SLOVENSKI STANDARD
SIST EN 14405:2017

01-december-2017

Nadomešča:
SIST-TS CEN/TS 14405:2004

Karakterizacija odpadkov - Izluževalni preskusi - Preskus v koloni s tokom navzgor (pri določenih pogojih)

Characterization of waste - Leaching behaviour test - Up-flow percolation test (under specified conditions)

Charakterisierung von Abfällen - Untersuchung des Elutionsverfahrens - Perkolationsprüfung im Aufwärtsstrom (unter festgelegten Bedingungen)

Caractérisation des déchets - Essais de comportement à la lixiviation - Essai de percolation à écoulement ascendant (dans des conditions spécifiées)

Ta slovenski standard je istoveten z: **EN 14405:2017**

ICS:

13.030.01 Odpadki na splošno Wastes in general

SIST EN 14405:2017 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14405

March 2017

ICS 13.030.10

Supersedes CEN/TS 14405:2004

English Version

Characterization of waste - Leaching behaviour test - Up-flow percolation test (under specified conditions)

Caractérisation des déchets - Essais de comportement à la lixiviation - Essai de percolation à écoulement ascendant (dans des conditions spécifiées)

Charakterisierung von Abfällen - Untersuchung des Elutionsverfahrens - Perkolationsprüfung im Aufwärtsstrom (unter festgelegten Bedingungen)

This European Standard was approved by CEN on 3 February 2017.

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, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, 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: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 14405:2017 E

Contents

Page

European foreword ... 4

Introduction ... 5

1 Scope ... 7

2 Normative references ... 7

3 Terms and definitions ... 8

4 Principle ... 9

5 Reagents ... 9

6 Equipment ... 10
6.1 General ... 10
6.2 Laboratory equipment ... 10

7 Sample pretreatment ... 11
7.1 General ... 11
7.2 Sample preparation ... 11
7.3 Test portion ... 12
7.4 Determination of dry residue ... 13

8 Procedure ... 13
8.1 Temperature ... 13
8.2 Preparation ... 13
8.3 Packing of the column ... 13
8.4 Start-up of the test ... 15
8.5 Collection of additional eluate fractions ... 16
8.6 Further preparation of the eluates for analysis ... 18
8.7 Blank test ... 18

9 Calculations ... 18

10 Performance characteristics ... 19

11 Test report ... 20
11.1 General ... 20
11.2 General data ... 20
11.3 Leaching test conditions ... 20
11.4 Analytical report ... 20
11.5 Results of the leaching test ... 21

Annex A (informative) **Illustration of the column and accompanying equipment** ... 22

Annex B (informative) **Suggestions for packing the column, water saturation and establishment and checking of equilibrium conditions** ... 23

B.1 General ... 23

B.2 Guidelines on column filling and packing ... 23

B.3 Water saturation ... 23

B.4 Equilibration and checking of equilibrium conditions ... 24

Annex C (informative) **Background for the choices made in developing this percolation test** ... 26

C.1 Introduction **26**
C.2 Particle size / particle size distribution **27**
C.3 Column dimensions **27**
C.4 Flow mode (up-flow/down-flow) **27**
C.5 Flow rate of the leachant **27**
C.6 *L/S* ratio and eluate fractions collected/duration of the test **28**
C.7 Nature of the leachant **28**
C.8 Temperature **29**
C.9 Beneficial use of the test results **29**
Annex D (informative) **Repeatability and reproducibility data** **30**
D.1 Materials used in the interlaboratory comparison study **30**
D.2 Interlaboratory comparison results **30**
Bibliography **40**

European foreword

This document (EN 14405:2017) has been prepared by Technical Committee CEN/TC 444 “Test methods for 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 September 2017, and conflicting national standards shall be withdrawn at the latest by September 2017.

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.

This document supersedes CEN/TS 14405:2004.

The following significant technical changes have been implemented in this new edition of the text:

— the status of the document has been changed from Technical Specification (CEN/TS) into a European Standard;

— based on CEN ISO/TS 21268-3 and CEN/TS 16637-3 the option for analysis of non-volatile organic substances has been added;

— performance data for inorganic substances has been added (see Annex D).

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association

This European Standard has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries.

This European Standard was elaborated on the basis of:

— NEN 7343:1995;

— NT ENVIR 002:1995.

