
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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91.100.01	Gradbeni materiali na splošno	Construction materials in general

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CEN/TS 16637-3

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English Version

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

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

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

This Technical Specification (CEN/TS) was approved by CEN on 15 March 2016 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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CEN/TS 16637-3:2016 (E)**European foreword**

This document (CEN/TS 16637-3:2016) 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 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 December 2016 and conflicting national standards shall be withdrawn at the latest by December 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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

This Technical Specification was elaborated on the basis of CEN/TS 14405.

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

CEN/TS 16637-1 deals with the determination and use of test methods for leaching of construction products taking specific situations into account. CEN/TS 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 and CEN/TR 16496).

According to the CEN-CENELEC Internal Regulations, the national standards organizations 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, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This introduction describes the interactions and interrelations between the three Technical Specifications CEN/TS 16637-1, CEN/TS 16637-2 and CEN/TS 16637-3 developed to assess the release of dangerous substances from construction products into soil, surface water and ground water in the framework of the 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.

CEN/TS 16637-1 specifies how the CEN Technical Product Committees and EOTA experts are to determine the appropriate leaching test for the determination of the release of Regulated Dangerous Substances from a construction product into soil, surface water and groundwater.

CEN/TS 16637-2 describes a test to assess surface dependent release from monolithic, plate-like or sheet-like construction products while CEN/TS 16637-3 describes a horizontal test to assess release from granular construction products. The test methods specified in CEN/TS 16637-2 and CEN/TS 16637-3 can be used for both steps in the hierarchy (type testing and factory production control) and are supposed to be used as the reference test for the intended uses and conditions specified in CEN/TS 16637-1. In this hierarchy of testing conditionally "indirect tests" can be used, but are not specified. CEN/TS 16637-2 includes a procedure with a modified tank leaching test GLHC for testing granular products with a low hydraulic conductivity as determined by failure to achieve a sufficient water flow in the percolation test.

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 Regulated Dangerous Substances from such products to soil, surface water and groundwater under intended use conditions in relation to CE marking.

Technical Product Committees are expected to apply the test standards developed in CEN/TC 351 for their products in order to test the potential release of Regulated Dangerous Substances to soil, surface water and groundwater. CEN/TS 16637-1 is intended to provide clear procedures to determine which test method is appropriate for a given product. CEN/TS 16637-1 aims to provide the information, needed in a CEN Technical Product Committee, on how to deal with the relevant test method(s) to enable the producer to declare a performance in the CE marking as a result of the test. CEN Technical Product Committees are referred to the informative Annexes A and B of CEN/TS 16637-1 and to CEN/TR 16098 for background information on the following aspects:

- a) identification of the products addressed in the product standards which have relevance with respect to the release of dangerous substances into soil, surface water and groundwater (products only applied in the interior of buildings are not subject to testing for these properties);
- b) description of the intended use conditions of the construction product (e.g. above ground exposed to the precipitation, or shielded from direct infiltration, in surface or ground water) in respect to the release of dangerous substances into soil, surface water and groundwater;
- c) identification of main release mechanisms.

Impact assessment is not part of the work of CEN/TC 351.

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 in different CEN-Member States and to check the influence of testing conditions on the test result (e.g.

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temperature, flow rate, renewal scheme, particle size in the percolation test etc., see bibliography. The results 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. However, the performance of the leaching test regarding repeatability and reproducibility is dependent on the tested construction product and on the testing conditions. When these Technical Specifications of the horizontal leaching tests are adopted by CEN, the leaching tests referred to in these Technical Specifications will not yet be fully validated. No data will be available on repeatability and reproducibility for the range of construction products. For other, sometimes comparable, matrices performance data are available from national as well as EU validation studies.

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1 Scope

(1) This Technical Specification 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 and test conditions may 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.

Construction products 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 materials showing solidification in the column, if the final hydraulic conductivity is within the specified range. Inert granular material should not be added to improve permeability in order to enable their testing.

NOTE 4 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.

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

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

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

CEN/TS 16637-1, *Construction products - Assessment of release of dangerous substances - Part 1: Guidance for the determination of leaching tests and additional testing steps*

EN 16687, *Construction products - Assessment of release of dangerous substances - Terminology*

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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)*

ISO 7027, *Water quality - Determination of turbidity*

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 from a leaching test

[SOURCE: EN 16687]

**3.3
granular construction 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.

**3.4
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]

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]

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]

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]

3.9**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]

3.10**release****emission**

<leaching> 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/m²) or in terms of mass related release (percolation test, e.g. mg/kg).

[SOURCE: EN 16687]

3.11**release mechanism**

physico-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.

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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]

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]

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3.14**test portion**

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

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[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 Technical Specification, 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 construction product, expressed as percentage (%)
Φ	leachant flow rate, expressed in millilitres per hour (in ml/h)

4.2 Abbreviations

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For the purposes of this Technical Specification, 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	polychlorotrifluoroethylene
PTFE	polytetrafluoroethylene
TPH	total petroleum hydrocarbons

5 Principle

(1) The percolation test described in this Technical Specification 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 Technical Specification 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

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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 standard 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.

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 may also affect the release of inorganic substances.

6.2 Rinsing solutions

Nitric acid (pro analyse) $c(\text{HNO}_3) = 0,1 \text{ mol/l}$

6.3 Organic solvent

E.g. acetone, pro analyse.

7 Equipment**7.1 General**

(1) 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.

(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 larger than 50 mm and a length that can accommodate a filling height of $300 \text{ mm} \pm 50 \text{ mm}$. 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 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.

(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 $\geq 100 \text{ mm}$. 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 $0,45 \mu\text{m}$ (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.

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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 $\pm 0,05 \text{ pH}$ 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.