

SLOVENSKI STANDARD
oSIST prEN ISO 21268-4:2018
01-april-2018

Kakovost tal - Postopki izluževanja za nadaljnje kemijsko in ekotoksikološko preskušanje tal in talnih (zemeljskih) materialov - 4. del: Vpliv pH na izluževanje z začetnim dodatkom kisline ali baze (ISO/DIS 21268-4:2018)

Soil quality - Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials - Part 4: Influence of pH on leaching with initial acid/base addition (ISO/DIS 21268-4:2018)

Bodenbeschaffenheit - Elutionsverfahren für die anschließende chemische und ökotoxikologische Untersuchung von Boden und von Bodenmaterialien - Teil 4: Einfluss des pH-Wertes unter vorheriger Säure/Base-Zugabe (ISO/DIS 21268-4:2018)

Qualité du sol - Modes opératoires de lixiviation en vue d'essais chimiques et écotoxicologiques ultérieurs des sols et matériaux du sol - Partie 4: Essai de dépendance au pH avec ajout initial d'acide/base (ISO/DIS 21268-4:2018)

Ta slovenski standard je istoveten z: prEN ISO 21268-4

ICS:

13.080.05 Preiskava tal na splošno Examination of soils in general

oSIST prEN ISO 21268-4:2018

en,fr,de

DRAFT INTERNATIONAL STANDARD

ISO/DIS 21268-4

ISO/TC 190/SC 7

Secretariat: DIN

Voting begins on:
2018-02-08Voting terminates on:
2018-05-03

Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials —

Part 4: Influence of pH on leaching with initial acid/base addition

Qualité du sol — Modes opératoires de lixiviation en vue d'essais chimiques et écotoxicologiques ultérieurs des sols et matériaux du sol —

Partie 4: Essai de dépendance au pH avec ajout initial d'acide/base

ICS: 13.080.05

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN ISO 21268-4:2020](https://standards.iteh.ai/catalog/standards/sist/8e3b400f-1f93-4142-856c-25037141e7f1/sist-en-iso-21268-4-2020)

<https://standards.iteh.ai/catalog/standards/sist/8e3b400f-1f93-4142-856c-25037141e7f1/sist-en-iso-21268-4-2020>

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING



Reference number
ISO/DIS 21268-4:2018(E)

© ISO 2018

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 21268-4:2020

<https://standards.iteh.ai/catalog/standards/sist/8e3b400f-1f93-4142-856c-25037141e7f1/sist-en-iso-21268-4-2020>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

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, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Published in Switzerland

Contents		Page
Foreword		v
Introduction		vi
1 Scope		1
2 Normative references		1
3 Terms and definitions		2
4 Symbols and abbreviations		3
5 Principle		4
6 Apparatus		4
7 Reagents		6
8 Sample pretreatment		6
8.1 Sample size		6
8.2 Particle size reduction		6
8.3 Determination of the dry matter content and of water content		7
8.4 Preparation of test portion		7
9 Procedure		8
9.1 Contact time		8
9.2 pH-range		8
9.3 Leaching test		8
9.3.1 General		8
9.3.2 Preparation of leachant		8
9.3.3 Leaching procedure		10
9.4 Natural pH		12
10 Eluate treatment, storage and analysis		12
11 Analytical determination		12
12 Blank test		12
13 Calculation		13
14 Test report		13
15 Performance characteristics		13
Annex A (informative) Example of a specific liquid-solid separation procedure for soil sample		15
A.1 Introduction		15
A.2 Apparatus		15
A.3 Procedure		15
Annex B (informative) Operation and uses of the test: influence of pH on the leaching behaviour		17
B.1 Comparison of the mode of operation of the test with the pH continuous control mode — Influence of pH on the leaching behaviour		17
B.2 Expression of results		17
B.3 Scope and limits of the application field of the test		19

ISO/DIS 21268-4:2018(E)

B.4	Example: Identification of the sensitivity of leaching to pH over the environmentally relevant pH range	19
Annex C (informative)	Preliminary determination of the acid/base consumption.....	21
C.1	General.....	21
C.2	Titration procedure to estimate the ANC and the BNC.....	21
C.2.1	Reagents	21
C.2.2	Apparatus.....	21
C.2.3	Test portion.....	21
C.2.4	Procedure.....	21
C.2.5	Expression of results.....	23
C.3	Arbitrary division of the maximum acid/base consumption for the extreme pH values.....	23
C.3.1	General.....	23
C.3.2	Reagents	23
C.3.3	Apparatus.....	23
C.3.4	Test portion.....	23
C.3.5	Procedure.....	23
C.3.6	Expression of results.....	24
Annex D (informative)	Repeatability and reproducibility data.....	26
D.1	Soil sample used in the interlaboratory comparison study	26
D.2	Interlaboratory comparison results	26
Bibliography.....	IT&H STANDARD PREVIEW	1

(standards.iteh.ai)

SIST EN ISO 21268-4:2020

<https://standards.iteh.ai/catalog/standards/sist/8e3b400f-1f93-4142-856c-25037141e7f1/sist-en-iso-21268-4-2020>

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 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Soil and site assessment*.

