric conditions.....	5
8.3.1 Method A1 — Thermal heating under reflux conditions.....	5
8.3.2 Method A2 — Thermal heating with a heating block with containers.....	6
8.4 Method B — Microwave digestion with temperature control, closed vessels.....	7
<b>9 Test report</b> .....	<b>7</b>
<b>Annex A (informative) Repeatability and reproducibility data for soil, biowaste and sludge samples</b> .....	<b>9</b>
<b>Annex B (informative) Repeatability and reproducibility data for waste samples</b> .....	<b>10</b>
<b>Bibliography</b> .....	<b>37</b>

**ISO 54321:2020(E)****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](http://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](http://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](http://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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 444, *Environmental characterization of solid matrices*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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](http://www.iso.org/members.html).

## Introduction

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 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.

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 this document 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**

**ISO 54321:2020(E)**

magnetron from shutting off when the door is open, which could result in operator exposure to hazardous levels of microwave energy.

All procedures should 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.

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST EN ISO 54321:2021](https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021)

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

# 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 an aqua regia digestion.

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

There are no normative references in this document.

## 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 <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 3.1

#### aqua regia

*digestion* (3.2) solution obtained by mixing 1 volume of nitric acid (mass fraction of 65 % to 70 %) and 3 volumes of hydrochloric acid (mass fraction of 35 % to 37 %)

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  $105\text{ °C} \pm 5\text{ °C}$  to the constancy of weight

**ISO 54321:2020(E)****3.4****laboratory sample**

sample (3.5) intended for laboratory inspection of testing

[SOURCE: ISO 11074:2015, 4.3.7]

**3.5****sample**

portion of material selected from a larger quantity of material

[SOURCE: ISO 11074:2015, 4.1.17]

**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* (3.7)

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, 4.3.15]

**3.7****test sample****analytical sample**

portion of material resulting from the *laboratory sample* (3.4) by means of an appropriate method of sample pre-treatment and having the size (volume/mass) necessary for the desired testing or analysis

[SOURCE: ISO 11074:2015, 4.1.3]

**iTeh STANDARD PREVIEW**  
(standards.iteh.ai)

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

**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 (120 ± 10) min, followed by filtration/centrifugation;
  - A2: heating block at (105 ± 5) °C for (120 ± 10) min, 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 during 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**,  $c(\text{HCl}) \approx 12 \text{ mol/l}$ .

**6.3 Nitric acid**,  $c(\text{HNO}_3) \approx 15 \text{ mol/l}$ .

**6.4 Nitric acid**,  $c(\text{HNO}_3) \approx 0,5 \text{ mol/l}$ .

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

**6.5 Antifoaming agent**, e.g. *n*-dodecane ( $\text{C}_{12}\text{H}_{26}$ ) or *n*-octanol ( $\text{C}_8\text{H}_{18}\text{O}$ ) are suitable.

<https://standards.iteh.ai/catalog/standards/sist/5dcbf586-28c2-4dc5-9e6c-f45a4e9b2178/sist-en-iso-54321-2021>

## 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 upon the concentration of the element of interest, particular care should be exercised with respect to the effective cleaning of the vessels.

### 7.2 Method A — Apparatus for thermal heating under atmospheric conditions

#### 7.2.1 Method A1 — Thermal heating under reflux conditions

**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 can 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).