PD CEN/TS 16637-2:2014

BSI Standards Publication

Construction products — Assessment of release of dangerous substances

Part 2: Horizontal dynamic surface leaching test

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National foreword

This Published Document is the UK implementation of CEN/TS 16637-2:2014.

The UK participation in its preparation was entrusted to Technical Committee B/557, Construction products — Assessment of dangerous substances.

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

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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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Foreword

This document (CEN/TS 16637-2:2014) 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.

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 15863 [1], which is based on NEN 7375:2005 [2].

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.

CEN/TS 16637-1 deals with the determination and use of test methods for leaching of construction products taking specific situations into account. Technical Specification FprCEN/TS 16637-3 (in preparation) specifies an up-flow percolation test to determine the leaching behaviour of granular construction products under standardized percolation 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 [3], CEN/TR 16496 [4]).

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: 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

The informative introduction describes the interactions and interrelations between the three Technical Specifications CEN/TS 16637-1, CEN/TS 16637-2 and FprCEN/TS 16637-3 (in preparation) 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 release method for the determination of the release of Regulated Dangerous Substances from a construction product into soil, surface water and groundwater.

CEN/TS 16637-2 and FprCEN/TS 16637-3 (in preparation) contain the two horizontal leaching tests that are needed to address the leaching properties of the construction products. CEN/TS 16637-2 describes a test to assess surface dependent release from monolithic, plate-like or sheet-like construction products while FprCEN/TS 16637-3 (in preparation) describes a test to assess release from granular construction products. The test methods specified in CEN/TS 16637-2 and FprCEN/TS 16637-3 (in preparation) 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.

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. 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 Annex A and Annex B of CEN/TS 16637-1:2014and to CEN/TR 16098, [3], 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. [5). 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 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 mm^3 (64 cm^3)). It also applies to plate- or sheet-like products with surface areas of minimum 10 000 mm^2 (100 cm^2) exposed to the leachant. Products designed to drain water (e.g. draining tiles, porous asphalt) and monolithic granular products according to CEN/TS 16637-1:2014, 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 CEN/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 CEN/TS 16637-2 is not appropriate.

(7) Guidance on the applicability of the test method to a given product is outlined in CEN/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 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 optimize 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 14346, *Characterization of waste - Calculation of dry matter by determination of dry residue or water content*

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)*

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*

3 Terms and definitions

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

3.1
eluate
solution recovered from a leaching test

3.2
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

[SOURCE: IUPAC 1990 [6], 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 demineralized 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 volume 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 l/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/m^2) 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: IUPAC 1990 [6], 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 1990 [6]]

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 [6], 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^2 exposed surface of test piece(s), in m^2
c_i	concentration of the substance in eluate i, in µg/l
m_a	loss of weight, in g/m^2
m_s	mass of the solid matter that has fallen off during the test, in g
R_n	cumulative area release of the substance for period n including fraction i = 1 to n, in mg/m^2
r_i	area release of the substance in fraction i, in mg/m^2
P	test piece
V_P	volume of the text portion
V_l	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	test for Granular construction products with Low Hydraulic Conductivity
FEP	Fluorinated Ethylene Propylene
FPC	Factory Production Control
HDPE	High-Density Polyethylene
PAH	Polycyclic Aromatic Hydrocarbon
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 demineralized 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 characterized 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 FprCEN/TS 16637-3 (in preparation) (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 characterized 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^2 of geometric surface area of the product exposed to leaching, and milligrams of the substances released per liter of eluate, each after 64 d.

NOTE The results may be additionally expressed and reported in terms of the flux (milligrams of substances released per m^2 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 recognized analytical grade, unless otherwise specified.

6.2 Leachant

Use as a leachant demineralized water or deionized water or water of equivalent purity with a conductivity < 0,5 mS/m according to grade 3 specified in EN ISO 3696.

When the release of 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 can also affect the release of inorganic substances.

