SLOVENSKI STANDARD
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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - 3. del: Horizontani preskus precejanja v koloni s tokom navzgor

Construction products - Assessment of release of dangerous substances - Part 3: Horizontal up-flow percolation test

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Teil 3: Horizontale Perkolationsprüfung im Aufwärtsstrom

Produits de construction - Evaluation de l'émission de substances dangereuses - Partie 3 : Essai horizontal de percolation à l'écoulement ascendant

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Construction products: Assessment of release of dangerous substances - Part 3: Horizontal up-flow percolation test

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Teil 3: Horizontale Perkolationsprüfung im Aufwärtsstrom

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 351.

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

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

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European foreword

This document (prEN 16637-3:2021) has been prepared by Technical Committee CEN/TC 351 “Construction Products: Assessment of release of dangerous substances”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

This document will supersede CEN/TS 16637-3:2016.

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

— Transfer of technical specification into European Standard;

— Addition of validation data from interlaboratory validation on repeatability and reproducibility (see Clause 12 and Annex E);

— Addition of requirements on the number of eluates (see 5.2);

— Alignment of the test conditions with the test conditions which are specified in prEN 17516;

— Updating of normative and informative cross-references.

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

This document was elaborated on the basis of CEN/TS 14405 [1].

This document specifies an up-flow percolation test to determine the leaching behaviour of granular construction products under standardized percolation conditions.

EN 16637, *Construction products: Assessment of release of dangerous substances*, consists of the following parts:

— Part 1: *Guidance for the determination of leaching tests and additional testing steps*,

— Part 2: *Horizontal dynamic surface leaching test*,

— Part 3: *Horizontal up-flow percolation test*.

prEN 16637-1 deals with the determination and use of test methods for leaching of construction products taking specific situations into account. prEN 16637-2 specifies a dynamic surface leaching test for determination of surface dependent release of substances from monolithic or plate-like or sheet-like construction products or granular construction products with low hydraulic conductivity under standardized conditions.

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 [2], CEN/TR 16496 [3]).

Introduction

The European Standards prEN 16637-1, prEN 16637-2 and prEN 16637-3 are developed to assess the release of regulated dangerous substances (RDS) from construction products into soil, surface water and groundwater in the framework of Mandate M/366. The horizontal test methods developed under the Mandate M/366 are intended to be used to show compliance with notified regulations. The tests cover the release of substances from construction products and in particular, those that are regulated in notified regulations in one or more EU Member States.

prEN 16637-1 specifies how the CEN Technical Product Committees and EOTA experts should determine the appropriate leaching test for the determination of the release of RDS from a construction product into soil, surface water and groundwater. prEN 16637-1 gives background information for CEN Technical Product Committees on the following aspects:

a) description of the intended conditions of use of the construction product (e.g. above ground exposed to the precipitation, or shielded from direct infiltration, in surface or groundwater) with respect to the release of RDS into soil, surface water and groundwater;

b) identification of main release mechanisms, and the appropriate leaching test for a given construction product.

prEN 16637-2 specifies a horizontal test to assess surface dependent release from monolithic, plate-like or sheet-like construction products (tank test).

prEN 16637-3 specifies a horizontal test to assess release from granular construction products.

The test methods can be used for both steps in the hierarchy (type testing (TT) and factory production control (FPC)) and form the reference tests for the intended uses and conditions specified in prEN 16637-1. In this hierarchy of testing conditionally "indirect tests" can be used, but are not specified.

The release of substances upon contact with water results in a potential risk to the environment during the intended use of construction products. The intent of these tests is to identify the leaching behaviour of construction products and thereby allow assessments of the release of RDS from such products to soil, surface water and groundwater under intended conditions of use in relation to CE marking and assessment and verification of constancy of performance.

This document does not address impact assessment. However, since the test methods described in the document may be used in the context of impact assessments and regulation based on impact assessments, some guidance on this issue is provided in prEN 16637-1:—, Annex A (informative).

