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BSI Standards Publication

Construction products — Assessment of release of dangerous substances

Part 3: Horizontal up-flow percolation test

National foreword

This Published Document is the UK implementation of CEN/TS 16637-3:2016.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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 Perkolationsprüfung 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.

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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of an identical text or by endorsement, at the latest by December 2016 and conflicting national standards shall be withdrawn at the latest by December 2016.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.

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.

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*

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/m^2) 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.

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]

3.14
test portion

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

[SOURCE: IUPAC 1990, 2.5.7]

3.15
test sample

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

[SOURCE: IUPAC 1990, 2.5.6]

4 Symbols and abbreviations

4.1 Symbols

For the purposes of this 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

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	polychlortrifluorehtylene
PTFE	polytetrafluorethylene
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

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) $c(HNO_3) = 0{,}1$ 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 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 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 ≥ 100 mm. Intermediate weights to be linearly interpolated by surface area.

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

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

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

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

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

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

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

7.10 Eluate bottles

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

7.11 Crushing equipment

Jaw crusher or a cutting device.

7.12 Sieving equipment (dry sieving)

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

7.13 Sample splitting equipment

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

7.14 Redox potential meter

(optional)

7.15 Turbidity meter

As specified in ISO 7027.

7.16 Centrifuge

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

(2) Alternatively, if a high speed centrifuge is not available, a centrifuge operating at 2 000 g to 2 500 g using bottles of an appropriate material may be used in combination with increased centrifugation time. Cooling shall be applied to maintain the desired temperature.

7.17 Filtration

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

8 Sample preparation

8.1 General

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

(2) Sampling shall be performed in accordance with the procedures described in the respective product Standard considering guidance given in CEN/TS 16637-1 in order to obtain a representative laboratory sample.

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

8.2 Preparation of the test sample

8.2.1 Principles

(1) A laboratory sample shall be received whose minimum amount depends on the maximum particle size of the granular product, the column diameter chosen and the number of tests to be carried out. The laboratory sample shall be stored in closed packages and kept under conditions which prevent any alterations of the construction product.

(2) The laboratory selects the appropriate column and determines the sieve diameter S_{CS} (7.12) for the test sample (see Table 1).

(3) Crushing of the product shall be applied no more than absolutely necessary in order to minimize the effect on the product properties, i.e. crushing is only needed in case of an oversized fraction larger than S_{CS} (see Table 1) or to fill a lack of fines less than 4 mm.

(4) For granular construction products at least 45 % by mass of the test sample shall have a particle size < 4 mm. If the grading of the laboratory sample already contains 100 % by mass particles < S_{CS} (see Table 1) and an amount of particles of at least 45 % by mass < 4 mm no further crushing or sieving is allowed.

(5) The choice of column width depends on the maximum particle size of the product to be tested (S_{CS}) and may also depend on equipment available in the laboratory, the mass of the laboratory sample or the amount of eluate required for chemical analysis or ecotoxicological testing.

(6) No part of the laboratory sample shall be discarded during the sample preparation.

8.2.2 Rules of application

(1) The following stepwise procedure to prepare the test sample shall be applied to fulfill the principles specified in 8.2.1:

a) If the laboratory sample of the product already contains 100 % by mass of particles < S_{CS} (see Table 1) and an amount of particles of least 45 % by mass < 4 mm no further crushing or sieving is allowed (see Annex A for an example).

b) If the laboratory sample contains an oversized fraction larger than S_{CS} gently crush this fraction to obtain a test sample with all particles ≤ S_{CS} (result: test sample with a maximum particle size to fit the chosen column size).

   Crusher jaws should be adjusted to avoid large amounts of particles < 4 mm.

c) Depending on the number and size of the test portions, the further procedure may be carried out on a representative subsample (see EN 933-1).

d) Check by sieving on 4 mm-sieve whether test sample contains an amount of particles < 4 mm larger than 45 % by mass. If this condition is fulfilled the test sample is ready to take test portions for the percolation test.

e) If the test sample does not contain an amount of at least 45 % by mass passing the 4 mm-sieve after the step d), gently crush a sufficient and representative portion of 4/ S_{CS} mm (see Annex A for an example) to an extent that after crushing the completely prepared (recombined) grading contains an amount of particles < 4 mm between 45 % by mass and 55 % by mass. If this condition is fulfilled, the test sample is ready to take test portions for the percolation test.

Table 1 — Column size and corresponding sieve diameter S_{cs}

Inner column diameter d [mm]	Sieve diameter for test sample preparation (crushing) to reduce the amount of oversized product to fit the inner column diameter S_{cs} [mm]
50 - 100	16
70 - 100	22,4
$d \geq 100$ and $d \geq 3D_{max}$	D_{max}[a]

[a] If the sample contains at least 45 % by mass particles < 4 mm, no crushing is allowed. If this condition does not apply, set S_{cs} to 22,4 mm.

(2) The particle size distribution of the original granular product should be reported, if available. Regarding the test sample, the maximum particle size and the percentage of particles < 4 mm shall be reported.

8.3 Test portion

(1) At least two test portions shall be taken from the test sample (one for determination of the dry residue, and one for the percolation test itself). The test portions shall be subsampled in line with 8.5.

It is recommended to reserve a test portion to be able to repeat the test in case of failure. It is often considered good practice to perform the test in duplicate or triplicate (which would require additional test portions).

(2) The amount of the test portion for the column is depending on the diameter of the column, the packing height, the density of the granular product and the porosity of the packing.

8.4 Drying

If the sample cannot be sieved or crushed, due to its moisture content, it may be dried, but no further than is required for the preparation of the test sample. Freeze drying or drying at room temperature is preferred prior oven drying. The drying temperature shall not exceed 40 °C.

NOTE Drying may alter the properties of the sample (oxidation and/or carbonation) or result in loss of volatile substances. Drying conducted in an inert atmosphere may prevent alteration of the sample properties. Guidance on drying of samples is given in EN 15002 and EN 16179.

8.5 Subsampling

A sample splitter (7.13) shall be used or coning and quartering shall be applied to divide the sample and prepare subsamples.

NOTE Guidance on subsampling is given in EN 15002 and EN 16179.

8.6 Sieving of samples into fractions

Sieving of samples into particle fractions shall be done by dry sieving only, according to EN 933-1.

8.7 Size reduction of particles

If oversized particles are to be size reduced, the entire oversized fraction shall be crushed with a crushing device (7.11) to grains smaller than the maximum particle size (S_{CS}) required for the test portion. Crushing shall proceed no further than is required for the sample preparation. Heating, loss of dust or contamination from the equipment shall be minimized. On no account shall the material be finely ground. Non–crushable material (e.g. metallic particles) shall be separated, weighed and the nature of the material recorded.

NOTE Plastic or bituminous products may be size-reduced after cryogenic treatment. Guidance on particle size reduction is given in EN 15002 and EN 16179.

9 Test procedure

9.1 Temperature

(1) The percolation test shall be carried out at a temperature between 19 °C and 25 °C.

NOTE A constant temperature in the test can be achieved by either controlling the temperature of the lab, or controlling the temperature of the leachant and insulating the column and accompanying equipment.

(2) For products releasing substances that are very sensitive to biological degradation, performance of the test for these substances at reduced temperature (e.g. 4 °C) and preventing direct exposure to light will limit biological activity significantly. If the test is modified in this way, this deviation shall be reported in the test report.

9.2 Determination of dry residue

(1) The dry residue of the test sample shall be known and taken into account when calculating the L/S-ratio.

(2) Determine the dry residue (w_{dr}) at 105 °C ± 3 °C according to EN 14346, on a separate test portion. Calculate the dry residue in accordance with Formula (1).

$$w_{\mathrm{dr}} = m_{\mathrm{d}} \times 100 / m_{\mathrm{r}} \ [\%] \qquad (1)$$

where

w_{dr}	dry residue of the construction product expressed as percentage (%);
m_{d}	dry mass of the test portion expressed in grams (g);
m_{r}	mass of the undried test portion expressed in grams (g).

9.3 Pre-treatment of the column

Rinse the column with top and bottom section and filters (7.2) and bottles (7.10) with diluted nitric acid (0,1 mol/l) and/or an organic solvent and water respectively. In case of organics acetone should be used. Weigh the dry column, including top and bottom sections filters and filter plates or layers of sand, to an accuracy of 1 g. Alternatively, a heat treatment (e.g. between 450 °C and 500 °C) can be applied to clean temperature resistant equipment of sorbed organic contaminants. Quartz sand and filter layers shall not be heated to red-heat.

9.4 Packing of the column

The column shall be packed as evenly and uniformly as possible, avoiding segregation of different particle sizes. This is generally best accomplished by packing the granular products in the column in stages. Annex C provides advice on the packing of columns.

9.5 Start of the test

9.5.1 Equilibration

(1) Saturate by hydrostatic pressure (preferably no more than 6 h) or by pumping (preferably 4 h ± 2 h).

NOTE See Annex C.4 for a description of the methods of saturation.

(2) Stop the pump, or take away the hydrostatic pressure, when the material in the column is completely saturated, but the outlet hose remains empty. Leave the saturated material for a period from 12 h to 72 h, in order to equilibrate the system.

9.5.2 Calculation of the flow rate

(1) After the equilibration period, start the pump (again) and set the flow rate ϕ through the empty column such that the linear velocity (V_L) is (300 ± 40) mm/d.

(2) Calculate the flow rate in accordance with Formula (2):

$$\phi = V_L \times \pi \times d^2 \times 0{,}000\,010\,4 \qquad (2)$$

where

ϕ leachant flow rate, expressed in millilitres per hour (ml/h);

V_L linear velocity of the leachant through the empty column, expressed in millimetres per day (mm/d);

d inner diameter of the column, expressed in millimetres (mm).