6.3 Rinsing solutions

Use as rinsing solution nitric acid (pro analyse), $c(HNO_3)$ = 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.

(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 C and 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 C and Annex D) or be placed in the tank by attaching it to the lid with a wire.

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.

Use membrane filters with a pore size of 0,45 µm (e.g. cellulose acetate, PTFE) for filtration of the eluates to be analysed for inorganic substances. Filters shall not be used for eluates to be analysed for organic substances.

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

7.10 Conductivity meter, with an accuracy of at least 0,1 mS/m.

7.11 Glass or plastic bottles, e.g. glass, HDPE, PMMA, PTFE, PE, PET, PP, PVC.

Use bottles with an appropriate volume, and with screw cap, for eluate collection and preservation of eluate samples (rinsed in accordance with EN ISO 5667-3).

NOTE For inorganic substances HDPE/PP bottles are preferred, except for samples analysed for mercury.

7.12 Redox potential meter (optional).

7.13 Centrifuge

Use a centrifuge 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 both inorganic and organic substances and suitable to high speed centrifugation.

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

8 Sample preparation

8.1 General

(1) The sample preparation shall consist of the preparation of the test sample from the laboratory sample and preparing a test portion including the determination of the geometric surface area.

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

NOTE Usually, the testing laboratory reports on sampling only when it performs the sampling to produce the laboratory sample itself. It is recommended by CEN/TC 351 to the product TCs to include the complement to sampling (for dangerous substances testing) in their product standard or guideline.

8.2 Provisions on test sample and test portion

(1) The test sample should be prepared from the laboratory sample according to the procedure developed by the respective product Technical Committees.

(2) Store the test sample under appropriate conditions given by the respective Product Technical Committee to minimize changes due to the exposure to atmosphere (drying, carbonation, etc.).

(3) The production date and/or curing time of the test sample shall be reported in the test report.

(4) For the performance of the DSLT, a test portion is required, consisting at least of one monolithic or plate- or sheet-like test piece.

(5) To carry out the test at least one test piece is necessary, the structure, homogeneity and composition of which are representative of the product to be tested. For each test piece (P) the smallest dimension shall be greater than 40 mm (to avoid depletion) and the volume (V_P) in litres must be known for practical purposes and reported.

(6) For plate-like and sheet-like products, only 2-D minimum dimensions (10 000 mm^2 (100 cm^2) of exposed surface) apply. However, in case of high release rates, it has to be justified, and corrections should be made by recalculating the release at 64 d or retesting a larger test piece

(7) If a moulded or monolithic product to be tested is produced in a product format of which the smallest dimension is less than 40 mm, this product can be used as test piece P if on one side a geometric area A to be accurately determined is present of at least 7 500 mm^2, and the other sides are covered so they don't contribute to leaching.

(8) If the test portion is prepared by cutting or coring, in order to prevent artefacts, the new created surfaces should be sealed (covered) with an inert material unless otherwise specified.

(9) For the partial covering of a test piece with an impermeable layer, material shall be used that does not have a disruptive effect on the diffusion process due to the giving off, absorption or (delayed) transmission of substances to be tested.

NOTE 1 It has been found that acrylic resin is a suitable impermeable material for leaching tests on inorganic substances with the DSLT.

NOTE 2 Guidance is given in CEN/TS 16637-1.

(10) Prior to testing, the test portion shall be inspected to ensure that it is in proper condition for testing (e.g. no deep cracks or other damages).

(11) In order to increase the representativeness of a test result in one vessel a test portion can be tested that consists of a number of test pieces. The exposed surface area of the test portion is, in that case, the total exposed surface area of the individual test pieces. The volume (V_P) and the geometric area A of test portion should then be equal to, respectively, the total volume and the total geometric area of the combined test pieces. The constituting test pieces shall be separated to prevent blockage of a surface.

