

## SLOVENSKI STANDARD kSIST-TS FprCEN/TS 16637-2:2013

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# Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - 2. del: Horizontalni dinamični preskus za izluževanje s površine

Construction products - Assessment of release of dangerous substances - Part 2: Horizontal dynamic surface leaching test

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Teil 2: Horizontale dynamische Oberflächenauslaugprüfung

Produits de construction - Evaluation de l'émission de substances dangereuses - Partie 2: Essais horizontaux et dynamiques de la lixivation des surfaces

Ta slovenski standard je istoveten z: FprCEN/TS 16637-2

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# TECHNICAL SPECIFICATION SPÉCIFICATION TECHNIQUE TECHNISCHE SPEZIFIKATION

## FINAL DRAFT FprCEN/TS 16637-2

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#### **English Version**

# Construction products - Assessment of release of dangerous substances - Part 2: Horizontal dynamic surface leaching test

Produits de construction - Evaluation de l'émission de substances dangereuses - Partie 2: Essais horizontaux et dynamiques de la lixivation des surfaces Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Teil 2: Horizontale dynamische Oberflächenauslaugprüfung

This draft Technical Specification is submitted to CEN members for formal vote. It has been drawn up by the Technical Committee CEN/TC 351.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

**Warning**: This document is not a Technical Specification. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a Technical Specification.

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

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#### **Foreword**

This document (FprCEN/TS 16637-2:2013) 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 Formal Vote.

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

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

This Technical Specification was elaborated on the basis of CEN/TS 15863 which is based on NEN 7375:2005.

This Technical Specification 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.

FprCEN/TS 16637-1 deals with the determination and use of test methods for leaching of construction products taking specific situations into account. Technical Specification prCEN/TS 16637-3 specifies an upflow percolation test to determine the leaching behaviour of granular construction products under standardized percolation conditions.

Further information on characterisation of leaching behaviour of construction products can be found in Technical Reports provided by CEN/TC 351 (i.e. CEN/TR 16098, CEN/TR 16496).

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#### Introduction

The informative introduction describes the interactions and interrelations between the three Technical Specifications prCEN/TS 16637-1, prCEN/TS 16637-2 and prCEN/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.

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

FprCEN/TS 16637-2 and prCEN/TS 16637-3 contain the two horizontal leaching tests that are needed to address the leaching properties of the construction products. FprCEN/TS 16637-2 describes a test to assess surface dependent release from monolithic, plate-like or sheet-like construction products while prCEN/TS 16637-3 describes a test to assess release from granular construction products. The test methods specified in FprCEN/TS 16637-2 and prCEN/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 FprCEN/TS 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 Regulated Dangerous Substances from such products to soil, surface water and groundwater under intended conditions of use 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. FprCEN/TS 16637-1 is intended to provide clear procedures to determine which test method is appropriate for a given product. FprCEN/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 FprCEN/TS 16637-1:2013 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 conditions of use 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 Regulated 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. temperature, flow rate,