NOTE 1 A linear velocity of 300 mm/d corresponds to a flow rate of 24,5 ml/h for a column with a diameter of 50 mm and for a column with a diameter of 100 mm it is equivalent to a flow rate of 98 ml/h.

NOTE 2 The parameter 0,000 010 4 includes the factor 1/4 from the calculation of column volume times 1/1 000 to convert from mm^3 to ml and times the factor 1/24 to convert from days to hours. Using this factor, the calculated flow rate is expressed in a common unit (ml/h).

9.6 Collection of eluates

(1) Connect the outlet hose (7.9) to an eluate collection bottle of appropriate size (7.10). Start the pump again and change the collection bottle after a quantity of (0,1 ± 0,02) times the mass of the test portion (m_d) of leachant has been collected (fraction 1 according to Table 2).

Measure the pH (7.7) and the electrical conductivity (7.8) in accordance with EN 16192 and redox potential (7.14, optional) of the eluate. If the pH value of the eluate is higher than 9, or if a later rise in pH is expected, it may be necessary to keep the eluate collection bottle and the following ones under inert (argon or nitrogen) atmosphere or another valid technique to prevent carbonation and precipitation. If the conductivity of an eluate fraction exceeds 7 500 mS/m, measure the volume (or density) of the eluate and dilute the eluate until the conductivity is below 7 500 mS/m. Ensure that the dilution factor is no more than 10. Refill gas every time the collection bottle is changed. This procedure is repeated for each eluate fraction.

NOTE 1 An air lock may be needed on the lid to avoid back-pressure in the column in case the bottle with lid is gas tight.

NOTE 2 Keeping the collection bottles under inert atmosphere may also be required in case of investigating products with reducing properties to prevent the occurrence of oxidation reactions.

NOTE 3 The pH, EC, DOC (optional) and redox measurements can be used for verification purposes in case measurements deviate from the expected release behaviour. This will provide the user with option to accept the results, verify deviations from normal or take corrective measures based on an understanding of the cause of the deviation.

(2) For organic substances proceed as follows:

a) If the turbidity of the eluate is below 100 FNU the eluate shall be analysed as it is. Otherwise, transfer the eluate to centrifuge tubes (7.16).

b) There are two options for solid-liquid separation:

   1) centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high speed centrifuge (7.16);

   2) centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower speed centrifuge (7.16).

NOTE 4 Limitation of maximum centrifugation duration is necessary especially for organic substances. The speed or *g*-force of the centrifuge is dependent on the specific rotor.

NOTE 5 Background information on sorption effects on container walls is given in “Sorption effects interfering with the analysis of polycyclic aromatic hydrocarbons (PAH) in aqueous samples”, see bibliography.

(3) If necessary, cooling shall be applied to maintain the temperature between 19 °C and 25 °C.

(4) Gentle braking shall be applied in order to avoid re-suspension. Deceleration time shall not exceed 20 min.

(5) If only leaching of inorganic substances and DOC is of interest, the eluate does not have to be centrifuged as described above but has to be filtered using the 0,45 µm membrane filters (7.4) and a vacuum or pressure filtration device (7.17).

(6) Check the flow rate of the leachant as often as needed (at least after collection of each eluate or three times per week whichever is most frequent), and adjust to the original linear velocity (300 ± 40) mm/d.

(7) Change the collection bottles with a new one as soon as a quantity of water (6.1) according to Table 2 has passed through. These are fractions 2 to 7. Mind that both criteria (concerning the volume of the actual eluate fraction as well as the cumulative L/S-ratio) shall be fulfilled.

(8) At each eluate collection point, measure time and volume of the eluate fraction and calculate the L/S-ratio and the average flow velocity of the leachant over the collection period of that fraction. Also, measure the actual flow velocity.

Table 2 — Table for collection of eluate fractions

Fraction number	**Fraction volume** [a, b] l	**Cumulative L/S ratio** l/kg
1	(0,1 ± 0,02) × m_d	0,1
2	(0,1 ± 0,02) × m_d	0,2
3	(0,3 ± 0,05) × m_d	0,5
4	(0,5 ± 0,05) × m_d	1,0
5	(1,0 ± 0,05) × m_d	2,0
6	(3,0 ± 0,1) × m_d	5,0
7	(5,0 ± 0,2) × m_d	10,0

[a] Numbers in parentheses are L/S of the fractions in l/kg.

[b] Because the density of the eluates from construction products is close to 1 g/ml, mass can be used instead of volume.

NOTE 6 An automatic eluate collection system can be used.

(9) For products with low density the volume collected in each of the first two fractions may not be sufficient for chemical analysis. In such cases, the first two volumes may be combined to one fraction prior to chemical analysis.

(10) The test itself is finished when the cumulative L/S-ratio of (10 ± 0,5) l/kg dry matter is reached.

(11) For specific scenarios it may be sufficient to know the leaching characteristics up to a pre-determined L/S ratio, for instance L/S = 2 or the cumulative release up to L/S = 2. In that case the test can be stopped after the collection of the fifth eluate fraction or the collection of one eluate up to L/S = 2. In other cases, e.g. for products functioning as drains, scenario descriptions may require much higher L/S values. If that is the case, additional fractions may be collected until the relevant L/S-range has been covered. This is an example of an indirect procedure, see Clause 13 for explanation.

NOTE 7 For ecotoxicological testing, the fractions 1 to 5 (or parts of these fractions) can be combined to receive a cumulative L/S-ratio of 2. If large amounts of eluates are needed for testing, eluates of the same fractions of several percolation tests can be combined.

NOTE 8 In case no automated eluate collection apparatus is available, a collection scheme can be composed within the ranges that are allowed in flow rate (e.g. (300 ± 40) mm/d) and in fraction volume (see Table 2) that enables eluate collection within working hours.

NOTE 9 The execution time of the test can be calculated from Formula (3):

$$t = (L/S \times m_\mathrm{d} \times 1000)/(24 \times \phi) \tag{3}$$

where

t is the execution time of the test expressed in days (d);

L/S final liquid to solid ratio expressed in litres per kilogram dry matter (l/kg dry matter);

m_d dry mass of the test portion expressed in kilogram (kg);

ϕ leachant flow rate expressed in millilitres per hour (ml/h).

9.7 Further preparation of the eluates for analysis

(1) Determine the concentrations of substances of interest using appropriate methods of eluate analysis for inorganic or organic substances. In case of known or expected presence of dissolved organic carbon (DOC) measure the total concentration of DOC.

NOTE 1 In CEN/TC 351/WG5 procedures for eluate analysis will be specified and validated. An overview of suitable analytical standards is given in CEN/TR 16045. Until these are ready, informative reference can be made to e.g. EN 16170, EN 16171, EN 16192.

(2) If a small column (with a diameter of 50 mm) is used, it may be desirable to dilute the first eluate fractions to get enough eluate to carry out all analyses. The dilution factor for this purpose should be no more than 4. The dilution factor should be reported. A dilution should not be applied if very low concentrations are expected.

(3) Divide each eluate into an appropriate number of sub-samples for different chemical analysis (e.g. for non-volatile organic substances, elements by ICP analysis, salts by liquid chromatography, etc.), after shaking well.

(4) Preserve the eluate sub-samples depending on the elements to be analysed, according to EN ISO 5667-3. Store the (sub) eluates in sealed bottles, according to EN ISO 5667-3.

NOTE 2 DOC may be of relevance when a product containing organic matter is stabilized, e.g. with a hydraulic binder. DOC is mobilised at high pH and is capable of mobilizing metals and organic substances well beyond its solubility limitation under the common alkaline conditions.

9.8 Blank test

(1) In order to check, as far as possible, how the entire procedure is performed, carry out blank tests on a regular basis. Submit two empty column volumes of leachant to the whole procedure (except the sample pre-treatment). Start the pump, until the empty column, complete with top and bottom sections and with tubing, is filled with leachant. After two days disconnect the pump and the column, empty the column via the bottom section and collect the first blank eluate. Connect the pump again, and fill the empty column and fittings with new leachant. After two other days empty the column in the same way and collect the second blank eluate. Measure the electrical conductivity (see 7.8) of these blank eluates. Preserve and analyse both blank eluates in accordance with 9.6.

(2) Ensure that the eluates of this blank test fulfil the following minimum requirements:

a) in the first eluate of the blank test the concentration of each considered substance shall be less than 10 % of the calculated average concentration in the first three eluate fractions of the product test, or below the detection limit (limit of quantification);

b) the electrical conductivity of the second blank eluate shall be < 0,5 mS/m.

(3) If these requirements are not fulfilled, reduce the contamination.

10 Evaluation of measurement results

10.1 Expression of results in concentrations

The analysis of the eluates produced by the leaching test provides the value of the concentration c_i of the substances in the eluates of the individual eluate fractions-expressed in mg/l (or µg/l). The measured value determined in accordance with 9.6 shall be corrected if the eluate fraction has been diluted and/or if the quantity of preservation fluid added in 9.6 was more than 1 ml per 100 ml of eluate.

10.2 Expression of results in terms of mass related release

(1) For each substance the quantities released in all eluate fractions shall be calculated as in Formula (4) as mass related release.

$$E_i = (V_i \times c_i)/(m_d) \tag{4}$$

where

i is the index of the eluate fraction (1, 2, ...,7);

E_i released quantity of a substance in eluate fraction *i* per quantity of test portion (mass related release), expressed in milligrams per kilogram dry matter (mg/kg dry matter);

V_i volume of the eluate fraction, *i*, expressed in litres (l);

c_i concentration of the substance concerned in the eluate fraction *I*, expressed in milligrams per litre (mg/l);

m_d dry mass of the test portion in the column, expressed in kilograms (kg).

