



SLOVENSKI STANDARD
oSIST prEN 17516:2022

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

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

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

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

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91.100.01	Gradbeni materiali na splošno	Construction materials in general

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EUROPEAN STANDARD
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English Version

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

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mit Verwertungspotential als Ersatzbaustoff -
Übereinstimmungsuntersuchung des
Elutionsverhaltens - Perkolationsprüfung im
Aufwärtsstrom

This draft European Standard is submitted to CEN members for second enquiry. It has been drawn up by the Technical Committee CEN/TC 444.

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COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (prEN 17516:2022) 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 second 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 construction product under standardized percolation conditions. The test is equal to the Horizontal up-flow percolation test for construction products (FprEN 16637-3), which in turn was elaborated based on the up-flow percolation test for characterization of waste (CEN/TS 14405:2004). 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 may 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 FprEN 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).

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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 save 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 European Standard together with EN 14405 or EN 12457-series is to identify the leaching behaviour of granular solid waste with potential for beneficial use as 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 basic characterization test EN 14405. The specified methods under Clauses 5 to 12 (Principle, Reagents, Equipment, Sample preparation, Test procedure, Evaluation of measurement results, Documentation and Test Report as well as Test Performance) are identical to the corresponding clauses of FprEN 16637-3 (Horizontal up-flow percolation test) with the exception that the term “construction product” has been changed to terms relevant for this standard such as “granular solid waste with potential to be reused as construction product” or similar.

In the different European countries, tests have been developed to characterize and assess dangerous substances which 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 Directive¹ and the Decision on Annex II of this Directive² for disposal of waste, while guidance of appropriate leaching test for determining leaching of dangerous substances from construction products is specified in CEN/TS 16637-1.

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

NOTE 1 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 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

(1) 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 solids with potential for use as construction material. The test is not suitable for substances that are volatile under ambient conditions. The granular solids are 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.

(2) This up-flow percolation test is performed under specified test conditions for granular solids with potential for use as construction material and 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. The test results enable the distinction between different leaching behaviour.

NOTE 2 It is not always possible to adjust test conditions simultaneously for inorganic and organic substances. 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 generally described in a way that they fit testing organic substances and are also applicable to inorganic substances depending on the set-up.

NOTE 3 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.

NOTE 4 Granular solid waste materials with a low hydraulic conductivity that can cause detrimental pressure build-up are not supposed to be subjected to this test.

NOTE 5 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.

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

NOTE 6 Granular solid waste materials without potential for beneficial use can be materials with gas generation or biodegradation during a potential reuse scenario.

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

(3) In this document the same test conditions as for FprEN 16637-3 (CEN/TC 351/WG 1) are applied in order to allow full comparability of testing construction products and waste derived construction products to avoid double testing. The FprEN 16637-3 test results are eligible in the context of testing granular solids with potential for use as construction material as well.

NOTE 7 If a leaching test according to FprEN 16637-3 has been performed, additional prEN 17516 testing does not need to be carried out.

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

FprEN 16637-3, *Construction products: Assessment of release of dangerous substances — Part 3: Horizontal up-flow percolation test*

EN 14405, *Characterization of waste — Leaching behaviour tests — Up-flow percolation test (under specified conditions)*

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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1

eluate

solution obtained from a leaching test

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[SOURCE: EN 16687:2015 [7], 4.2.7]

3.2

granular solid waste

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

3.3

laboratory sample

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

Note 1 to entry: When the laboratory sample is further prepared by subdividing, cutting, sawing, coring, drying, grinding, mixing, 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 or for analysis.

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.

[SOURCE: EN 16687:2015 [7], 3.2.1]

3.4**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 [7], 4.2.6]

3.5**leaching behaviour**

release and change with time in release from a granular solid waste 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 [7], 4.2.9 (edited to refer to granular waste)]

3.6**liquid to solid ratio** **L/S**

ratio between the total volume of liquid (L) percolated through the granular solid waste and of granular solid waste (S) packed into the column

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

[SOURCE: EN 16687:2015 [7], 4.2.13 (edited to refer to granular waste)]

3.7**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

Note 1 to entry: Even when local equilibrium exists at all points along the column the equilibrium concentrations may be different at different points.

[SOURCE: EN 16687:2015 [7], 4.2.14]

3.8**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 [7], 4.2.4 (edited to refer to granular waste)]

3.9**release****emission**

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

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

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Note 2 to entry: The terms “emission” and “release” have fundamentally the same meaning. However, it is often a tradition to use the term “emission” when describing liberation of chemical substances or radiation into air and to use the term “release” when describing the liberation of chemical substances into soil or water.

[SOURCE: EN 16687:2015 [7], 4.2.15 (edited to refer to granular waste)]

3.10**release mechanism**

physical-chemical processes that control the release of substances from a granular solid waste into a leachant

Note 1 to entry: In case of granular solid waste 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 [7], 4.2.17 (notes edited to refer to percolation only and to refer to granular waste)]

3.11**sample**

portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be prescribed in a sampling plan (3.12).

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.