renewal scheme, etc. [1]). 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 a Dynamic Surface Leaching Test (DSLT) which is aimed at determining the release per unit surface area as a function of time of inorganic and/or non-volatile organic substances from a monolithic, plate- or sheet-like product, when it is put into contact with an aqueous solution (leachant). The test method is not suitable for substances that are volatile under ambient conditions.
- (2) This test is a parameter specific test focusing on identifying and specifying parameter specific properties tested under specified conditions. It is not aimed at simulating real situations. The application of results to specific intended conditions of use may be established by means of modelling (not included in this Technical Specification).
- (3) The modification for granular construction products with low hydraulic conductivity (Annex A) applies for granular particles with so little drainage capacity between the grains that percolation in percolation tests and in practice is nearly impossible.
- (4)The test method applies to more or less regularly shaped test portions consisting of monolithic test pieces with minimum dimensions of 40 mm in all directions (volume > 64 000 mm³ (64 cm³)). It also applies to plate-or sheet-like products with surface areas of minimum 10 000 mm² (100 cm²) exposed to the leachant. Products designed to drain water (e.g. draining tiles, porous asphalt) and monolithic granular products according to FprCEN/TS 16637-1:2013, Table 1, are also tested by this test method. All products to be tested are assumed to maintain their integrity over a time frame relevant for the considered intended use.
- (5) Metals, metallic coatings and organic coatings on metals are excluded from the scope of FprCEN/TS 16637-2 because the principles of this test (diffusion) are not obeyed by these products. Guidance on the need for testing of these products is under consideration.
- (6) For some coatings (e.g. some renders with organic binders according to EN 15824) in intermittent contact to water, physical and chemical properties might be changed in permanent contact with water. For these products FprCEN/TS 16637-2 is not appropriate.
- (7) Guidance on the applicability of the test method to a given product is outlined in FprCEN/TS 16637-1.
- NOTE 1 This test method is only applicable if the product is chemically stable and the matrix does not dissolve. For construction products that may be used in contact with water this usually should not be the case as construction products should then be dimensionally stable. If a product may substantially wear in its intended use, the test cannot provide proper (37-2-2014 information. If the product contains a substantial amount of water-soluble compounds, e.g. gypsum or anhydrite, the matrix may (partially) dissolve and lead to dimensional instability of the test piece. In this case the test standard also cannot be used.
- NOTE 2 Volatile organic substances include the low molecular weight substances in mixtures such as mineral oil.
- NOTE 3 It is not always possible to optimise test conditions simultaneously for inorganic and organic substances and optimum test conditions may also vary between different groups of organic substances. Test requirements for organic substances are generally more stringent than those for inorganic substances. The test conditions suitable for measuring the release of organic substances will generally also be applicable to inorganic substances.

#### 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 16192, Characterization of waste — Analysis of eluates

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

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

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

#### 3 Terms and definitions

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

#### 3.1

#### eluate

solution obtained from a leaching test

#### 3.2

#### 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, 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.

#### 3.3

#### leachant

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

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

#### 3.4

#### leaching behaviour

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

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

#### 3.5

#### release mechanism

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

Note 1 to entry: In the case of monolithic products, the main release mechanisms for substances are diffusion, dissolution, initial surface wash-off and depletion. Additional factors like pH or DOC also have influence on the magnitude 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). 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.

#### 3.6

#### liquid to surface area-ratio

L/A

ratio between the amount of liquid (L) which in a given step of the test is in contact with the exposed surface area (A) of the test portion

Note 1 to entry: L/A is expressed in  $I/m^2$ .

#### 3.7

#### monolithic product

product which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered intended conditions of use

Note 1 to entry: Monolithic products are usually tested by a dynamic surface leaching test.

#### 3.8

#### plate-like product

product formed as a semi-rigid or rigid plate, which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered intended conditions of use

Note 1 to entry: Plate-like products are usually tested by a dynamic surface leaching test.

#### 3.9

#### 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 ground water or into the leachant of a test facility

Note 1 to entry: Release to soil, surface and groundwater can 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).

#### 3.10

#### release rate

product specific rate describing the mass of a substance released from a product at a given time interval in a laboratory leaching test

Note 1 to entry: The release rate is expressed in  $(mg/m^2)/d$  (tank leaching test) or in (mg/kg)/d (percolation test). The first, area related release rate is also called "flux".

## 3.11 leachant renewal scheme

selection of time intervals after which the leachant is renewed

#### 3.12

#### sample

portion of material selected from a larger quantity of material

[SOURCE: UIPAC 1990, 2.1.1]

Note 1 to entry: The manner of selection of the sample is usually prescribed in a sampling plan (x, y, z).

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

#### sheet-like product

product formed as a flexible or semi-flexible sheet, which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered intended conditions of use

Note 1 to entry: Sheet-like products are usually tested by the dynamic surface leaching test.