(2) Where the concentration of a substance in one or more eluate fractions is below the limit of quantification, a calculation shall be carried out for this substance in these fractions by making c_i = limit of quantification, thereby estimating the upper limit of E_i.

(3) For each substance, the measured cumulative release shall be calculated as follows:

$$U_n = \sum_{i=1}^{n} E_i \quad \text{for n = 1 to 7} \tag{5}$$

where

U_n is the measured cumulative release (mass) 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);

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).

(4) Where the concentration of a substance in one or more eluate fractions is below the quantification limit, for this substance a calculation shall be carried out using the value of E_i corresponding to the quantification limit to indicate the upper limit of U_n. As an option, the lower limit of U_n may be calculated using $E_\mathrm{i} = 0$ for measurements below the quantification limit.

11 Documentation and test report

The report shall contain at least the following data:

a) General:

  1) reference to the present Technical Specification;

  2) date of receipt of the laboratory sample;

  3) identification of the laboratory sample, including sample code;

  4) ageing conditions (if relevant);

   5) sample preparation (e.g. method of size-reduction, drying, sub-division) and storage conditions of the laboratory sample and test sample;
   6) crushing equipment;
   7) maximum particle size and percentage of particle < 4 mm of the laboratory sample and test sample;
   8) drying temperature (°C);
   9) dry residue of the test sample (w_{dr});
   10) dry mass of the test portion in the column (m_{d});
   11) compaction method;
   12) sampling report, if available.

b) Leaching test conditions:
   1) date of the test (beginning and end);
   2) identification of the test equipment and instruments used, including the diameter of the column;
   3) actual flow rate during the course of the test, and any significant changes in flow rate, deviations from the prescribed flow rate shall be commented on;
   4) true cumulative L/S-ratios of the eluate fractions collected;
   5) temperature range during the performance of the test;
   6) any deviation from the test method and the reason of this deviation together with all circumstances that have influenced the results.

c) Analytical report:
   1) pH and conductivity and redox potential (optional) of the eluate fractions collected;
   2) any dilutions that were carried out;
   3) methods of preservation of the eluate fractions for the different elements to be analysed;
   4) quantities of preservation fluid added where this is more than 1 ml per 100 ml eluate;
   5) analytical method applied;
   6) all measured concentrations.

d) Results of the leaching test:
   1) the measured concentration c_i per fraction i for each substance, in mg/l, in a table and preferably plotted against the true L/S-ratio;
   2) the cumulatively released quantities U_{n} for fraction n for n = 1 to 7, calculated for each substance, in mg/kg dry mass, in a table and preferably plotted against the true L/S-ratio. If relevant, lower and upper limits shall be indicated.

NOTE 1 In many cases it may be useful to be able to determine the mechanism controlling the release of substances from the granular construction product in the percolation test. This is discussed in Annex D (informative).

NOTE 2 In some cases it may be useful or necessary to be able to interpolate or extrapolate results obtained by the percolation test.

12 Test performance

When the Technical Specification was adopted by CEN, the procedures specified in this document were not validated and no data were available on repeatability and reproducibility. The robustness of the method was shown in CEN/TS 14405.

13 Indirect methods

13.1 Definition

This Technical Specification specifies the "horizontal" reference methods in Clauses 1 to 12. The term "indirect" method is variously applied to mean simplified method, secondary method, derived method or alternative method.

NOTE 1 Such "indirect" methods may be easier and/or cheaper to apply for a specific application. An 'indirect' method is generally not "horizontal", but dedicated to a specific product or range of products (as specified in a hEN or in ETAs).

NOTE 2 The term "indirect" has been selected to underline that, instead of a direct determination by the horizontal reference method, an "indirect" method provides a result indirectly through the mandatory comparability, correlation or practical relationship to the reference method (in accordance with the purpose of the release determination).

13.2 Provisions

(1) As indicated in the introduction, one of the aims of this Technical Specification is — according to the horizontal concept specified in Mandate M/366 — that construction products are evaluated under comparable conditions with regard to release to soil and water according to the horizontal concept specified in Mandate M/366 (see point IV-10).

(2) When applied to a specified product or range of products an "indirect" method shall provide a result that is comparable to or correlates with that produced by the reference method.

(3) The requirements, especially the sampling requirements, specified for the reference method apply also for the "indirect" method unless this "indirect" method specifies different requirements adapted to its specific field of application.

(4) The specific and limited field of application of an "indirect" method may include requirements on raw materials, product formulation and operating parameters. In any event, the validity of the above correlation, comparability remains strictly in the field of application for which it has been established.

13.3 Examples of "indirect" methods

Examples of possible "indirect" method are:

EXAMPLE Batch tests according to EN 12457 (all parts).

Annex A
(informative)

Examples for sample preparation procedure

A.1 General

This Annex provides two typical product grading examples with a description of the different steps whether crushing is needed and the right choice of the inner column diameter for the percolation test.

A.2 Example 1

Product grading according to EN 13285: 0/8 – UF_9 – LF_N – OC_{85} – G_E

Sieve size	**Percentage passing sieve (% by mass)**	
mm	**Minimum**	**Maximum**
11,2	100	
8	85	100
5,6		
4	50	90
2	30	75
1		
0,500	0	35
0,063	0	9

Product grading contains at least 45 % by mass particles ≤ 4 mm:

Conclusion: no further sample preparation required.

Choose a column with an inner diameter $d \geq 50$ mm.

A.3 Example 2

Product grading according to EN 13285: 0/31,5 – UF_7 – LF_N – OC_{75} – G_C

Sieve size **mm**	**Percentage passing sieve (% by mass)**		
	Minimum	**Maximum**	**Additional information from the producer**
63	100		100
45			100
31,5	75	100	84 – 97
22,4			69 – 86
16	50	90	56 – 74
11,2			43 – 63
8	30	75	35 – 53
5,6			30 – 46
4	20	60	26 – 40
2	14	45	22 – 34
1	8	35	19 – 29
0,500	5	25	16 – 23
0,063	0	7	1 – 4

a) Example 2: A Sieve diameter 22,4 mm according to 8.2.2, Table 1 is chosen:

From table: the residue on 22,4 mm-sieve is between 14 % by mass and 31 % by mass;

1st step: amount of (14 to 31) % by mass needs to be gently crushed (all particles ≤ 22,4 mm);

2nd step: check whether the test sample contains an amount of particles of at least 45 % by mass passing the 4-mm sieve;

a) if yes: test sample is ready to take test portions for the percolation test;

b) if no: gently crush a sufficient and representative portion of 4/22,4 mm to an extent that after crushing the completely prepared (recombined) grading contains an amount of particles < 4 mm between 45 % by mass and 55 % by mass. If this condition is fulfilled the test sample is ready to take test portions for the percolation test;

3rd step: choose a column with an inner diameter *d* between 70 mm and 100 mm.

b) Example 2: A Sieve diameter 16 mm according to 8.2.2, Table 1 is chosen:

From table: the residue on 16 mm-sieve is between 26 % by mass and 44 % by mass;

1st step: amount of (26 to 44) % by mass needs to be gently crushed (all particles ≤ 16 mm);

2nd step: check by sieving on a 4 mm-sieve whether the test sample contains an amount of particles < 4 mm between 45 % by mass and 55 % by mass;

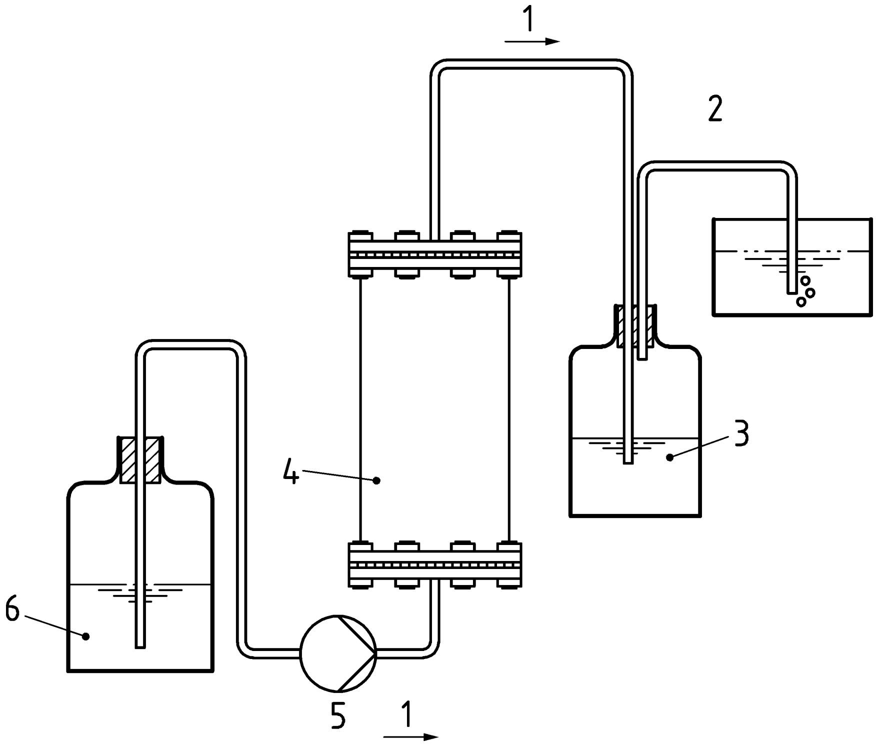
a) if yes (will be most probably the case): test sample is ready to take test portions for the percolation test;

b) if no: gently crush a sufficient and representative portion of 4/16 mm to an extent that after crushing the completely prepared (recombined) grading contains an amount of particles < 4 mm between 45 % by mass and 55 % by mass. If this condition is fulfilled the test sample is ready to take test portions for the percolation test.

