SLOVENSKI STANDARD
oSIST prEN 17516:2020

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Karakterizacija granuliranih trdnih snovi (granulatov), ki se lahko uporabljajo kot gradbeni material - Preskus skladnosti izluževanja - Preskus precejanja v koloni s tokom navzgor

Characterization of granular solids with potential for use as construction material - Compliance leaching test - Up-flow percolation test

Charakterisierung von granularen Feststoffen mit Verwertungspotential als Ersatzbaustoff - Übereinstimmungsuntersuchung des Elutionsverhaltens - Perkolationsprüfung im Aufwärtsstrom

Caractérisation des soudes granulées avec un potential d'utilisation comme matériau de construction - Essai de comportement à la lixiviation - Essai de percolation d'un flux ascendant

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Characterization of granular solids with potential for use as construction material - Compliance leaching test - Up-flow percolation test

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

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Contents

Page

European foreword

This document (prEN 17516:2020) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

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

This document specifies an up-flow percolation test to determine the leaching behaviour of granular solid waste with potential for beneficial use as a construction product under standardized percolation conditions. The test is equal to the horizontal up-flow percolation test for construction products (CEN/TS 16637-3:2016), which in turn was elaborated based on the up-flow percolation test for characterization of waste (CEN/TS 14405). The reason for implementing this Technical Specification is to provide a method which is in line with the investigation of the leaching behaviour of granular waste as well as granular construction products, particularly to avoid double testing of waste-derived aggregates that can have potential as construction products.

NOTE Granular solid waste materials are subjected to the Waste Frame Directive (2008/98/EC) where the release of dangerous substances of granular waste is measured according to EN 14405, while construction products derived from the same waste are subjected to the Construction Products Regulation (305/2011) where the release of dangerous substances is measured according to CEN/TS 16637-3.

Background information on characterization of leaching behaviour of construction products can be found in Technical Reports provided by CEN/TC 351 (i.e. CEN/TR 16098).

Introduction

The implementation of a Circular Economy has a high priority on the agenda of the European Commission. To fulfil the needs of a Circular Economy granular solid waste should be used as construction product as much as possible, thus diminishing the amount of landfilling to a minimum and saving natural resources.

The release of dangerous substances upon contact with water results in a potential risk to the environment during the intended utilization of waste materials as construction products. The intent of this document together with EN 14405 or EN 12457-series is to identify the leaching behaviour of granular solid waste with potential for beneficial use as a construction product and thereby allow assessments of the release of regulated dangerous substances to soil, surface water and groundwater under intended use conditions in relation to CE marking of construction products derived from waste.

This document describes an up-flow percolation test for granular solid waste with potential for beneficial use as a construction product. It has been elaborated to avoid double testing, i.e. to assess the compliance with both waste and construction products regulations in one run. This test addresses granular solid waste with existing knowledge on long-term leaching behaviour in waste management scenarios obtained, i.e. using the basic characterization test EN 14405. The specified methods under Clause 5 to 11 (Principle, Reagents, Equipment, Sample preparation, Test procedure, Evaluation of measurement results as well as Documentation and test report) are identical to the corresponding Clauses of CEN/TS 16637-3 (Horizontal up-flow percolation test), with the exception that the term "construction product" has been changed to "solid waste".

In different European countries, tests have been developed to characterize and assess dangerous substances that can be leached from solid matrices (e.g. construction products, contaminated soils, solid waste materials). The intent of these tests is to identify the leaching behaviour of these materials. The complexity of the leaching process makes simplifications necessary. All relevant aspects of leaching behaviour cannot be addressed in one single standard. The test hierarchy for waste is described in the Landfill Directive[1] and the Decision on Annex II of this Directive[2] for the disposal of waste, while guidance on the appropriate leaching test for determining the leaching of dangerous substances from construction products is specified in CEN/TS 16637-1.

The test procedure described in this document should be regarded as a compliance test to be used to verify compliance to regulatory limit values. Column percolation tests according to EN 14405 should be applied for basic characterization of granular solid waste.

NOTE 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 can provide general information on the waste (i.e. 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.

[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.

1 Scope

This document specifies an up-flow percolation test (PT) which is applicable to determine the leaching behaviour of inorganic and non-volatile organic substances from granular solid waste materials with potential for beneficial use as construction products. The test is not suitable for substances that are volatile under ambient conditions. The granular solid waste is subjected to percolation with water as a function of liquid to solid ratio under specified percolation conditions. The method is a once-through column leaching test.