#### 3.14

#### test piece

single monolithic, plate- or sheet-like piece as part of the test portion

#### 3.15

#### test portion

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

[SOURCE: IUPAC, 1997]

Note 1 to entry: The test portion might consist of more than one test piece.

#### 3.16

#### test sample

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

[SOURCE: IUPAC, 1990, 2.5.6]

#### 3.17

#### exposed surface

A

<leaching> part of the total surface of the test portion exposed to the leachant

#### 3.18

#### compacted granular product

granular product with a low permeability, due to very small pores between the particles

Note 1 to entry: Compacted granular products are usually tested by the method for granular construction products with low hydraulic conductivity, because the percolation test is not applicable due to the low permeability of the products.

#### 3.19

#### monolithic granular product

granular product with specific requirements on the grain size distribution to be tested in the dynamic surface leaching test (DSLT)

#### 3.20

## release method for granular construction products with low hydraulic conductivity GLHC

release test method in which a construction product with low hydraulic conductivity is exposed with one defined surface to a leachant renewed at subsequent time intervals

#### 3.21

#### dynamic surface leaching test

#### **DSLT**

release test method in which a monolithic, sheet-like or plate-like product is immersed in a leachant renewed at subsequent time intervals

#### 3.22

#### Limit of quantification

#### LOQ

lowest value of a determinant that can be determined with an acceptable level of accuracy and precision, determined at a level of 3 times the Limit of Detection

#### 4 Symbols and abbreviations

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

A geometric area of the test portion in m<sup>2</sup> exposed surface of test piece(s) in m<sup>2</sup>

 $c_i$  concentration of the substance in eluate i, in  $\mu g/I$ 

 $m_a$  loss of weight [g/m<sup>2</sup>]

 $m_{\rm s}$  mass of the solid matter that has fallen off during the test [g]

 $R_n$  cumulative area release of the substance for period n including fraction i = 1 to n, in mg/m<sup>2</sup>

 $r_{\rm i}$  area release of the substance in fraction i, in mg/m<sup>2</sup>

P test piece

 $V_{\mathsf{P}}$  volume of the text portion

 $V_{\rm I}$  volume of the leachant, in l

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

DOC Dissolved Organic Carbon

DSLT Dynamic Surface Leaching Test

GLHC method for Granular construction products with Low Hydraulic Conductivity

FEP Fluorinated Ethylene Propylene

FPC Factory Production Control Comment

HDPE High-Density Polyethylene

PAH /standa Polycyclic Aromatic Hydrocarbon //a99e030f-81fd-49ef-902e-23ac74864fb8/sist-ts-cen-ts-16637-2-2014

PMMA polymethylmethacrylate

PE polyethylene

PET polyethyleneterephthalate

PP polypropylene

PTFE polytetrafluorethylene

TPH Total Petroleum Hydrocarbons

#### 5 Principle

(1) This Technical Specification describes a method to determine as a function of time the release of substances from a monolithic, plate-like or sheet-like product with a leachant in contact with its surface.