3rd step: choose a column with an inner diameter *d* between 50 mm and 100 mm.

Annex B
(informative)

Illustration of the column and accompanying equipment



Key

1	flow direction
2	air lock (optional)
3	eluate collection
4	column filled with construction product
5	pump
6	leachant

Figure B.1 — Example of a column and accompanying equipment

Annex C
(normative)

Procedures for packing and saturation of the column

C.1 General

This annex contains procedures on how to fill and pack the column. It also gives a more detailed description of the method to conduct water saturation.

C.2 Column filling and packing

Procedures for filling the column and packing the product are:

a) Granular (non-powdery) products can generally be packed as they are (moist or dry). However, powdery, dry materials should be humidified either at an arbitrary and imposed ratio. The actual moisture content shall be known, in order to be able to determine the dry mass of the test portion in the column (in 8.3).

b) Products may be too wet to pack well in the column. It is usually possible to air-dry the test sample or the test portion. The drying temperature should not exceed 40 °C. Drying may lead to oxidation and/or carbonation. If the material is fresh and has to be tested as a non-oxidized / non-carbonated material, the drying should be conducted in an inert atmosphere.

c) In other cases, it may be possible to pack the (wet) product, but the product may settle even more after the start of the test, causing the formation of headspace. If a system with a piston is used, the piston should be lowered accordingly.

d) Some products can cause problems later, even if packed well. These are materials with hydraulic binding properties. Hardening reactions may lead to expansion, leading to a very low permeability, or even to cracking of the column. This means that the product is likely to behave as a monolith and should be tested with the appropriate method (e.g. CEN/TS 16637-2:2014). However, if it is the intention to study the behaviour of the material in percolating conditions, a system with a piston that can be moved upward may be used. Another solution may be, in some cases, to compact less. Both solutions, however, lead to an inferior repeatability.

C.3 Procedure for packing

(1) Fit the bottom section, equipped with a filter plate, glass beads or a thin layer of quartz sand with a grain size range of 1,0 mm to 2,5 mm (7.2) to the column. Fill the column with the test portion, up to a bed height of 300 mm ± 50 mm in five consecutive layers.

a) Introduce each layer into the column in three sub-layers and level each sub-layer separately.

b) Pack each layer using as a rammer a weight of 125 g in case of a column with a diameter of 50 mm, and of 500 g in case of a column with a diameter of 100 mm. Drop the weight three times on each layer falling down 200 mm along a rod used as a guide. Fix this rod to the centre of a disk, which is placed on the layer to be packed. Cover the whole surface of the column with the disk.

c) For the last layer, check the remaining height and adjust the necessary mass in order to get 300 mm ± 50 mm.

In order to determine the proper mass for each layer, a preliminary test may be carried out. In that case, put a 70 mm to 80 mm layer in the column, pack it and calculate the mass necessary to get a layer of approximately 60 mm.

(2) Top section of the column: Place upon the material bed a filter plate, glass beads or a thin layer of quartz sand with a grain size range of 1,0 mm to 2,5 mm to prevent entrainment of fine particles with the eluate.

(3) The top section and pre-filter or the sand shall be fitted such that the liquid flow cannot bypass the filter plate, or glass beads or the sand layer and such that no open space (dead volume) is left above the material.

(4) Weigh the column thus filled to an accuracy of 1 g. Determine the dry mass (m_0) of the test portion in the column, in accordance with Formula (2):

$$m_\mathrm{d} = m_\mathrm{W} \cdot w_\mathrm{dr} / 100 \tag{C.1}$$

where

w_dr is the dry matter content, expressed in percentage (%);

m_d is the mass of the dried test portion in the column, expressed in kilograms (kg);

m_W is the mass of the (moist) test portion in the column, expressed in kilograms (kg);

(5) Fit the outlet-hose to the top section of the column.

C.4 Water saturation

(1) In 9.5.1, two methods are mentioned to saturate the packed column with leachant: using the pump (1) or using initial hydrostatic pressure (2).

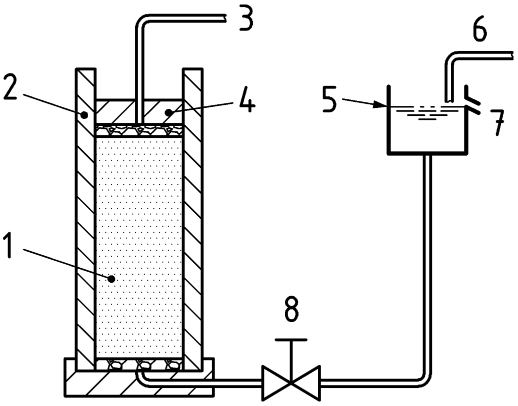
1: Saturating the column using the pump

(2) Connect the pump (7.5) to the bottom section of the column and pass demineralized water (6.1) through the column from bottom to top. Saturate by pumping (preferably using no more than 6 h). Stop the pump when the material in the column is completely saturated, but the outlet hose is still empty.

2: Saturating the column using initial hydrostatic pressure

(3) To avoid the need to watch over the column in order to prevent overflowing, water saturation can also be done under an initial hydrostatic pressure of 300 mm (as is shown in Figure C.1).

(4) The hydrostatic pressure should be stopped when the material in the column is completely saturated, but the outlet hose is still empty.



Key

1 layer of compacted construction product
2 column
3 water outflow
4 layer of inert material
5 water level
6 water inlet
7 water outflow
8 water inlet

Figure C.1 — Typical column for saturation by initial hydrostatic pressure

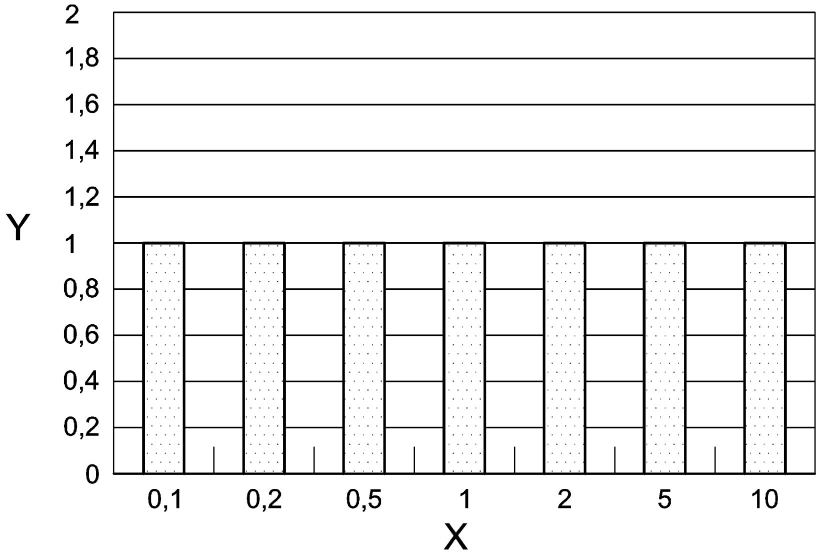
Annex D
(informative)

Assessment of release mechanism(s)

D.1 Overview of release mechanisms

(1) Prediction of the long term release of dangerous substances is important with respect to the evaluation of the release behaviour of a construction product during its service life in a construction. In order to be able to predict the long term release by extrapolation the release mechanism has to be known. The release mechanism of a substance may be derived from the test results.

(2) Basically the percolation test is designed to satisfy (local) equilibrium during the course of the test and release is controlled by solubility. Under this condition, the concentration of a substance will be (more or less) constant during the test for each L/S-ratio see Figure D.1.



Key

X cumulative L/S ratio in l/kg

Y concentration in µg/l

Figure D.1 — Basic release pattern percolation test

(3) Assuming that (local) equilibrium is maintained during the entire test (up to L/S 10) any deviation from this pattern indicates a different release mechanism than solubility controlled release due to:

a) wash-out of the substance (depletion), or

b) release controlled by diffusion rather than solubility (apparent depletion).

(4) The product's release mechanism information should be incorporated in the reported result. This is required:

a) to characterize the release performance with respect to type testing;

b) to establish conformity with a product class assignment (CE-marking) and environmental requirements;

c) to link test results from a standard test duration (normally L/S 10) to shortened FPC tests and to historic data from existing comparable test procedures with other L/S-ratios.

NOTE It may be noted that this annex only concerns the release performance of the products. It does not cover the modelling of an environmental evaluation in a source-path-target evaluation. It will only provide indispensable information which can be used as a basis for further environmental evaluation.

The procedure in this annex should be carried out for each of the substances to be tested.

D.2 Overview of release mechanisms

D.2.1 Overall low concentrations

(1) If the average concentration of a substance has a value close to the detection limit it is not possible to determine a release mechanism properly. Overall low concentrations for a substance are established if:

$$\frac{c_{2\text{-}7}}{\text{detection limit } [\mu\text{g/l}]} < 1{,}5 \qquad \text{(D.1)}$$

where

$c_{2-7} = \frac{\sum_{i=2}^{7} c_\mathrm{i}}{6}$ is the average concentration of the substance in eluate 2 to 7, expressed in micrograms per litre (µg/l);

c_i is the concentration of the substance in eluate *i*, expressed in micrograms per litre (µg/l).