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

Introduction

This European Standard specifies an up-flow percolation test to determine the leaching behaviour of inorganic and non-volatile organic substances from granular waste materials under standardized percolation conditions.

NOTE Validation data for non-volatile organic substances are not currently available, but will be added on revision when available.

For the complete characterization of the leaching behaviour of waste under specified conditions the application of other test methods is required (see EN 12920).

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples can contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction.

Consequently these samples should be handled with special care. Gases which can be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations will be followed with respect to all hazards associated with this method.

In the different European countries, tests have been developed to characterize and assess the substances which can be leached from waste materials. The release of soluble substances upon contact with water is regarded as one of the main mechanism of release which results in a potential risk to the environment during life-cycle of waste materials (disposal or re-use scenario). The intent of these tests is to identify the leaching properties of waste materials. The complexity of the leaching process makes simplifications necessary. Not all of the relevant aspects of leaching behaviour can be addressed in one single standard. Procedures to characterize the behaviour of waste materials can generally be divided into three steps, using different tests in relation to the objective. The following test hierarchy is taken from the Landfill Directive[1] and the Decision on Annex II of this Directive[2] for disposal of waste.

a) Basic characterization constitutes a full characterization of the waste by gathering all the necessary information for a safe management of the waste in the short and long term. Basic characterization may provide information on the waste (type and origin, composition, consistency, leachability, etc.), information for understanding the behaviour of waste in the considered management scenario, comparison of waste properties against limit values, and detection of key variables (critical parameters as liquid/solid (*L/S*) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters) for compliance testing and options for simplification of compliance testing. Characterization may deliver ratios between test results from basic characterization and results from simplified test procedures as well as information on a suitable frequency for compliance testing. In addition to the leaching behaviour, the composition of the waste should be known or determined by testing. The tests used for basic characterization should always include those to be used for compliance testing.

b) Compliance testing is used to demonstrate that the sample of today fits the population of samples tested before by basic characterization and through that, is used to carry out compliance with regulatory limit values. The compliance test should therefore always be part of the basic characterization program. The compliance test focuses on key variables and leaching behaviour identified by basic characterization tests. Parts of basic characterization tests can also be used for compliance purposes.

[1] Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste.

[2] Council Decision 2003/33/EC of 19 December 2002.

c) On-site verification tests are used as a rapid check to confirm that the waste is the same as that which has been subjected to characterization or compliance tests. On-site verification tests are not necessarily leaching tests.

The test procedure described in this document is a basic characterization test and falls in category a).

1 Scope

This European Standard is applicable for determining the leaching behaviour of inorganic and non-volatile organic substances from granular waste (without or with size reduction (see 7.2)). The waste body is subjected to percolation with water as a function of liquid to solid ratio under specified percolation conditions. The waste is leached under hydraulically dynamic conditions. The method is a once-through column leaching test and the test results establish the distinction between different release patterns, for instance wash-out and release under the influence of interaction with the matrix, when approaching local equilibrium between waste and leachant. This test method produces eluates, which can subsequently be characterized by physical, chemical and ecotoxicological methods according to existing standard methods.

NOTE 1 The mentioned specified percolation conditions are arbitrary and are not necessarily simulating a specific scenario.

NOTE 2 Waste materials that show a saturated hydraulic conductivity between 10^{-7} m/s and 10^{-8} m/s can be subjected to this test, but it can be difficult to maintain the imposed flow rate. If a waste shows a saturated hydraulic conductivity below 10^{-8} m/s, the test will preferably not be carried out (See C.5 for a definition of 'hydraulic conductivity').

NOTE 3 This procedure is generally not applicable to biologically degrading materials and materials reacting with the leachant, leading, for example, to excessive gas emission or excessive heat release.

NOTE 4 This procedure is applicable to materials showing solidification in the column, if the final hydraulic conductivity is within the specified range (see NOTE 2).

NOTE 5 It is not always possible to optimise test conditions simultaneously for inorganic and non-volatile organic substances and optimum test conditions may also vary between different groups of non-volatile organic substances. Test requirements for non-volatile organic substances are generally more stringent than those for inorganic constituents. The test conditions suitable for measuring the release of non-volatile organic substances will generally also be applicable to inorganic substances.