(12) Certain construction products are produced as standard with a thickness of less than 40 mm, such as slate roof covering, coarse ceramic roof tiles, thin tiles, hollow bricks or grass paver tiles. Generally, the required strength of these products implicitly leads to materials with such a low release rate that during the DSLT no depletion phenomena occur. For an optimum result in the DSLT such thin pieces shall be covered on one side (see (8)).

(13) The age of products/test pieces is an important factor, which can influence the leaching behaviour. In case the product results from a preparation in the laboratory the product should be cured sufficiently long according to the common practice in product standards (if applicable) to determine the product properties to avoid major variations in leaching due to ongoing changes in pore structure and in formation of release controlling mineral phases.

8.3 Determination of the geometric surface area

8.3.1 General

(1) Shortly before the start of the leaching test, remove dust and loose particles from the test portion by blowing gently using compressed air (quality sufficient to avoid oil contamination).

(2) The determination of the area of the test piece P is carried out by measuring characteristic geometric variables of the geometric outer surface A of the test piece. A distinction is made between:

a) test pieces with a regular, clearly definable geometric area. The geometric area shall be determined in accordance with 8.3.2, where these are test pieces that in all directions, measured perpendicularly to each point of the outer surface, have a minimum dimension of 40 mm;

b) test pieces with a partial, irregular geometric area or test pieces thinner than 40 mm. The geometric area shall be determined according to 8.3.3;

c) test pieces for which no regular side face can be identified. The geometric area shall be determined according to 8.3.4.

NOTE For an accurate determination of the diffusion coefficient it is necessary to accurately and clearly determine the geometric area of a test piece. For this purpose it is preferable to examine test pieces or parts of test pieces, of which the geometric area can be easily and clearly determined.

8.3.2 Regular test pieces

(1) Determination of the geometric area of a regular test piece for which the geometric area of the whole test piece can be clearly measured.

a) Divide the surface of the test piece into a number of flat or curved planes (units), such that for each unit the area can be determined geometrically from measured characteristic variables such as length, width, height and radius.

b) The units referred to under a) shall be selected such that the deviation of distance between the products surface and the defined (imaginary) geometric surface plane (regular flat or regular curved) is nowhere more than 3 mm.

c) Determine the length of the characteristic variables with an uncertainty of ± 1 mm.

d) Calculate the geometric area of each of the units selected with the characteristic units measured. The geometric area (A), expressed in m^2, is the sum of the calculated surface areas of each of the units.

8.3.3 Test pieces with partly irregular side face

(1) Determination of the geometric area of the test piece for which:

a) the whole geometric area cannot be clearly measured;

b) one or more sides have been produced by sawing or drilling the test piece from a larger element and where these sides are not involved in the leaching;

c) one dimension is less than 40 mm.

(2) Cover the parts of the surface:

a) for which the geometric area cannot be clearly determined with a waterproof layer;

b) that has been produced as sawn and drilled faces with a waterproof layer;

c) of a thin test piece (with a thickness of 40 mm or less) with a waterproof layer, such that the uncovered test piece of the geometric area nowhere have a distance between them of 40 mm or less, measured perpendicularly to each point of the geometrically described area. For the covering use a waterproof and good bonding material (for example acrylic resin or paraffin) that is applied to the surface of the test piece. Determine the geometric area remaining free after the resin has hardened.

(3) Divide the uncovered part of the test piece surface into a number of flat or curved planes, such that for each unit the surface area can be determined geometrically from measured characteristic variables such as length, width, height and radius.

(4) The test piece under point (2) shall be selected such that the defined geometric areas coincide with the relevant plane of the test piece, where the actual distance between the material and the defined plane of that unit in case of irregularities in the surface is nowhere greater than 3 mm.

Determine the length of the characteristic variables with an uncertainty of ± 1 mm.