(2) If for a specific substance concentrations are overall low subclauses D.2.2, D.2.3, D.2.4 and D.2.5 should be skipped for this substance.

D.2.2 Effect of pH on release

(1) If the check on overall low concentrations for a substance is negative, it is checked if the release of the substance is pH dependent by plotting concentrations versus pH. A more or less horizontal line or a cloud implies no pH dependency over the pH range of the test: If the slope of the linear regression line is significantly greater or smaller than zero, then release may be pH dependent.

(2) Some substances are not sensitive for pH changes of the eluate during the test; e.g. bromide and chloride.

(3) If the release is pH dependent go to D.2.3 and if the release is not pH dependent go to D.2.4.

D.2.3 (Apparent) pH dependent release

D.2.3.1 Wash-out of a substance (depletion)

(1) If the check on overall low concentrations is negative for the substance it is checked whether washout of the substance occurs. When a substance is fully dissolved in the matrix, it washes out readily. Mostly within L/S = 1 to 2 l/kg.

NOTE In the specific case that the pH of the first eluates differs significantly from the pH of the later eluates it is not possible to distinguish wash-out from pH-dependent dissolution. In this case an (apparent) relation between pH and concentration may occur, and the observed release could be caused either by wash-out or dissolution.

(2) Wash-out (depletion) is established if:

$$\frac{c_{1-3}}{c_{5-7}} > 2{,}0 \tag{D.2}$$

and

$$\frac{c_{6-7}}{\text{detection limit } [\mu\text{g/l}]} < 1{,}5 \tag{D.3}$$

and

$$\text{pH}_{4-7} - 2\sigma_{\text{pH}_{4-7}} < \text{pH}_{1-2} < \text{pH}_{4-7} + 2\sigma_{\text{pH}_{4-7}} . \tag{D.4}$$

where

$c_{1-3} = \frac{\sum_{i=1}^{3} c_\text{i}}{3}$	is the average concentration of the substance in eluate 1 to 3, expressed in micrograms per litre (µg/l);
$c_{5-7} = \frac{\sum_{i=5}^{7} c_\text{i}}{3}$	is the average concentration of the substance in eluate 5 to 7, expressed in micrograms per litre (µg/l);
$c_{6-7} = \frac{\sum_{i=6}^{7} c_\text{i}}{2}$	is the average concentration of the substance in eluate 6 to 7, expressed in micrograms per litre (µg/l);
c_i	is the concentration of the substance in eluate *i*, expressed in micrograms per litre (µg/l);
$pH_{1-2} = \frac{\sum_{i=1}^{2} pH_\text{i}}{2}$	is the average pH of eluates 1 and 2;
$pH_{4-7} = \frac{\sum_{i=4}^{7} pH_\text{i}}{4}$	= average pH of eluates 4 to 7;
$\sigma_{pH_{4-7}} = \sqrt{\frac{\sum_{i=4}^{7} \left(pH_\text{i} - pH_{4-7}\right)^2}{4}}$	is the standard deviation of the pH of eluates 4 to 7;
pH_i	is the pH of eluate *i*.

(3) If conditions D.2 and D.3 are met, but condition D.4 is not, it is not possible to establish the release mechanism as the release could be pH dependent solubility controlled or washout. The release mechanism of that substance remains unidentified and D.2.3.2, D.2.4 and D.2.5 should be skipped for this substance.

D.2.3.2 Solubility controlled release

(1) For most substances solubility controlled release is pH dependent and the concentration is a function of pH. In this case, a defined concentration will be in equilibrium with the material as the pH changes. A significant pH change with L/S-ratio will then imply a pH dependent concentration. Therefore, in all other cases of (apparent) pH dependent release pH dependent solubility controlled release is established and D.2.4 and D.2.5 should be skipped for this substance.

D.2.4 No pH dependent release

D.2.4.1 Solubility controlled release

(1) If the check on overall low concentrations for a substance is negative, it is checked if the release of the substance is controlled by solubility. Solubility controlled release occurs if:

$$\frac{\sigma_{\text{c}}}{c_{1-7}} < 0{,}25. \qquad \text{(D.5)}$$

where

$\sigma_{\text{c}} = \sqrt{\dfrac{\sum_{i=1}^{7}\left(c_{\text{i}} - c_{1-7}\right)^2}{7}}$	is the standard deviation of the concentrations of the substance in eluates 1 to 7;
$c_{1-7} = \dfrac{\sum_{i=1}^{7} c_{\text{i}}}{7}$	is the average concentration of the substance in eluate 1 to 7, expressed in micrograms per litre (µg/l)
c_{i}	is the concentration of the substance in eluate *i*, expressed in micrograms per litre (µg/l).

(2) If for a specific substance solubility controlled release is established D.2.4.2, D.2.4.3 and D.2.5 should be skipped for this substance.

D.2.4.2 Wash-out of a substance (depletion)

(1) If the check on overall low concentrations and solubility controlled release is negative for the substance it is checked whether washout of the substance occurs. When a substance is fully dissolved in the matrix, it washes out readily. Mostly within L/S = 1 to 2 l/kg. Wash-out (depletion) is established if:

$$\frac{c_{1-3}}{c_{5-7}} > 2{,}0 \qquad \text{(D.6)}$$

and

$$\frac{c_{6-7}}{\text{detection limit } [\mu\text{g/l}]} < 1{,}5 \qquad \text{(D.7)}$$

where

$c_{1-3} = \frac{\sum_{i=1}^{3} c_i}{3}$ is the average concentration of the substance in eluate 1 to 3, expressed in micrograms per litre (µg/l);

$c_{5-7} = \frac{\sum_{i=5}^{7} c_i}{3}$ is the average concentration of the substance in eluate 5 to 7, expressed in micrograms per litre (µg/l);

$c_{6-7} = \frac{\sum_{i=6}^{7} c_i}{2}$ is the average concentration of the substance in eluate 6 to 7, expressed in micrograms per litre (µg/l);

c_i is the concentration of the substance in eluate *i*, expressed in micrograms per litre (µg/l).

(2) If for a specific substance wash-out is established D.2.4.3 and D.2.5 should be skipped for this substance.

D.2.4.3 Apparent depletion

(1) If the check on overall low concentrations, solubility control and wash-out is negative for the substance it is checked whether apparent depletion due to a change in the release mechanism from mainly solubility controlled release to mainly diffusion controlled release occurs. Apparent depletion is established if:

$$\frac{c_{1-4}}{c_{6-7}} > 1.5 \qquad \text{(D.8)}$$

and

$$\frac{c_{6-7}}{\text{detection limit } [\mu\text{g/l}]} > 1.5 \qquad \text{(D.9)}$$

where

$c_{1-4} = \frac{\sum_{i=1}^{4} c_i}{4}$ is the average concentration of the substance in eluate 1 to 4, expressed in micrograms per litre (µg/l);

$c_{6-7} = \frac{\sum_{i=6}^{7} c_i}{2}$ is the average concentration of the substance in eluate 6 to 7, expressed in micrograms per litre (µg/l);

c_i is the concentration of the substance in eluate *i*, expressed in micrograms per litre (µg/l).

(2) If for a specific substance apparent depletion is established D.2.5 should be skipped for this substance.

D.2.5 Unidentified release mechanism

If none of the previously mentioned mechanisms can be identified as the main release mechanism the release mechanism of that substance remains unidentified.

D.3 Overview of release mechanisms

D.3.1 Release

(1) See Clause 9 for the calculation of the release E_i and cumulative release U_n for each period:

$$U_\mathrm{n} = \sum_{i=1}^{n} E_\mathrm{i} \quad \text{for } n = 1 \text{ to } 7 \tag{D.10}$$

where

U_n is the cumulative release of a substance for a cumulative L/S-ratio *n* including fraction *i* = 1 to *n*, expressed in milligrams per kilogram dry matter (mg/kg dry matter);

E_i is the 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);

L/S_n is the cumulative L/S-ratio *n* including fraction *i* = 1 to *n*, expressed in litres per kilogram (l/kg).

D.3.2 Extrapolation and interpolation of the release to other L/S-ratios

(1) For some situations, interpolation or extrapolation of the reported release is required. In principle for all situations interpolation and extrapolation are possible. Release over time patterns differ per substance and material but often the release pattern can more or less be approached by a generic formula. Interpolation and extrapolation might be based on the following equation:

$$U_{\mathrm{L/Sx}} = U_{\mathrm{L/Sy}} * \left(1 - e^{-k(L/Sx)}\right) / \left(1 - e^{-k(L/Sy)}\right) \tag{D.11}$$

where

k is the constant that represents the speed of the release of a certain substance (release increases when k decreases), see e.g. publication of Aalbers et al. in the bibliography.

L/Sx is the target L/S-ratio;

L/Sy is the reference L/S-ratio;

$U_{\mathrm{L/Sx}}$ is the release at target L/S, expressed in milligrams per kilogram dry matter (mg/kg dry matter);

$U_{\mathrm{L/Sy}}$ release at reference L/S, expressed in milligrams per kilogram dry matter (mg/kg dry matter).

(2) For predicting L/S values ≥ 1,0 the fractions 3 to 7 should be used (cumulative L/S 0,5 to 10).

NOTE 1 In case of a quick dissolution, the model may underestimate the release for higher values of L/S (>10). In this case, the model predicts a lower release rate at L/S 10 than measured. The release rate is expressed in (mg/kg)/d.