- (2) The test portion of the product is placed in a reactor/leaching vessel and the exposed surface is completely submerged in a leachant. The leachant is introduced in the reactor up to a given volume of liquid to surface area ratio (*L/A* ratio), at a given temperature and renewed at predetermined time intervals.
- (3) The following test conditions are fixed:
- a) the type of leachant (standard leachant pH-neutral demineralised water as specified in 6.2);
- b) the temperature;
- c) the L/A ratio ( $l/m^2$ );
- d) the total duration of the test;
- e) the number of eluates to be collected at fixed time intervals.
- (4) This test method produces eluates, which shall subsequently be characterised by physical, chemical and ecotoxicological methods according to existing standard methods. Special precautions may be necessary with respect to the choice of equipment design and materials, handling of eluates and prevention of biodegradation when the leaching of non-volatile organic substances is of interest.
- (5) For those products that have a too low hydraulic conductivity to perform a regular percolation test according to prCEN/TS 16637-3 (e.g. clayey or paste-like products), a modified "Method for granular construction products with low hydraulic conductivity (GLHC) is given in Annex A. This method is different from the DSLT with respect to the test equipment and sample preparation in order to ensure the exposure of a well-defined surface. All other conditions (e.g. liquid to area ratio, renewal times, leachant and data handling) are the same as for the regular DSLT.
- (6) The eluate fractions are characterised physically and chemically according to existing standards.
- (7) Loss of solid material from the test piece during testing is an indication of limited long term stability.
- (8) The results of the test are expressed as a function of time, in terms of both milligrams of substances released cumulatively per m<sup>2</sup> of geometric surface area of the product exposed to leaching, and milligrams of the substances released per liter of eluate, each after 64 days.
- NOTE described in the results may be additionally expressed and reported in terms of the flux (milligrams of substances released 10.14 per m<sup>2</sup> per second) for each eluate fraction as a function of time.
- (9) The test conditions (L/A ratio, leachant renewal scheme) have been designed such, that the identification of leaching mechanisms and their relative importance is enabled. The main leaching mechanisms that can be distinguished and identified are:
- a) diffusion (through the pores and / or from the surface to the bulk of the leachant);
- b) initial surface wash-off;
- c) other processes (e.g. solubility control, changes in speciation, depletion).

#### 6 Reagents

#### 6.1 General

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

#### 6.2 Leachant

Use as a leachant demineralised water or deionised 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 organic compounds is studied, the leachant might be stabilised with a preservative in order to avoid biodegradation, e. g. sodium azide. For ecotoxicological tests, preservatives should not be used. Addition of preservatives can also affect the release of inorganic substances.

#### 6.3 Rinsing solutions

Use as rinsing solution nitric acid (pro analyse), c(HNO3) = 0,1 mol/l and an organic solvent (e.g. acetone, pro analyse).

#### 7 Equipment

#### 7.1 General

- (1) Check the materials and equipment specified in 7.2 to 7.11 and 7.13 before use for proper operation and absence of interfering elements that may affect the result of the test (see 9.6).
- (2) The equipment specified in 7.4, 7.9, 7.10 and 7.12 shall also be calibrated.
- (3) Usual laboratory apparatus, and in particular the following:

#### 7.2 Leaching vessels or tanks

- (1) Use leaching vessels or tanks of different size from glass or plastics (e.g. HDPE, PMMA, PTFE, PE, PET, PP, PVC), which can be closed, to avoid prolonged contact with the air. If only non-volatile organic substances are of interest, stainless steel may be used. If both inorganic substances and non-volatile organic substances are of interest, glass equipment should be used.
- (2) The contact with the air is kept limited to avoid uptake of  $CO_2$  from the air in case of leaching from alkaline products. If necessary, the test may be carried out in a  $N_2$  atmosphere.  $2 \cdot 20 \cdot 14$
- (3) The minimum distance between the exposed surface(s) of the test portion(s) facing the walls and the walls of the vessel or tank shall be 20 mm, all around the test portion. Examples are given in Annex D.
- (4) Supports made of inert material shall be used to allow direct contact with water also on the bottom sides of the test piece. Supports shall not affect significantly the surface area of the sample exposed to the leachant. Sheet-like or plate-like products may be fixed on the inside of leaching vessel or tank with the exposed area facing into the vessel/tank (see Annex D).
- 7.3 Diamond blade cutting device and/or core drilling device (dry process) or other appropriate cutting tools
- **7.4** Analytical balance, with an accuracy of at least 0,1 g.
- **7.5 Device for measuring sample dimensions**, with an accuracy of at least 1 mm.
- 7.6 Measuring cylinders for volume determination, with 1 % accuracy.
- **7.7 Filtering device,** either a vacuum filtration device (between 30 kPa and 70 kPa) (300 mbar to 700 mbar) or a high-pressure filtration apparatus (< 0,5 MPa) (5 bars).
- 7.8 Membrane filters for filtration of the eluates