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

NOTE 2 It is not always possible to adjust test conditions simultaneously for inorganic and organic substances and test conditions can also vary between different groups of organic substances. Test conditions for organic substances are generally more stringent than those for inorganic substances. The test conditions are described in a way that they fit for testing of organic substances and are also applicable to inorganic substances depending on the set-up.

Granular solid waste without potential for beneficial use is excluded from the scope.

NOTE 3 Granular solid waste materials without potential for beneficial use can be materials with gas generation or biodegradation during a potential reuse scenario.

NOTE 4 This procedure is generally not applicable to solids that are easily biologically degradable and solids reacting with the leachant, leading to, for example, excessive gas emission or excessive heat release, impermeable hydraulically bound solids or solids that swell in contact with water.

This test is applicable to types of granular solid waste of which the general long-term leaching behaviour is known based on previous investigations.

This up-flow percolation test is performed under specified test conditions, which are equal to the test conditions given in CEN/TS 16637-3 (for granular construction products). It does not necessarily produce results that mimic specific intended use conditions. This test method produces eluates, which can subsequently be characterized by physical, chemical and ecotoxicological methods according to existing standard methods. The results of eluate analysis are presented as a function of the liquid/solid ratio.

NOTE 5 For ecotoxicity testing, eluates representing the release of both inorganic and organic substances are needed. In this document, ecotoxicological testing is meant to include also genotoxicological testing.

Identical test conditions as for CEN/TS 16637-3 are applied in this test in order to allow full comparability for verifying compliance to regulatory limit values of construction products and waste-derived construction products and to avoid double testing. Due to this prerequisite it is accepted that, once CEN/TS 16637-3 is carried out under the legislative context of testing construction products and the granular solid material is rejected as a construction product so that it remains waste, the test results are eligible in the context of testing waste materials as well and that prEN 17516 does not need to be carried out again.

Granular solids that exhibit a saturated hydraulic conductivity of about 10^{-8} m/s or higher can usually be subjected to this test. This procedure is also applicable to granular solid waste showing solidification in the column, if the final hydraulic conductivity is within the specified range.

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 933-1, *Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution - Sieving method*

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

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

CEN/TR 16192, *Waste - Guidance on 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.

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/ui
- IEC Electropedia: available at http://www.electropedia.org/

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 from a leaching test

[SOURCE: EN 16687:2015]

3.3
granular solid waste
waste composed of solid particles with a particle size smaller than a specified size or grading

3.4
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

[SOURCE: IUPAC 1990, 2.5.5]

Note 1 to entry: When the laboratory sample is further prepared by subdividing, cutting, sawing, coring, mixing, drying, crushing, and curing 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/analysis or for the preparation of the test specimen.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

3.5
leachant
liquid that is brought into contact with the test portion in the leaching procedure

Note 1 to entry: Usually, demineralized water is used as leachant for laboratory leaching tests.

[SOURCE: EN 16687:2015]

3.6
leaching behaviour
release and change with time in release from a solid product in contact with a leachant as a function of major release controlling factors

Note 1 to entry: Such factors are diffusion, pH, L/S-ratio or time.

[SOURCE: EN 16687:2015]

3.7
liquid to solid-ratio
L/S
ratio between the total volume of liquid (L) percolated through the solid product and of solid product (S) packed into the column

Note 1 to entry: L/S is expressed in l/kg dry matter.

[SOURCE: EN 16687:2015]

3.8
local equilibrium
LE
situation where chemical equilibrium exists between a substance in solution and the same substance in the solid phase at any point in the column

[SOURCE: EN 16687:2015]

3.9
percolation test
PT
column test
release test method to determine the release of substances from a granular solid waste packed in a column with a leachant percolating through it

[SOURCE: EN 16687:2015]

3.10
release
emission
<leaching> liberation of chemical substances (e.g. non-volatile organic compounds, heavy metals, salts) from granular solid waste into soil, surface water or groundwater into the leachant of a test facility

Note 1 to entry: Release to soil and groundwater is expressed in terms of mass related release (e.g. mg/kg).