(3) For predicting L/S-values < 1,0 the fractions 1 to 3 should be used (cumulative L/S 0,1 to 0,5)

NOTE 2 In case the release is solubility controlled and pH does not change significantly, the results of the percolation test can be used directly in conjunction with the L/S – time conversion to estimate release under field conditions. The L/S- ratio in l/kg relates to a time-scale through the infiltration rate, the density and the height of the construction according to:

$$L / S = \frac{t \cdot N}{d \cdot h} \tag{D.12}$$

where

t is the time, expressed in years (year);

N is the infiltration rate, expressed in litres per square meter per year (l/m^2/year);

h is the height or layer thickness of the construction, expressed in metres (m);

d is the density of the construction product, expressed in kilograms per cubic meter (kg/m^3).

D.4 Examples

The following pages show examples of different release mechanisms.

EXAMPLE 1 pH dependent solubility controlled release

Eluate data	
c_1 [µg/I]	710
c_2 [µg/I]	1100
c_3 [µg/I]	1500
c_4 [µg/I]	1500
c_5 [µg/I]	1200
c_6 [µg/I]	660
c_7 [µg/I]	200

pH_1	7,90
pH_2	8,00
pH_3	8,20
pH_4	8,35
pH_5	8,30
pH_6	8,05
pH_7	7,25

Detection limit (DL)	100

Fluoride

Overall low concentrations			
c_{2-7}	1000	c_{2-7}/DL	10,0
$c_{2-7}/DL < 1,5$	no		

Solubility controlled release			
Inert substance	no		
(Apparent) pH dependent release	yes		
c_{1-7}			
σ_C		σ_C/c_{1-7}	
$\sigma_C/c_{1-7} < 0,25$			

Wash-out (depletion)					
c_{1-3}	1100				
c_{5-7}	690	c_{1-3}/c_{5-7}	1,6		
c_{6-7}	430	c_{6-7}/DL	4,3		
pH_{1-2}	7,95	$pH_{4-7}-2\sigma_{pH4-7}$	7,11	$pH_{4-7}+2\sigma_{pH4-7}$	8,87
$c_{1-3}/c_{5-7} > 2,0$ and $c_{6-7}/DL < 1,5$			n.a.	(pH dependent release)	
$c_{1-3}/c_{5-7} > 2,0$ and $c_{6-7}/DL < 1,5$ and $pH_{4-7}-2\sigma_{pH4-7} < pH_{1-2} < pH_{4-7}+2\sigma_{pH4-7}$			no		

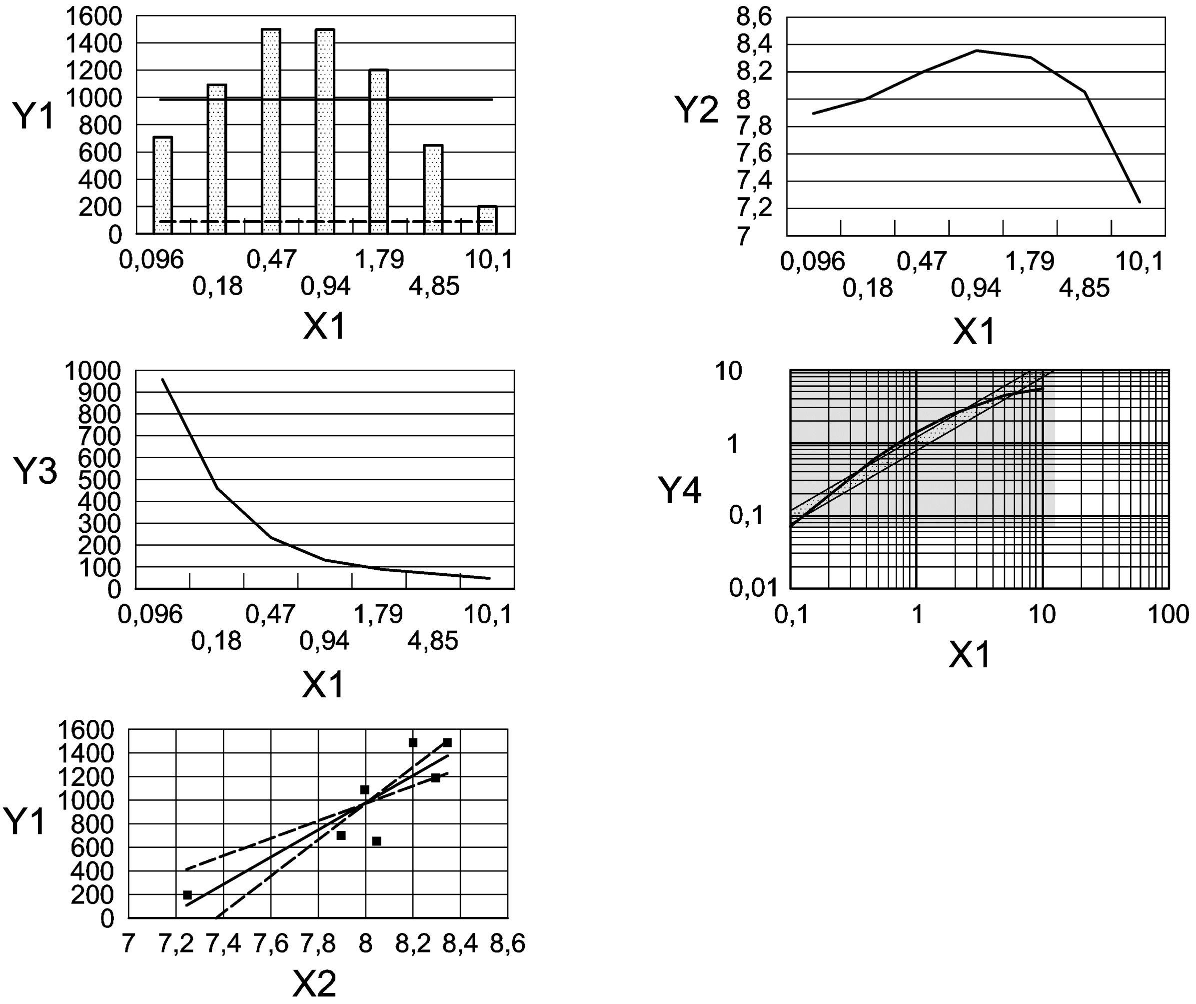
pH dependent solubility controlled release	
pH dependent release and no wash-out	yes

Apparent depletion			
c_{1-4}		c_{1-4}/c_{6-7}	
$c_{1-4}/c_{6-7} > 1,5$ and $c_{1-4}/DL > 1,5$			

Conclusion:	pH dependent solubility controlled release

Calculations					
Grain size:	option A				
E_1	0,068	[mg/kg dry matter]	U_1	0,068	[mg/kg dry matter]
E_2	0,092	[mg/kg dry matter]	U_2	0,16	[mg/kg dry matter]
E_3	0,44	[mg/kg dry matter]	U_3	0,6	[mg/kg dry matter]
E_4	0,71	[mg/kg dry matter]	U_4	1,3	[mg/kg dry matter]
E_5	1	[mg/kg dry matter]	U_5	2,3	[mg/kg dry matter]
E_6	2	[mg/kg dry matter]	U_6	4,3	[mg/kg dry matter]
E_7	1,1	[mg/kg dry matter]	U_7	5,4	[mg/kg dry matter]

Cumulative release for L/S 2:	2,3	[mg/kg dry matter]
Cumulative release for L/S 10:	5,4	[mg/kg dry matter]



Linear regression (pH,c)

80 % confidence interval slope – lower limit: 747,9

80 % confidence interval slope – upper limit: 1 544,1

pH independent release (slope = 0): no

Key

Y1	concentration in µg/l	X1	cumulative L/S ratio
Y2	pH	X2	pH
Y3	electrical conductivity in µS/cm		
Y4	cumulative release in mg/kg dry matter		

Figure D.2 — pH dependent solubility controlled release

EXAMPLE 2 Solubility controlled release

Eluate data	
c_1 [µg/l]	110
c_2 [µg/l]	86
c_3 [µg/l]	79
c_4 [µg/l]	77
c_5 [µg/l]	82
c_6 [µg/l]	110
c_7 [µg/l]	89

pH_1	8,85
pH_2	8,35
pH_3	8,70
pH_4	8,70
pH_5	9,00
pH_6	9,55
pH_7	9,85

Detection limit (DL)	1

Barium

Overall low concentrations			
c_{2-7}	87	c_{2-7}/DL	87,0
$c_{2-7}/DL < 1,5$	no		

Solubility controlled release			
Inert substance	no		
(Apparent) pH dependent release	no		
c_{1-7}	90		
σ_C	13	σ_C/c_{1-7}	0,1
$\sigma_C/c_{1-7} < 0,25$	yes		

Wash-out (depletion)		
c_{1-3}		
c_{5-7}	c_{1-3}/c_{5-7}	
c_{6-7}	c_{6-7}/DL	
pH_{1-2}	$pH_{4-7}-2\sigma_{pH4-7}$	$pH_{4-7}+2\sigma_{pH4-7}$
$c_{1-3}/c_{5-7} > 2,0$ and $c_{6-7}/DL < 1,5$		(pH dependent release)
$c_{1-3}/c_{5-7} > 2,0$ and $c_{6-7}/DL < 1,5$ and $pH_{4-7}-2\sigma_{pH4-7} < pH_{1-2} < pH_{4-7}+2\sigma_{pH4-7}$		(no pH dependent release)