8.3.4 Irregular test pieces without any regular sides

(1) Determination of the geometric area of very irregular test pieces using the aluminium foil method.

a) Cover each face of the test piece as tightly as possible with a piece of aluminium foil.

b) Fold the aluminium foil around the edges of each face of the test piece and tear or cut the aluminium foil as accurately as possible along the folds. Remove any pieces of aluminium foil projecting outside the plane.

c) Determine the total weight of the pieces of aluminium foil obtained after step b).

d) Determine the weight of a sheet of aluminium foil with a known area and comparable properties to the foil used in step a).

e) Calculate the area of the test piece from the ratio of the foil weights determined according to steps c) and d).

f) Repeat steps a) to e), unless in the diffusion test four or more test pieces from one batch are used together. Determine the average of the measurements carried out.

(2) Instead of aluminium foil, paper (e.g. normal printer paper or copier paper) can be used if the test pieces have a dry surface. It is important that the paper does not have highly water-absorbent properties. If the test piece is damp, it may be necessary to dry the paper used before the weighing in step c).

NOTE An example of the application of the aluminium foil method is given in [8].

9 Test procedure

9.1 Testing conditions

The DSLT shall be carried out at a temperature of the leachant in the range from 19 °C to 25 °C.

NOTE A constant temperature from 19 °C to 25 °C in the test can be achieved by either controlling the temperature of the lab, or by controlling the temperature of the leachant and insulating the leaching vessel.

9.2 Step 1 of the leaching procedure

(1) For monolithic products the test is carried out at an L/A ratio of (80 ± 10) l/m^2. For sheet- or plate-like products lower L/A ratios ≥ 20 may be applied. Calculate the leachant volume V_l:

$$V_l = (L/A) \times A \tag{1}$$

where

V_l is the volume of the leachant, in l.

NOTE 1 A lower L/A ratio for sheet and plate like products is allowed due to risks of running into limit of quantification for some of these products.

(2) Place the test portion in the leaching vessel (7.2) using the support (7.2), in order to prevent the test portion from touching the inner side of the leaching vessel. Alternatively, the test portion can be suspended in strings of an appropriate material attached to the lid of the leaching vessel. If the test portion consists of more than one test piece, the support shall be used in such a way that there is at least 20 mm space between the test pieces. If the test portion is a plate-like or sheet-like product, it may instead be affixed to the inner walls of the leaching vessel with the exposed surface area facing into the vessel. Care shall be taken to avoid water contact with the edges unless such contact is part of the intended use.

(3) Fill the vessel with the calculated volume (V_l) of leachant (specified in 6.2), such that the top of the test portion is at least 20 mm submerged. Close the leaching vessel. Note the time t_0. If the test piece is sheet-like and is affixed to the vertical, inner walls of the vessel, care shall be taken to fill the vessel with the exact same amount of water in each step of the test to ensure that the same surface area of the product is exposed to water in each step. The exposed surface area (A) is calculated as the submerged part of the wall.

NOTE 2 To cope with different test piece dimensions it might be necessary to use various dimensions for the test vessel to meet the requirements on V_l and a gap of 20 mm.

Sheet-like products that soak up water should be completely submerged.

(4) Allow the leaching process of this first step until (6 h ± 15 min) after the addition of the leachant.

(5) At the end of the first step, remove the eluate from the leaching vessel, without removing small parts of material that may have fallen off the test portion. If the test piece is standing on a support, it may be necessary to lift it out while the leaching vessel is emptied. Alternatively, the vessel can be equipped with a tap at bottom, or with an arrangement to remove leachate by hydrostatic pressure. If the test piece is suspended from the lid, it will automatically be removed from the vessel when the lid is lifted. The lid and test piece can be placed on a vessel similar to the test vessel during the exchange of leachant. Note the time t_1. Immediately continue with step 2 (9.3).

(6) If only inorganic substances are of interest, the following procedure may be used:

(7) Immediately measure pH and conductivity. Filter the eluate fraction off-line over a 0,45 µm membrane filter and further prepare the eluate for analysis (see 9.5).

(8) If non-volatile organic substances are of interest, the following procedure should be followed:

(9) Transfer the supernatant to centrifuge tubes (7.13). There are two options for solid-liquid separation:

a) centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high speed centrifuge (7.13), or

b) centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower speed centrifuge (7.13).