[SOURCE: EN 16687:2015]

3.11
release mechanism
physico-chemical processes that control the release of substances from solids into a leachant

Note 1 to entry: In case of solids the main release mechanisms are washout and solubility control. Diffusion and additional factors like pH or DOC also have influence on the mechanism of the release.

Note 2 to entry: The release mechanism for every substance can be determined using the results of the release test (tank leaching test, percolation test). Determination of the release mechanism is relevant for modelling of the source term and so for determination of the effects on soil and water over a time period.

[SOURCE: EN 16687:2015]

3.12
sample

portion of material selected from a larger quantity of material

[SOURCE: IUPAC 1990, 2.1.1]

Note 1 to entry: The manner of selection of the sample should be prescribed in a sampling plan (3.13).

Note 2 to entry: The term “sample” is often accompanied by a prefix (e.g. laboratory sample, test sample) specifying the type of sample and/or the specific step in the sampling process to which the obtained material relates.

3.13
sampling plan
predetermined procedure for the selection, withdrawal, on-site pre-treatment, preservation and transportation of samples to be removed from a population

[SOURCE: EN 16687:2015]

3.14
test portion
amount of the test sample (3.15) taken for testing/analysis purposes, usually of known weight or volume

[SOURCE: IUPAC 1990, 2.5.7]

3.15
test sample
sample, prepared from the laboratory sample (3.4), from which test portions (3.14) are removed for testing or for analysis

[SOURCE: IUPAC 1990, 2.5.6]

4 Symbols and abbreviations

4.1 Symbols

For the purposes of this document, the following symbols apply.

D_{max}	sieve diameter 31,5 mm, 45 mm or 63 mm
E_i	released quantity of a substance per quantity of sample for analysis in eluate fraction *i*, expressed in milligrams per kilogram dry matter (mg/kg dry matter)
S_{cs}	sieve diameter for test sample preparation (crushing) to reduce the amount of oversized product to fit the inner column diameter
U_n	measured cumulative release of a substance for cumulative L/S-ratio *n* including fraction *i* = 1 to *n*, expressed in milligrams per kilogram dry matter (mg/kg dry matter)
V_i	volume of the eluate fraction *i,* expressed in litres (l)
V_L	linear velocity of the leachant through the empty column, expressed in millimetres per day (mm/d)
c_i	concentration of the substance concerned in the eluate fraction *i*
d	inner diameter of the column, expressed in millimetres (mm)
h	packing height of the column, expressed in millimetres (mm)
m_d	dry mass of the test portion, expressed in grams (g)
m_r	mass of the undried test portion, expressed in grams (g)
t	execution time of the test, expressed in days (d)
w_{dr}	dry residue of the granular solid waste, expressed as percentage (%)
Φ	leachant flow rate, expressed in millilitres per hour (in ml/h)

4.2 Abbreviations

For the purposes of this document, the following abbreviations apply.

DOC	dissolved organic carbon
EC	electrical conductivity
ETFE	ethylene tetrafluoroethylene
FEP	fluorinated ethylene propylene
GLHC	test method for granular products with low hydraulic conductivity
HDPE	high-density polyethylene
PAH	polycyclic aromatic hydrocarbon
PCTFE	polychlortrifluorethylene
PTFE	polytetrafluorethylene
TPH	total petroleum hydrocarbons

5 Principle

The percolation test described in this document starts with the representative laboratory sample. The methodology for the collection of a representative laboratory sample is part of EN 14899 or CEN/TR 15310-series.

This document describes a method to determine the release of substances from a granular solid waste with potential for beneficial use as a construction product, with or without size reduction to a maximum particle size, packed in a column with a leachant percolating through it. Pre-equilibration is applied to approach (local) equilibrium at the start. The column size is related to the amount of eluate needed for subsequent analysis and testing and the size of the largest particles in the test portion. 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 substances are rapidly being washed out and which substances are released under the influence of interaction with the matrix. The method is a once-through column leaching test. It is assumed that conditions approach local equilibrium between the granular solid waste and leachant for most of substances in the test.

NOTE The results obtained under local equilibrium (LE) can be up-scaled and used in the modelling of in-use conditions for various scenarios.

The eluate is collected in fractions that are characterized physically and chemically and possibly ecotoxicologically according to existing standards. The results of the test are expressed as a function of the L/S-ratio, in terms of mg of the substances released cumulatively per kg of product or of mg of substance determined per litre of eluate.