This first edition of ISO 21268-4 cancels and replaces ISO/TS 21268-4:2007 which has been technically revised.

The main changes compared to the previous document are as follows:

- Clause 15 Performance characteristics technically revised;
- Annex D “Repeatability and reproducibility data” has been added.

A list of all parts in the ISO 21268 series can be found on the ISO website.

ISO/DIS 21268-4:2018(E)

Introduction

In various countries, tests have been developed to characterize and assess the constituents that can be released from materials. The release of soluble constituents upon contact with water is regarded as a main mechanism of release, resulting in a potential risk to the environment during the use or disposal of materials. The intent of these tests is to identify the leaching properties of materials. The complexity of the leaching process makes simplifications necessary.

Not all of the relevant aspects of leaching behaviour can be addressed in one standard.

Tests to characterize the behaviour of materials can generally be divided into three categories (see References [1], [2] and [4]). The relationships between these tests are summarized below.

- a) “Basic characterization” tests are used to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials. Liquid/solid (L/S) ratios, leachant composition, factors controlling leachability, such as pH, redox potential, complexing capacity, role of dissolved organic carbon (DOC), ageing of material and physical parameters, are addressed in these defined tests.
- b) “Compliance” tests are used to determine whether the material complies with a specific behaviour or with specific reference values. These tests focus on key variables and leaching behaviour previously identified by basic characterization tests.
- c) “On-site verification” tests are used as a rapid check to confirm that the material is the same as that which has been subjected to the compliance test(s). On-site verification tests are not necessarily leaching tests.

The test procedure described in this method belongs to category a) “Basic characterization” tests.

NOTE Up to now, the test procedures described in this part of ISO/TS 21268 have not been validated.

Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials — Part 4: Influence of pH on leaching with initial acid/base addition

1 Scope

This part of ISO/TS 21268 specifies a test method to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials.

It applies to the determination of the influence of pH on the leachability of inorganic and organic constituents from soil and soil material, and the ecotoxicological effects of eluates with respect to microorganisms, fauna and flora. The test is not suitable for constituents that are volatile under ambient conditions. The equilibrium condition, as defined in this part of ISO/TS 21268, is established by the addition of predetermined amounts of acid or base to reach desired final pH values.

The test procedure specified in this part of ISO/TS 21268 produces eluates that are subsequently characterized by physical, chemical and ecotoxicological standard methods.

For the purposes of ecotoxicological tests, the relevant pH range (see 9.2) will usually be pH 5 to 9.

NOTE 1 Volatile organic constituents include the low molecular weight components in mixtures such as mineral oil.

NOTE 2 It is not always possible to optimize test conditions simultaneously for inorganic and organic constituents and optimum test conditions may also vary between different groups of organic constituents. Test requirements for organic constituents are generally more stringent than those for inorganic constituents. The test conditions suitable for measuring the release of organic constituents will generally also be applicable to inorganic constituents.

NOTE 3 For ecotoxicological testing, eluates representing the release of both inorganic and organic contaminants are needed. In this document, ecotoxicological testing is meant to include genotoxicological testing.

This test cannot be used alone to determine the total leaching behaviour of a soil. More leaching tests are needed for that extended goal. This part of ISO/TS 21268 does not address issues related to health and safety. It only determines the leaching properties outlined in Clause 5.

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 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

ISO 7027, *Water quality — Determination of turbidity*

ISO/DIS 21268-4:2018(E)

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

ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 10381-3, *Soil quality — Sampling — Part 3: Guidance on safety*

ISO 10381-4, *Soil quality — Sampling — Part 4: Guidance on the procedure for investigation of natural, near-natural and cultivated sites*

ISO 10381-5, *Soil quality — Sampling — Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*

ISO 10523, *Water quality — Determination of pH*

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 following terms and definitions apply.

3.1 leaching test
test during which a soil or soil material is put into contact with a leachant under strictly defined conditions and some constituents of the material are extracted

3.2 leachant
liquid used in a leaching test

3.3 eluate
solution obtained by a laboratory leaching test

3.4 single batch leaching test
leaching test in which a fixed amount of material is leached in one step with a fixed amount of leachant

3.5 liquid to solid ratio
L/S
ratio between the amount of liquid (L) and of solid (S) in the test

Note 1 to entry: L/S is expressed in litres per kilogram (l/kg) of dry matter.