NOTE 3 Based on Stoke's law the results of both centrifugation methods are expected to be comparable.

(10) However, for clear eluates with no visible particulates or turbidity below 20 FNU, centrifugation may not be necessary.

(11) Control the temperature at (19 to 25) °C (see 9.1).

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

(13) Measure pH and conductivity of non-filtered eluate and further prepare the eluate for analysis (see 9.5).

9.3 Steps 2 – 8 of the leaching procedure

(1) Fill the vessel again with the calculated volume of leachant (V_1), such that the top of the test portion is at least 20 mm submerged. Close the leaching vessel. If the test piece is sheet-like and is affixed to the vertical, inner walls of the vessel, special care shall be taken to fill the vessel with the exact same amount of water in each step of the test.

(2) Allow the leaching process of each step until the time mentioned in Table 1.

(3) At the end of each step remove the eluate from the leaching vessel, without removing small parts of material that may have fallen off the test portion. Note the time t_i for i being the number of the subsequent steps. Immediately continue with the next step, following the procedure described above.

(4) Centrifuge or filter the eluate fraction as described in 9.2. Immediately measure pH and conductivity, and further prepare the eluate for analysis (see 9.5).

Table 1 — Renewal times of the leachant

Step/fraction	Duration of the specific step	Duration from the start of the test (t_0)
1[a]	6 h ± 15 min	6 h
2	18 h ± 15 min	1 day
3	1 day and 6 h ± 45 min	2 d and 6 h
4	1 day and 18 h ± 75 min	4 d
5	5 d ± 75 min	9 d
6	7 d ± 75 min	16 d
7	20 d ± 7 h	36 d
8	28 d ± 12 h	64 d

[a] Step 1 is described in 9.2.

NOTE 1 The liquid renewal frequency is designed and carried out such that the concentration in solution does not affect the release rate from the test portion significantly, because the concentration in the solution is far below the saturation concentration.

NOTE 2 If the full duration of the test is not required (e.g. for factory production control) a test consisting of less than eight steps can be performed as "indirect test" as explained in the Introduction. This is an example of an indirect procedure; see Clause 12 for an explanation.

9.4 Measuring the loss of weight

Collect the solid matter at the bottom of the tank that has fallen off the test piece(s) at the end of step 2 (total of step 1 and 2) and at the end of the last step (total of step 3 to N, where N is the last step). Dry and weigh the two amounts of solid matter according to EN 14346.

NOTE The loss of weight is focused on the solid matter at the bottom of the tank; the dissolved matter is not considered.

9.5 Further preparation of the eluates for analysis and analysis steps

(1) Measure the pH in accordance with EN 16192 and the conductivity in accordance with EN 16192 (and optionally redox potential E_h in mV) of each eluate fraction, just after collection (see 9.2). 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.

(2) 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.

(3) 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.

(4) Determine the concentrations of substances of interest using appropriate methods of eluate analysis for inorganic or organic substances. If relevant, the total concentration of dissolved organic carbon (DOC) should be measured.

NOTE 1 The pH, electric conductivity, DOC and redox measurements can be used for verification purposes in case measurements deviate from the expected release behaviour. This will provide the user with the option to accept the results, verify deviations from normal or take corrective measures based on an understanding of the cause of the deviation.

NOTE 2 In CEN/TC 351/WG5, procedures for eluate analysis will be specified and validated. Until these are ready, reference can be made to, e.g. EN 16192 or CEN/TR 16045 [7].

NOTE 3 DOC may be of relevance when a product containing organic matter is stabilized, e.g. with a hydraulic binder. DOC is mobilized at high pH and is capable of mobilizing metals and organic substances well beyond its solubility limitation under the common alkaline conditions.