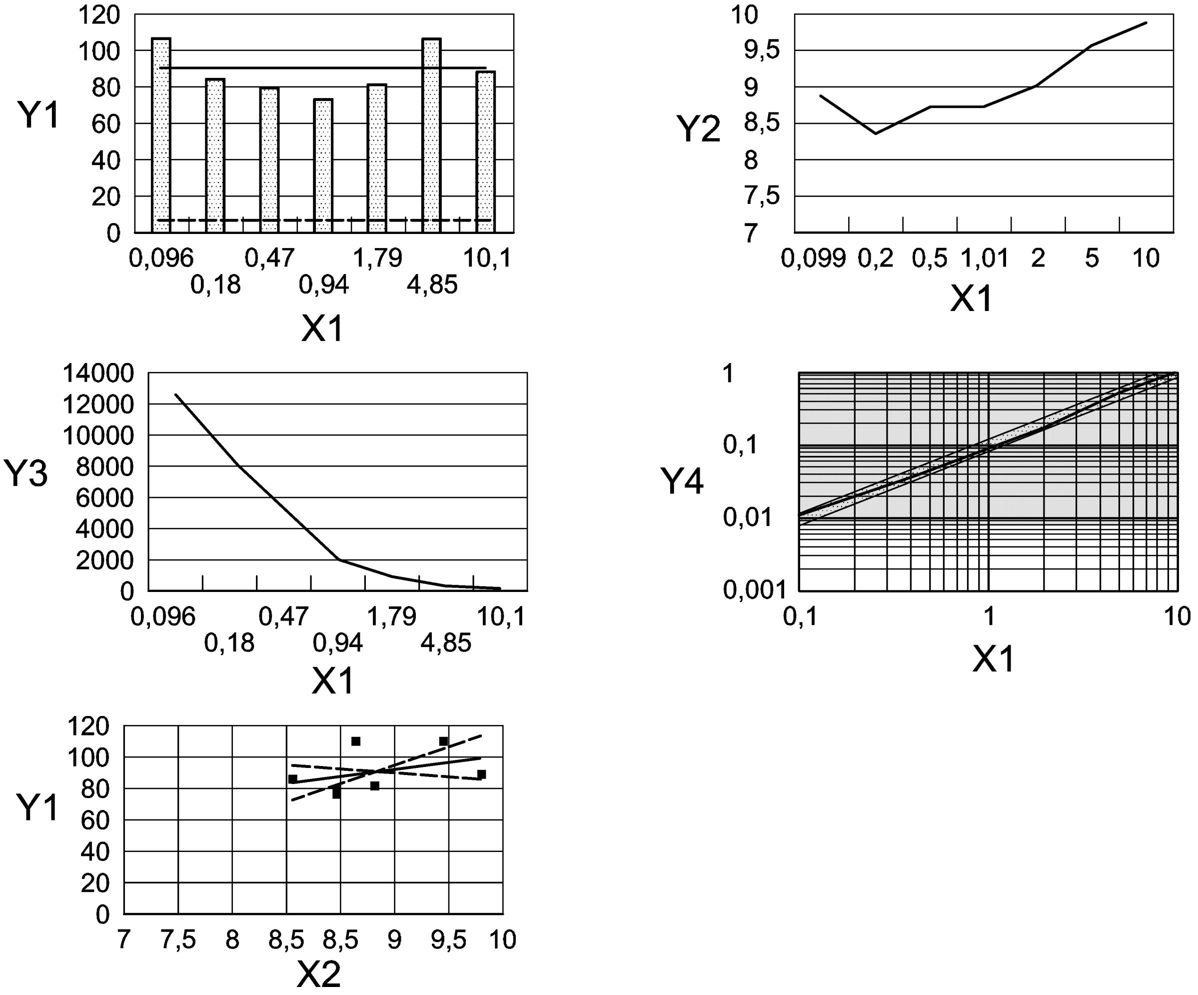
pH dependent solubility controlled release
pH dependent release and no wash-out

Apparent depletion	
c_{1-4}	c_{1-4}/c_{6-7}
$c_{1-4}/c_{6-7} > 1,5$ and $c_{1-4}/DL > 1,5$	

Conclusion:	solubility controlled release

Calculations					
Grain size:	option A				
E_1	0,011	[mg/kg dry matter]	U_1	0,011	[mg/kg dry matter]
E_2	0,0087	[mg/kg dry matter]	U_2	0,02	[mg/kg dry matter]
E_3	0,024	[mg/kg dry matter]	U_3	0,043	[mg/kg dry matter]
E_4	0,039	[mg/kg dry matter]	U_4	0,083	[mg/kg dry matter]
E_5	0,081	[mg/kg dry matter]	U_5	0,16	[mg/kg dry matter]
E_6	0,33	[mg/kg dry matter]	U_6	0,49	[mg/kg dry matter]
E_7	0,45	[mg/kg dry matter]	U_7	0,94	[mg/kg dry matter]

Cumulative release for L/S 2:	0,16	[mg/kg dry matter]
Cumulative release for L/S 10:	0,94	[mg/kg dry matter]



Linear regression (pH,c)

80 % confidence interval slope – lower limit: −5,9

80 % confidence interval slope – upper limit: 26,5

pH independent release (slope = 0): yes

Key

Y1	concentration in µg/l	X1	cumulative L/S ratio
Y2	pH	X2	pH
Y3	electrical conductivity in µS/cm		
Y4	cumulative release in mg/kg dry matter		

Figure D.3 — Solubility controlled release

EXAMPLE 3 Wash-out

Eluate data	
c_1 [µg/I]	5300
c_2 [µg/I]	2100
c_3 [µg/I]	1500
c_4 [µg/I]	570
c_5 [µg/I]	230
c_6 [µg/I]	100
c_7 [µg/I]	100

pH_1	12,20
pH_2	12,15
pH_3	12,15
pH_4	12,10
pH_5	12,05
pH_6	12,10
pH_7	12,05

Detection limit (DL)	100

Bromide

Overall low concentrations			
c_{2-7}	770	c_{2-7}/DL	7,7
$c_{2-7}/DL < 1{,}5$	no		

Solubility controlled release			
Inert substance	yes		
(Apparent) pH dependent release	no		
c_{1-7}	1400		
σ_C	1700	σ_C/c_{1-7}	1,2
no	yes		

Wash-out (depletion)					
c_{1-3}	3000				
c_{5-7}	140	c_{1-3}/c_{5-7}	21,4		
c_{6-7}	100	c_{6-7}/DL	1,0		
pH_{1-2}	12,18	$pH_{4-7}-2\sigma_{pH4-7}$	12,03	$pH_{4-7}+2\sigma_{pH4-7}$	12,13
$c_{1-3}/c_{5-7} > 2{,}0$ and $c_{6-7}/DL < 1{,}5$			yes		
$c_{1-3}/c_{5-7} > 2{,}0$ and $c_{6-7}/DL < 1{,}5$ and $pH_{4-7}-2\sigma_{pH4-7} < pH_{1-2} < pH_{4-7}+2\sigma_{pH4-7}$			n.a.	(no pH dependent release)	

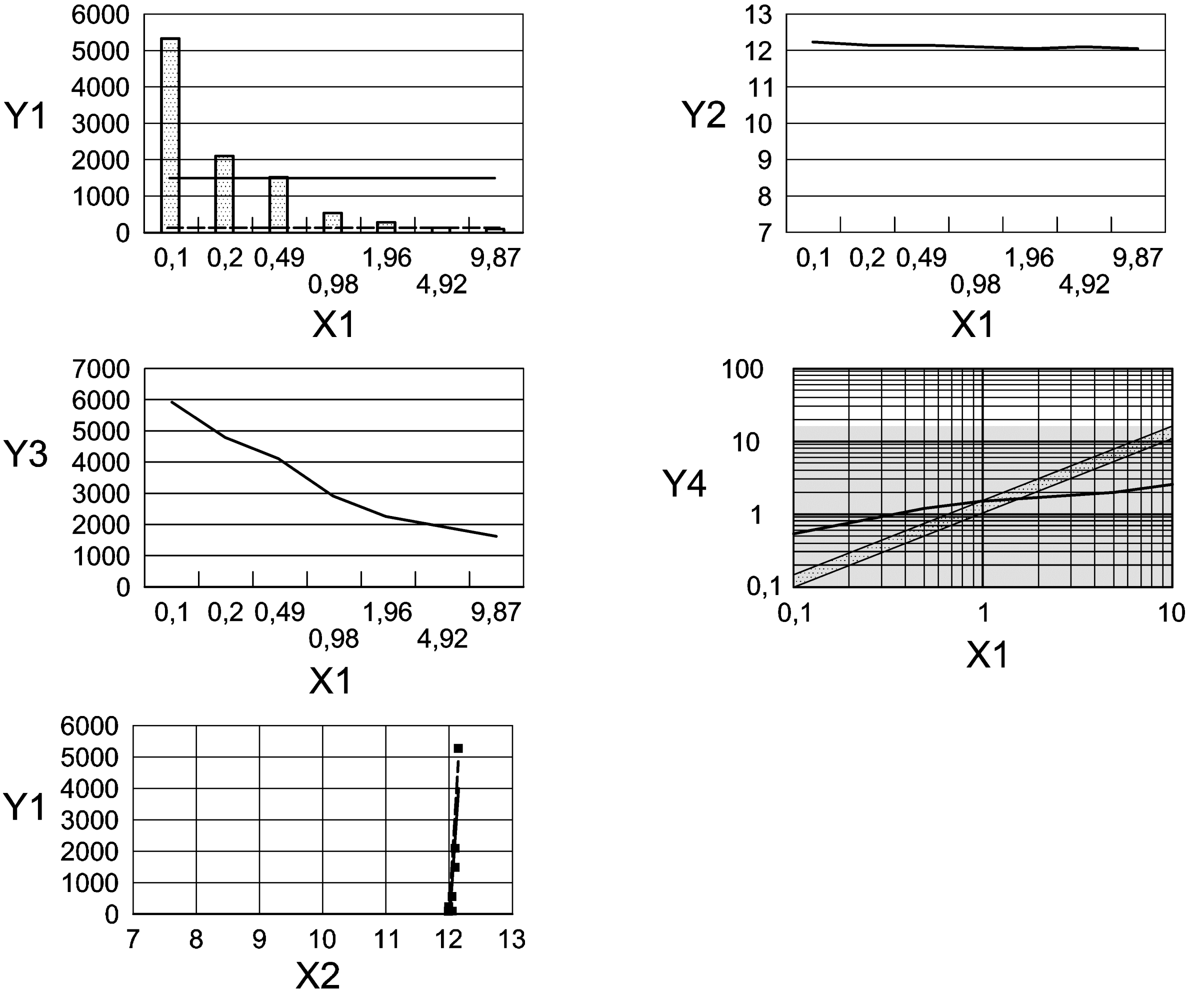
pH dependent solubility controlled release	
pH dependent release and no wash-out	no