3.6 dry matter content
 w_{dm}
ratio, expressed in percent, between the mass of the dry residue, determined in accordance with ISO 11465, and the corresponding raw mass

3.7 water content
 w_{H_2O}
ratio, expressed in percent, between the mass of water contained in the material as received and the corresponding dry residue of the material.

Note 1 to entry: The basis for the calculation of the moisture content is the mass of the dry residue in this part of ISO/TS 21268, as specified in ISO 11465 (for the determination of the water content of soil).

3.8

laboratory sample

sample or subsample(s) sent to or received by the laboratory

[SOURCE: IUPAC:1997]

3.9

test sample

sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

[SOURCE: IUPAC:1997]

3.10

test portion

amount or volume of the test sample taken for analysis, usually of known weight or volume

[SOURCE: IUPAC:1997]

3.11

soil material

soil-like material

excavated soil, dredged materials, manufactured soils, treated soil and fill materials

[SOURCE: ISO 15176:2002, 3.1.4]

4 Symbols and abbreviations

ANC acid neutralization capacity

BNC base neutralization capacity

C_B is the concentration of the base (mol/l) (see 7.4)

DOC dissolved organic carbon

L/S liquid to solid ratio

m_D is the mass of the dried sample (kg)

m_W is the mass of non-dried sample (kg)

n_B is the base consumption for the particular pH (mol /kg OH⁺ dry matter)

t_0 time at the start of the leaching test

V_A, V_B volume of acid /base used in leachant

V_d volume of demineralized water used in leachant

V_L volume of prepared leachant

w_{dm} dry matter content of the soil

ISO/DIS 21268-4:2018(E)

$w_{\text{H}_2\text{O}}$ water content

5 Principle

Several separate test portions (up to eight) are leached at a fixed L/S ratio ($L/S = 10 \text{ l/kg}$) with leachants containing different preselected amounts of acid or base containing a low concentration (0,001 mol/l) of calcium chloride in order to reach stationary pH values at the end of the extraction period (see 8.4). Each leachant is added in three steps in the beginning of the test. In the full test eight final pH-values are required, covering the range pH 4 to pH 12 (both included, i.e. the lowest value 4 and the highest value 12). The amounts of acid or base needed to cover the pH range can be derived from the results of a preliminary titration, from available experimental data on the material to be tested or from an arbitrary division of the predetermined maximum consumption of acid and base. The tests are carried out at a fixed contact time at the end of which an equilibrium condition can be assumed to be reached for most constituents in most soil materials to be characterized. The equilibrium condition, as defined in this part of ISO/TS 21268, is verified at the end of the extraction period.

The results are expressed in milligrams per litre (mg/l) of constituents for each final pH value. For each final pH value, the quantity of acid that is added is also expressed in mol/kg H^+ dry matter and the quantity of base that is added is expressed as negative mol/kg H^+ dry matter.

NOTE 1 This test can also be performed using continuous pH control. The results are generally consistent (see Annex B).

NOTE 2 Other expressions of results are possible (including mg/kg of dry matter).

From the amount of acid and base used to reach a given end pH, the acid or base neutralization capacity (ANC, BNC) of the soil or soil material can also be determined.

NOTE 3 The pH range covered by the test can be restricted to a pH range relevant for the specific material and the considered problem (see 9.2).

NOTE 4 The leachant is made with 0,001 mol/l CaCl_2 to minimize the mobilization of DOC caused by a too-low ionic strength of the leachant. At the level of 0,001 mol/l CaCl_2 the complexation of metals with chloride is considered to be negligible.

The constituents in the eluate(s) are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates. The eluate may also be applied for subsequent ecotoxicity or genotoxicity testing.

After the test, the leaching conditions (in terms of pH, electrical conductivity, DOC and, optionally, turbidity and redox potential dictated by the material) are recorded.

NOTE 5 These parameters often control the leaching behaviour of soil materials and are therefore important for checking the leaching test.

6 Apparatus

6.1 Borosilicate glass, of high purity in accordance with ISO 5667-3, with a nominal volume of 1 l, glass bottles having caps of inert material, for example, PTFE (polytetrafluoroethylene). Rinsing is compulsory.

NOTE 1 If only inorganic parameters are analysed, alternative materials such as HDPE/PP bottles can be used, except for unpreserved samples for mercury analysis.

NOTE 2 If Boron analyses are necessary, any plastics bottles may be used, e.g. PTFE (polytetrafluoroethylene).