9.6 Blank test

(1) A blank test shall be carried out on a regular basis, in order to detect, as far as possible, contamination from equipment and reagents. Fill the leaching vessel (7.2) completely with leachant (6.2), and leave the leachant in the vessel for (24 ± 1) h. Then submit it to the subsequent filtration, storage, preservation and analysis steps as described in the procedure for the regular eluates. Fill the leaching vessel again with leachant (6.2) and leave the leachant in the vessel for another (24 ± 1) h. Submit this second blank eluate to the regular filtration, and measure the conductivity.

NOTE The frequency of blank testing is related to the number of tests a laboratory performs and is to be assured in the quality control system of the laboratory.

(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 limit of quantification.

b) The electrical conductivity of the second blank eluate shall be < 0,2 mS/m.

(3) If these requirements are not fulfilled, take the necessary steps to reduce the contamination.

10 Evaluation of measurement results

10.1 Expression of results in concentrations

(1) The analysis of the eluates produced by the leaching test provides the value of the concentration of the substances in the eluates of the individual time steps expressed in mg/l or µg/l.

(2) The test results and the outcome of the calculations should be reported and checked according to Clause 10.

10.2 Expression of results in terms of area related release

(1) Calculate the area release of a substance for each period (fraction):

$$r_\mathrm{i} = \frac{c_\mathrm{i} \times V}{A} \times 0{,}001 \qquad (2)$$

where

period i is the sum of steps from step 1 to step i;

r_i is the area related release of a substance in period i (i = 1 to 8), in mg/m^2;

c_i is the concentration of the substance in eluate i, in µg/l;

V is the volume of the leachant, in l;

A is the area of the test portion, in m^2.

(2) If the concentration of a substance in a particular eluate i is below the quantification limit, two calculations shall be carried out for that substance. The upper limit of r_i is calculated by setting c_i equal to the quantification limit. The lower limit of r_i is calculated by setting c_i = 0.

(3) Calculate for a substance the cumulative area release R_n for each of the periods 1 to 8, where the period 1 lasts from the start of the test to the first renewal time (includes the fraction 1), period 2 from the start of the test to the second renewal time (includes the fractions 1 and 2), etc. Calculate R_n as follows:

$$R_\mathrm{n} = \sum_{i=1}^{n} r_\mathrm{i} \quad \text{for } n = 1 \text{ to } 8 \qquad (3)$$

where

R_n is the cumulative area release of the substance for period n including fraction i = 1 to n, in mg/m^2;

r_i is the area release of the substance in fraction i, in mg/m^2.

(4) The test results and the outcome of the calculations should be reported and checked according to Clause 10.

10.3 Calculation of release mechanism

If the release mechanism has to be determined, the calculation shall be performed as given in Annex B and reported.

10.4 Calculating the loss of weight

(1) Calculate the loss of weight as follows:

$$m_{\mathrm{a}} = \frac{m_{\mathrm{s}}}{A} \qquad (4)$$

where

m_{a} is the loss of weight, in g/m^2;

m_{s} is the mass of the dried solid matter that has fallen off during the test, in g;

A is the exposed surface of test piece(s), in m^2.

(2) Report the loss of weight during step 1-2 ($m_{\mathrm{a1\text{-}2}}$) and step 3-N ($m_{\mathrm{a3\text{-}N}}$) separately and the sum of the two numbers ($m_{\mathrm{a}} = m_{\mathrm{a1\text{-}2}} + m_{\mathrm{a3\text{-}N}}$), where N is the last step.

NOTE A relatively large loss of weight during step 1-2 compared to the loss of weight during step 3-N may indicate a loss which is the result of the preparation and handling of the test piece(s). This may for instance be observed when testing slow hardening mixtures with limited strength development. On the other hand, a relatively large and increasing loss of weight during step 3-N compared to the loss of weight during step 1-2 may indicate a poor degree of long term dimensional stability under water.