[SOURCE: EN 16687:2015 [7], 3.1.5]

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3.12**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, 3.1.6]

3.13**test portion****analytical portion**

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

[SOURCE: EN 16687:2015 [7], 3.2.3]

3.14**test sample**

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

[SOURCE: EN 16687:2015 [7], 3.2.2]

3.15**limit of quantification****LOQ**

lowest value of an analyte (determinant) that can be determined with an acceptable level of accuracy and precision, generally determined as three times the limit of detection of the method

[SOURCE: EN 16687:2015 [7], 4.1.14]

4 Symbols and abbreviations**4.1 Symbols**

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

c_i	concentration of the substance concerned in the eluate fraction i , in $\mu\text{g/l}$;
D_{max}	sieve diameter, in mm; NOTE Common sizes are 31,5 mm, 45 mm or 63 mm.
d_i	inner diameter of the column, in mm;
d_p	is the density of the solid, expressed in kilograms per cubic metre (kg/m^3).
E_i	released quantity of a substance per quantity of sample for analysis in eluate fraction i , in mg/kg dry matter;
h	is the height or layer thickness of the construction, in m;
k_C	constant that represents the speed of the release of a certain substance;
k_e	number of eluates collected in the test;
m_d	dry mass of the test portion, in g;
m_r	mass of the undried test portion, in g;
m_W	mass of the (moist) test portion in the column, in g;
S_{CS}	sieve diameter for test sample preparation (crushing) to reduce the amount of oversized part of the sample to fit the inner column diameter, in mm;
t	execution time of the test, in days;
U_n	measured cumulative release of a substance for cumulative L/S ratio n including fraction $i = 1$ to n , in mg/kg dry matter;
V_i	volume of the eluate fraction i , in l;
V_L	linear velocity of the leachant through the empty column, in mm per day;
w_{dr}	dry residue of the granular solid waste, in %;
Φ	leachant flow rate, in ml per hour.

prEN 17516:2022 (E)**4.2 Abbreviations**

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

DL	detection limit
DOC	dissolved organic carbon
EC	electrical conductivity
EOTA	European organization for technical assessment
ETA	European technical assessment
ETFE	ethylene tetrafluoroethylene
FEP	fluorinated ethylene propylene
FPC	factory production control
GLHC	test method for granular products with low hydraulic conductivity
HDPE	high-density polyethylene
hEN	harmonized European Standard
PAH	polycyclic aromatic hydrocarbon
PCTFE	polychlorotrifluoroethylene
PT	percolation test
PTFE	polytetrafluoroethylene
RDS	regulated dangerous substances
TPH	total petroleum hydrocarbons

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5 Principle**5.1 General principles**

(1) The percolation test described in this document starts with the representative laboratory sample. The methodology for the collection of a representative laboratory sample is in accordance with the procedures described in EN 15310-series and EN 15002 considering guidance given in FprEN 16637-1.

(2) This document describes a method to determine the release of substances from a granular solid waste with potential for beneficial use as 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 inorganic 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.

(3) 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 granular solid waste or of mg of substance determined per litre of eluate.

(4) 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, requirements on equipment (e.g. column and tubing material, centrifugation) are specified in the relevant clauses for some steps of the procedure.

5.2 Number of eluates

(1) By reference the percolation test requires the analysis of seven eluates to determine the release of dangerous substances for an L/S ratio up to 10. These results are needed for characterization of the granular solid waste and allow the determination of the release mechanism according to Annex D.

(2) For specific scenarios, other than characterization, it may be beneficial to combine eluates or adopt other testing schemes based on the one of this document. Examples of such scenarios are:

- a) *Release at a specific L/S ratio.* If it is sufficient to know the cumulative release at a predetermined L/S ratio, for instance $L/S = 2$, the test can be stopped after the collection of the fifth eluate fraction.
- b) *Long-term monitoring.* In the case scenario descriptions require much higher values of the L/S ratio than $L/S = 10$ (e.g. usage of granular solids as construction products functioning as unbound drains), additional fractions may be collected until the relevant range of L/S ratio has been covered. Additionally, for specific monitoring scenarios or research tasks, a closer sampling frequency may be applied in a certain range of L/S ratios.
- c) *Factory production control.* In the case of FPC it is checked whether the product conforms to the previously determined characteristics (type testing). Different test methods may be used:
 - In the case of testing in accordance with the standard it is sufficient to check the release for the specified L/S ratio and to collect and combine successive eluates in proportion to the volumes, e.g. L/S 0-2 l/kg dry matter or L/S 0-10 l/kg dry matter.
 - Following an alternative testing procedure (indirect test method), the release at for instance L/S 0-0,5 l/kg dry matter or L/S 0-1 l/kg dry matter, may be extrapolated to predict the release after L/S 0-10 l/kg dry matter or another appropriate L/S ratio. Also other leaching tests may be used. Further guidance on the use of indirect test methods is given in Clause 13.

(3) As long as the adapted test scheme is based on the one of this document and the test is performed according to this document, the cumulative release at a specified L/S ratio is equal to the cumulative release based on the reference test performance. The release mechanisms specified in Annex D of this document cannot be determined if less than seven eluates are analysed.

6 Reagents

6.1 General

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

6.2 Leachant

Use as a leachant demineralized water or deionized water or water of equivalent purity with a conductivity $< 0,5$ mS/m or better.

When the release of biodegradable organic compounds is studied, the eluate might be stabilized with a preservative in order to avoid biodegradation.