NOTE 6 For ecotoxicological testing, eluates representing the release of both inorganic and non-volatile organic substances are needed. In this standard ecotoxicological testing is meant to include also genotoxicological testing.

NOTE 7 Validation data for non-volatile organic substances are not currently available, but will be added on revision when available.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14346, *Characterization of waste - Calculation of dry matter by determination of dry residue or water content*

EN 14899, *Characterization of waste - Sampling of waste materials - Framework for the preparation and application of a Sampling Plan*

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

EN 16192, *Characterization of waste - Analysis of eluates*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 5667-3, *Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO 5667-3)*

3 Terms and definitions

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

3.1
dry residue
w_{dr}
remaining mass fraction of a sample after a drying process at 105 °C

[SOURCE: EN 14346:2006]

3.2
eluate
solution obtained by a leaching test

3.3
equilibrium condition
condition achieved when the pH deviation during a checking period is below a specified value

Note 1 to entry: See 8.4.

3.4
granular waste
solid waste that is neither monolithic, liquid, gas nor sludge

3.5
laboratory sample
sample or sub sample(s) sent to or received by the laboratory

[SOURCE: IUPAC, 1990]

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, crushing, sawing, coring, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

Note 2 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

3.6
leachant
liquid that is brought into contact with the test portion in the leaching procedure

3.7
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 l/kg dry matter.

3.8
sample
quantity that is representative of a certain larger quantity

3.9
test portion
amount or volume of the test sample taken for analysis, usually of known weight or volume

[SOURCE: IUPAC, 1990]

3.10
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: IUPAC, 1990]

4 Principle

This standard describes a method to determine the release of substances from waste, with or without size reduction, packed in a column with a leachant percolating through it. A continuous vertical up-flow is used, so that the column is water saturated. The test conditions, including the flow rate of the leachant, enable a conclusion to be drawn from the results as to which components are rapidly being washed out and which components are released under the influence of interaction with the matrix.

The test portion of the waste to be tested is packed in a column in a standardized manner. The leachant is percolated in up-flow through the column at a specified flow rate up to a fixed *L/S* ratio. The eluate is collected in several separate fractions that are characterized physically and chemically according to existing standard methods. In the test, equilibrium conditions at the outlet of the column are verified after an equilibration period by measuring a pH deviation.

The procedure described in this standard is based on the more stringent test requirements for determining the release of non-volatile organic substances and/or for subsequent ecotoxicological testing. If only the release of inorganic substances is to be measured, less stringent requirements may be adapted for some steps of the procedure.

The results of the test are expressed as a function of *L/S* ratio, in terms of both mg of the substances released per litre of eluate, and mg of the substances released cumulatively per kg of waste material (dry matter).

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Distilled water, demineralized water, de-ionized water or water of equivalent purity ($5 < pH < 7,5$) with a conductivity < 0,1 mS/m according to grade 2 specified in EN ISO 3696.

When the release of biodegradable organic compounds is studied, the leachant might be stabilized with a preservative in order to avoid biodegradation, e.g. sodium azide (NaN_3) at a concentration of 0,1 %.

For ecotoxicological tests, preservatives should not be used. Addition of preservatives may also affect the release of inorganic substances.

5.2 Nitric acid, $c(HNO_3)$ = 1 mol/l.

5.3 Organic solvent, acetone, $(CH_3)_2CO$, pro analyse

6 Equipment

6.1 General

Check the materials and equipment specified in 6.2.2, 6.2.3 and 6.2.7 before use for proper operation and absence of interfering elements that may affect the result of the test (see 8.7).

Calibrate the equipment specified in 6.2.10 to 6.2.13.

6.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

6.2.1 Column

Column made of glass or plastics (e.g. PMMA, PTFE) with an internal diameter of 5 cm to 10 cm and a filling height of about 30 cm ± 5 cm, fitted with filters (see 6.2.6) in bottom and top section. In the top and bottom sections of the column (e.g. made of polychlorotrifluoroethylene (PCTFE)) a filter plate or a thin layer of fine-grained non-reactive material (e.g. fine quartz sand with a particle size range of 1,0 mm to 2,5 mm and SiO_2-content at least 98 % by mass) is applied to ensure proper water flow over the width of the column and as a support for the pre-filter.