Apparent depletion			
c_{1-4}		c_{1-4}/c_{6-7}	
$c_{1-4}/c_{6-7} > 1{,}5$ and $c_{1-4}/DL > 1{,}5$			

Conclusion:	wash-out (depletion)

Calculations					
Grain size:	option A				
E_1	0,53	[mg/kg dry matter]	U_1	0,53	[mg/kg dry matter]
E_2	0,21	[mg/kg dry matter]	U_2	0,74	[mg/kg dry matter]
E_3	0,44	[mg/kg dry matter]	U_3	1,2	[mg/kg dry matter]
E_4	0,28	[mg/kg dry matter]	U_4	1,5	[mg/kg dry matter]
E_5	0,23	[mg/kg dry matter]	U_5	1,7	[mg/kg dry matter]
E_6	0,3	[mg/kg dry matter]	U_6	2	[mg/kg dry matter]
E_7	0,5	[mg/kg dry matter]	U_7	2,5	[mg/kg dry matter]

Cumulative release for L/S 2:	1,7	[mg/kg dry matter]
Cumulative release for L/S 10:	2,5	[mg/kg dry matter]



Linear regression (pH,c)

80 % confidence interval slope – lower limit: 19187,8

80 % confidence interval slope – upper limit: 40266,0

pH independent release (slope = 0): no

Key

Y1	concentration in µg/l	X1	cumulative L/S ratio
Y2	pH	X2	pH
Y3	electrical conductivity in µS/cm		
Y4	cumulative release in mg/kg dry matter		

Figure D.4 — Wash-out

EXAMPLE 4 Apparent depletion

Eluate data	
c_1 [µg/I]	3800000
c_2 [µg/I]	2100000
c_3 [µg/I]	990000
c_4 [µg/I]	280000
c_5 [µg/I]	46000
c_6 [µg/I]	5500
c_7 [µg/I]	1000

pH_1	8,85
pH_2	8,35
pH_3	8,70
pH_4	8,70
pH_5	9,00
pH_6	9,55
pH_7	9,85

Detection limit (DL)	100

Chloride

Overall low concentrations			
c_{2-7}	570000	c_{2-7}/DL	> 100
$c_{2-7}/DL < 1{,}5$	no		

Solubility controlled release			
Inert substance	yes		
(Apparent) pH dependent release	no		
c_{1-7}	1000000		
σ_C	1300000	σ_C/c_{1-7}	1,3
$\sigma_C/c_{1-7} < 0{,}25$	no		

Wash-out (depletion)					
c_{1-3}	2300000				
c_{5-7}	18000	c_{1-3}/c_{5-7}	> 100		
c_{6-7}	3300	c_{6-7}/DL	33,0		
pH_{1-2}	8,60	$pH_{4-7}-2\sigma_{pH4-7}$	8,37	$pH_{4-7}+2\sigma_{pH4-7}$	10,18
$c_{1-3}/c_{5-7} > 2{,}0$ and $c_{6-7}/DL < 1{,}5$			no		
$c_{1-3}/c_{5-7} > 2{,}0$ and $c_{6-7}/DL < 1{,}5$ and $pH_{4-7}-2\sigma_{pH4-7} < pH_{1-2} < pH_{4-7}+2\sigma_{pH4-7}$			n.a.	(no pH dependent release)	

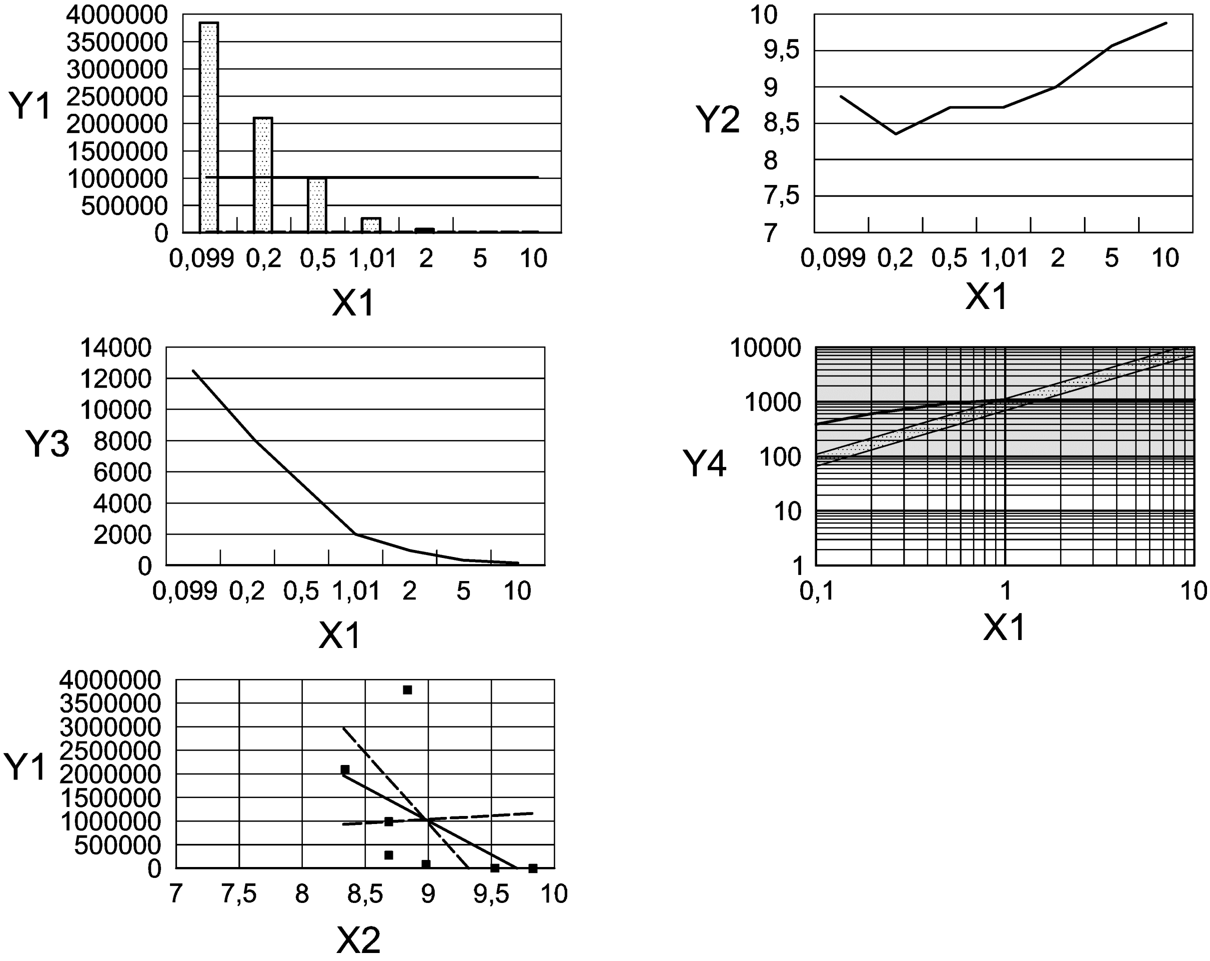
pH dependent solubility controlled release	
pH dependent release and no wash-out	no

Apparent depletion			
c_{1-4}	1800000	c_{1-4}/c_{6-7}	> 100
$c_{1-4}/c_{6-7} > 1{,}5$ and $c_{1-4}/DL > 1{,}5$	yes		

Conclusion:	apparent depletion

Calculations					
Grain size:	option A				
E_1	380	[mg/kg dry matter]	U_1	380	[mg/kg dry matter]
E_2	210	[mg/kg dry matter]	U_2	590	[mg/kg dry matter]
E_3	300	[mg/kg dry matter]	U_3	890	[mg/kg dry matter]
E_4	140	[mg/kg dry matter]	U_4	1000	[mg/kg dry matter]
E_5	46	[mg/kg dry matter]	U_5	1100	[mg/kg dry matter]
E_6	17	[mg/kg dry matter]	U_6	1100	[mg/kg dry matter]
E_7	5	[mg/kg dry matter]	U_7	1100	[mg/kg dry matter]

Cumulative release for L/S 2:	1100	[mg/kg dry matter]
Cumulative release for L/S 10:	1100	[mg/kg dry matter]



Linear regression (pH,c)

80 % confidence interval slope – lower limit: −2961408,8

80 % confidence interval slope – upper limit: 158833,1

pH independent release (slope = 0): yes (apparent pH depency)

Key

Y1	concentration in µg/l	X1	cumulative L/S ratio
Y2	pH	X2	pH
Y3	electrical conductivity in µS/cm		
Y4	cumulative release in mg/kg dry matter		

Figure D.5 — Apparent depletion

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