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;
4) way in which the test sample is obtained from the laboratory sample (e.g. selection of test sample, moulding test pieces in the laboratory, cutting/sawing/coring), and justification of this way of preparation of the test sample;
5) curing time and conditions of the test sample;
6) storage conditions of the laboratory sample and test sample;
7) dimensions of the test portion, and calculation method of the geometric surface area (A) of the test portion, as well as the geometric surface area itself and the volume of the test portion;
8) the way in which the test portion is suspended in or affixed to the leaching vessel;
9) 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 dimensions of the leaching vessel;

   3) description and justification of the choice of materials for the equipment;

   4) volume V_1 of the leachant, added to the test portion each period;

   5) leachant renewal times of all periods (t_i *to* t_8);

   6) L/A ratio;

   7) temperature range during the performance of the test;

   8) loss of weight ($m_{a1\text{-}2}$ and $m_{a3\text{-}N}$ and the sum of the two numbers ($m_a = m_{a1\text{-}2} + m_{a3\text{-}N}$) during the test;

   9) any deviation from the test method and the reason of this deviation together with all circumstances that may have influenced the results.

c) Analytical report:

   1) pH, 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 substances 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 concentrations per step for each substance of interest, in mg/l or µg/l, presented in a table of results which also shows contact time and volume of the eluate from each step;

   2) the released quantities r_i calculated per step for each substance, in mg/m^2 geometric surface area of the product presented in a table; for analysis results below the limit of quantification for the substances concerned both the lower limit and the upper limit of the released quantity shall be indicated;

   3) the cumulatively released quantities R_n (n = 1 to 8) for each substance, in mg/m^2 geometric surface area of the product, tabled and plotted against time (on a double logarithmic scale). Again, if relevant, lower and upper limits shall be indicated;

   4) if required, description of the release mechanism, according to Annex B;

   5) area related release rate (flux) for each substance and eluate fraction tabled, in mg/(m^2·s); optional.

12 Test performance

When this Technical Specification was adopted by CEN, the robustness was assessed [5]. At that time there was no available data for repeatability and reproducibility.

13 Indirect methods

13.1 Definition

This Technical Specification specifies the “horizontal” reference methods in Clause 1 to Clause 11. 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 emission 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

An example of a possible "indirect" method is a tank leaching test with less than eight steps in the leachant renewal scheme (e.g. for factory production control).

Annex A
(normative)

Method for Granular construction products with Low Hydraulic Conductivity (GLHC)

A.1 Scope

(1) The (GLHC)-mode of the DSL-test applies to those products that have a hydraulic conductivity too low to perform a regular percolation test (e.g. clayey, paste-like products, highly compacted granular products).

(2) The (GLHC)-mode 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. In CEN/TS 16637-1, guidance is given under what circumstances to apply this procedure.

A.2 Terms, definitions and abbreviations

The same terms, definitions and abbreviation are valid as for DSLT; see Clause 3 and Clause 4.

A.3 Principle

The (GLHC)-mode is intended to determine the release of inorganic and/or non-volatile organic substances by diffusion from a granular product. A test set-up for this diffusion-dominated release is achieved by mildly compacting the granular product in a cylindrical vessel and then exposing it on one side to the leachant. The product is first moistened to a moisture content corresponding to saturation after compaction. The degree of compaction is not particularly critical. Figure A.1 shows the test set-up schematically.

NOTE Although mainly focussed on diffusion, solubility control cannot be ruled out, but this mechanism can generally be identified from the release pattern.

A.4 Equipment

A.4.1 General

(1) The equipment and requisites listed below shall be checked before use for proper operation and absence of disrupting elements that may affect the result of the test. They may not give off or absorb any of the components to be determined in the eluates.

(2) The equipment listed below shall also be calibrated.

A.4.2 Sealable outer vessel with cylindrical inner vessel

(1) Sealable outer vessel with a cylindrical inner vessel, both of material suitable for the substances that will be analysed (e.g. plastic without softeners in case of inorganic substances or glass in case of organic substances), in which the product to be tested can be compacted and exposed on one side to a leachant. The inner vessel shall have an internal diameter of (100 ± 5) mm and an internal height of (100 ± 5) mm. The outer

vessel shall be of such dimensions that during the test for each refreshment cycle no more than 0,7 l leachant is necessary to allow the fluid level to end up at least 20 mm above the inner vessel.