In addition to existing validation results, in 2011 CEN/TC 351 began an extensive research program on robustness validation of the existing tank leaching and percolation tests. This was carried out by a consortium of European experts on 20 construction products to unify differences from the protocols of the different CEN Members and to check the influence of testing conditions on the test result (e.g. temperature, flow rate, renewal scheme). The results ([4]) of the research program confirmed the robustness of the horizontal tests known from former works. Conclusions from the program have been implemented into the Technical Specifications for the test methods. The performance of the leaching tests regarding repeatability and reproducibility was deduced from a second validation step and respective data ([5], [6]) are included in prEN 16637-2 and in this document.

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 construction products. The test is not suitable for substances that are volatile under ambient conditions. The construction products 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.

(2) This up-flow percolation test is performed under specified test conditions for construction products 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 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. 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 Construction products 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 products that are easily biologically degradable and products reacting with the leachant, leading, for example, to excessive gas emission or excessive heat release, impermeable hydraulically bound products or products that swell in contact with water.

(3) In this document the same test conditions as for prEN 17516 (CEN/TC 444/WG 1) are applied in order to allow full comparability of testing construction products and waste derived construction products to avoid double testing. The prEN 17516 test results are eligible in the context of testing construction products as well.

NOTE 6 If a leaching test according to prEN 17516 has been performed, additional prEN 16637-3 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*

prEN 16637-1, *Construction products: Assessment of release of dangerous substances – Part 1: Guidance for the determination of leaching tests and additional testing steps (under development)*

CEN/TS 17195, *Construction products: Assessment of release of dangerous substances - Analysis of inorganic substances in eluates*

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

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

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

3.2
granular product
product composed of solid particles with a particle size smaller than a specified size or grading

Note 1 to entry: Granular products are usually tested by a percolation test.

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 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 [7], 4.2.9]

3.6
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 [7], 4.2.13]

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 construction product packed in a column with a leachant percolating through it

[SOURCE: EN 16687:2015 [7], 4.2.4]

3.9
release
emission
liberation of chemical substances (e.g. non-volatile organic compounds, heavy metals, salts) from a construction product into soil, surface water or groundwater into the leachant of a test facility

Note 1 to entry: Release to soil, surface and groundwater may be expressed in terms of area related release (tank leaching test, e.g. mg/m^2) or in terms of mass related release (percolation test, e.g. mg/kg).

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]

3.10
release mechanism
physical-chemical processes that control the release of substances from a solid construction product into a leachant

Note 1 to entry: In case of granular products 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)]

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 (3.13).

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.

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

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 at a level of three times the limit of detection

[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 µg/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 construction product, expressed in kilograms per cubic metre (kg/m³).

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 product 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 construction product, in %;

Φ leachant flow rate, in ml per hour.

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	polytetrafluorethylene
RDS	regulated dangerous substances
TPH	total petroleum hydrocarbons
TT	type testing

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 part of the respective product standard.

(2) This document describes a method to determine the release of substances from 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 construction product 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 product 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, less stringent requirement on equipment (e.g. column and tubing material, centrifugation) may be adapted 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 a *L/S* ratio up to 10. These results are needed for characterization of the construction product 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. 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 an other 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 according to grade 3 specified in EN ISO 3696:1995 or better.

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

For ecotoxicological tests, preservatives are not allowed.

6.3 Rinsing solutions

Use as rinsing solution nitric acid (pro analysis), $c(HNO_3)$ = 0,1 mol/l and an organic solvent (e.g. acetone, pro analysis).

7 Equipment

7.1 General

(1) Check the materials and equipment specified in 7.2 to 7.17 before use for proper operation and absence of interfering substances that may affect the result of the test.

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

7.2 Column

(1) Column made of plastics or glass for inorganic substances and glass or stainless steel for organic substances with an inner diameter d_i 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.

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

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

(4) 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.

(5) 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).

Membranes 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 9.4.3.

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

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

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