Filter plates, quartz sand (SiO_2-content at least 98 % by mass) or glass beads can be washed but when heated to remove any sorbed organic contaminants they shall not be heated to red-heat. They shall also be low in substances (i.e. determinants) which could lead to increased levels in procedural blanks.

NOTE 1 An illustration of the column and its accompanying equipment is given in Annex A.

NOTE 2 Glass of high quality is usually considered adequate for both metals and organic contaminants, particularly, since the pH range usually covered in soil testing does not reach the conditions (pH > 10 and pH < 3) where glass itself is attacked. For ecotoxicity testing, eluates with both metals and organic contaminants are needed, which emphasises the need to generate integrated eluates.

NOTE 3 In case only organic substances are analysed, stainless steel column and fittings can be applied.

6.2.2 Crushing equipment, jaw crusher or a cutting device.

6.2.3 Sieving equipment, with sieves of 4 mm and 10 mm nominal screen size.

6.2.4 Glass or plastic bottles, e.g. high density polyethylene (HDPE)/polypropylene (PP)/polytetrafluoroethylene (PTFE)/polyethyleneterephtalate (PET)

Use bottles with an appropriate volume, and with screw cap, for eluate collection and preservation of eluate samples (rinsed in accordance with EN ISO 5667-3).

6.2.5 Membrane filters, for in-line or off-line filtration of the eluates, with a pore size of 0,45 µm.

Membrane filters for the vacuum or pressure filtration device, fabricated from inert material, which is compatible with the waste. Filter shall be pre-rinsed with demineralized water or similarly clean in order to remove DOC. Filters shall not be used for eluates to be analysed for organic substances.

6.2.6 Pre-filters for the column, with a pore size of 1,5 µm to 8 µm.

NOTE If the eluate is to be used for bio-assays or other specific situations, in which fine particles are important, pre-filters with a larger pore size will preferably be used.

6.2.7 Volumetric pump

The volumetric pump shall be adjustable (peristaltic or equivalent). For small columns, running at a linear velocity of 15 cm/day, the capacity shall be adjustable between 10 ml/h and 20 ml/h and for wide columns, running at the same linear velocity, the capacity shall be adjustable between 40 ml/h and 60 ml/h.

6.2.8 Tubing material

Tubing material inert and adapted to the substances to be analysed (see EN ISO 5667-3). In case only organic substances are to be analysed, stainless steel or glass tubings can be used in contact with the eluate. In case only inorganic substances are analysed PE, PP, PTFE, ETFE, FEP or similar tubing materials can be used.

6.2.9 Centrifuge

(1) Operating at 20 000 *g* to 30 000 *g* using centrifuge tubes of fluorinated ethylene propylene (FEP) or tubes of an alternative material, which is inert with regard to the substances to be measured and suitable to high speed centrifugation.

(2) Alternatively, if a high speed centrifuge is not available, a centrifuge operating at 2 000 *g* to 2 500 *g* using bottles of an appropriate material may be used in combination with increased centrifugation time. Cooling shall be applied to maintain the desired temperature.

6.2.10 Analytical balance, with an accuracy of at least 0,1 g.

6.2.11 Conductivity meter, with an accuracy of at least 0,1 mS/m.

6.2.12 pH meter, with an accuracy of at least 0,05 pH units.

6.2.13 Redox potential meter (optional).

7 Sample pretreatment

7.1 General

Sampling shall be performed in accordance with EN 14899 or a standard derived from EN 14899, in order to obtain a representative sample. Sample pretreatment shall consist of sample preparation, taking a test portion and determining the dry residue of the test sample.

NOTE If it is the intention to determine the leaching behaviour of a waste material, including ageing effects, the test portion will preferably be representative for the material in the practical situation, also as far as ageing conditions are concerned.

7.2 Sample preparation

A laboratory sample shall be obtained of at least 2,5 kg of the material if a small column (diameter 5 cm) is to be used and of at least 10 kg, if a wide column (diameter 10 cm) is to be used. The laboratory sample shall be stored in closed packages and kept under conditions, which prevent any alterations of the waste material.

NOTE 1 Storage at low temperatures (4 °C) may be required, in order to minimise unwanted alterations of the waste material.

To investigate whether the material has to be crushed and to investigate which type of column is to be used, the laboratory sample shall be sieved (see 6.2.3).

The use of the small or wide column shall depend on the particle size of the material, according to Table 1.