The procedure described in this document is based on the more stringent test requirements for determining the release of organic substances and/or for subsequent ecotoxicological testing. If only the release of inorganic substances is to be measured, requirement on equipment (e.g. column and tubing material, centrifugation) may be adapted for some steps of the procedure.

6 Reagents

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

6.1 Leachant

Demineralized water or deionized water or water of equivalent purity with a conductivity < 0,5 mS/m according to grade 3 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 can also affect the release of inorganic substances.

6.2 Rinsing solutions

Nitric acid (pro analyse) $c(HNO_3)$ = 0,1 mol/l

6.3 Organic solvent

E.g. acetone, pro analyse.

7 Equipment

7.1 General

The materials and equipment specified in 7.2 to 7.17 shall be checked before use for proper operation and absence of interfering substances, which can affect the result of the test.

The equipment specified under 7.6, 7.7, 7.8, 7.14 and 7.15 shall be calibrated.

7.2 Column

Column made of plastics or glass for inorganic substances and glass or stainless steel for organic substances with an inner diameter *d* larger than 50 mm and a length that can accommodate a filling height of 300 mm ± 50 mm. In top and bottom of the column a filter plate, glass beads or a thin layer of quartz sand with a particle size range of 1,0 mm to 2,5 mm is applied to ensure proper water flow over the total width of the column.

Filter plates, quartz sand (SiO_2-content at least 98 % by mass) or glass beads can be washed but where 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.

A drawing of the column and accompanying equipment is given in Annex B.

Glass of high quality is usually considered adequate for both metals and organic contaminants, particularly, since the pH range usually covered in product testing does not reach the conditions where glass itself is attacked. For ecotoxicity testing, eluates with both metals and organic contaminants are needed, which emphasizes the need to generate integrated eluates.

In case of organic substances to be analysed, it shall be checked that the material does not interfere significantly with the substances to be measured.

7.3 Packing equipment Rammer with a weight of 125 g in the case of a column with a diameter of 50 mm up to 500 g for a column with a diameter of ≥ 100 mm. Intermediate weights to be linearly interpolated by surface area.

7.4 Membrane for off-line filtration of eluates to be analysed for inorganic substances, with a pore size of 0,45 µm (e.g. PTFE). They shall be of inert material and not adsorb compounds of interest. Filters shall not be used for eluates to be analysed for organic substances.

7.5 Volumetric pump, with an adjustable capacity to be suitable to allow for the flow rate specified according to 9.5.2.

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

7.7 pH meter with accuracy of at least ± 0,05 pH units.

7.8 Electrical conductivity meter with an accuracy of at least 0,1 mS/m.

7.9 Tubing material inert and adapted to the substances to be analysed (see EN ISO 5667-3).

NOTE In case organic substances are to be analysed, stainless steel or glass and FEP can be used in contact with the eluate. In case inorganic substances are analysed PE, PP, PTFE, ETFE, FEP or similar tubing materials can be used.

7.10 Eluate bottles

High quality glass bottles with an appropriate volume and with screw cap with PTFE, ETFE or PTFE inlay, for eluate collection and preservation of eluate samples (in accordance with EN ISO 5667-3). If only inorganic contaminants are to be analysed, alternative bottle materials can be selected, e.g. PE or PP.

7.11 Crushing equipment

Jaw crusher or a cutting device.

7.12 Sieving equipment (dry sieving)

E.g. with sieves 4 mm, 11,2 mm and 22,4 mm nominal screen size.

7.13 Sample splitting equipment

For sub-sampling of laboratory samples, e.g. riffle divider or dividing cross for coning and quartering (optional).

7.14 Redox potential meter

(optional)

7.15 Turbidity meter

As specified in ISO.

7.16 Centrifuge

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

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.

7.17 Filtration

Vacuum filtration device 30 kPa to 70 kPa or pressure filtration device (≤0,5 MPa).

8 Sample preparation

8.1 General

Sample preparation shall consist of preparation of the test sample from the laboratory sample, preparing a test portion and determining the dry residue of the test sample.

Sampling shall be performed in accordance with EN 14899 taking into account the procedures described in the respective product Standard considering guidance given in CEN/TS 16637-1 in order to obtain a representative laboratory sample.

NOTE As usual in an accreditation procedure, the testing laboratory reports on sampling only when it performs sampling itself to produce laboratory sample.