(2) The outer vessel shall be fitted at the bottom with a tap in such a way that during the different refreshment cycles eluates can be drawn off without disrupting the diffusion profile in the test portion P. Alternatively, an arrangement for removal of the leachant by hydrostatic pressure (without a bottom tap) can be used. In this case the inner vessel can stand directly on the bottom of the outer vessel.

NOTE 1 The dimensions of the inner and outer vessel are selected such that for the products tested measurable concentrations are usually obtained in the eluates (A.5).

NOTE 2 The height of the inner vessel is selected such that in the product to be tested no exhaustion of any component can occur during the course of the test.

A.4.3 Glass beads

Glass beads with a diameter between 2 mm and 3 mm.

A.5 Sampling

(1) The sample preparation shall consist of the preparation of the test sample from the laboratory sample and preparing a test portion.

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

NOTE As usual in accreditation procedures, the testing laboratory reports on sampling only when it performs sampling to produce laboratory sample itself. As provided by CEN/TC 351, the product TCs have been invited to include the complement to sampling (for dangerous substances testing) in their product standard or guideline.

A.6 Procedure

A.6.1 Testing conditions

(1) The GLHC shall be carried out at a temperature of the leachant in the range from 19 °C to 25 °C.

NOTE 1 A constant temperature from 19 °C to 25 °C in the test can be achieved by either controlling the temperature of the lab, or by controlling the temperature of the leachant and insulating the leaching vessel.

(2) Determine the area A of the internal cylinder diameter of the inner vessel (A.4.2).

(3) Rinse the outer and inner vessel (A.4.2) before carrying out the test with rinsing solution (6.3) and rinse afterwards with demineralized water (6.2).

(4) The inner vessel shall be filled as follows:

a) Weigh the vessel empty (M_{ves1}) with an accuracy of ± 0,1 g (7.4).

b) Weigh with the same accuracy a quantity of product that is estimated to be necessary as a minimum to completely fill the inner vessel (M_{m1}).

c) Weigh with an accuracy of ± 100 mg an excess of demineralized water (6.2) that is necessary as a minimum to fill the outer vessel to 20 mm above the rim of the inner vessel (M_{w1}).

d) Place a 10 mm to 20 mm thick layer of the weighed quantity of product on the bottom of the inner vessel and saturate this layer with part of the quantity of water weighed.

e) Compact the layer lightly by dropping a drop weight of approximately 100 g three times from a height of approximately 200 mm along a rod used as a guide onto the product. Fix this rod to the centre of a disk, which is placed on the layer to be packed. Cover the whole surface of the vessel with the disk. Add another layer of from 10 mm to 20 mm, saturate this layer too with part of the quantity of water weighed and compact the layer with the drop weight. Repeat this layer by layer build-up until the inner vessel is filled to (10 ± 2) mm below the rim. Before applying the last layer, check the remaining height and adjust the necessary mass in order to get (10 ± 2) mm distance from the rim of the vessel.

f) Weigh the vessel thus filled (M_{ves2}), the remainder of the excess product (M_{m2}) and the remainder of the quantity of water weighed (M_{w2}).

g) Calculate the volume of the test portion (V_{P}) from the dimensions of the vessel. Calculate the density of the product in the inner vessel (ρ) according to:

$$\rho = \left(M_{\mathrm{m1}} - M_{\mathrm{m2}}\right)/V_{\mathrm{p}} \tag{A.1}$$

where

ρ is the density of the product in the test portion P, in kg/m^3;

M_{m1} is the weighed quantity of product with which the filling procedure of the inner vessel was started, in g;

M_{m2} is the remainder of the weighed quantity of product M_{m1} after the end of the filling procedure, in g;

V_{P} is the volume of the test portion P, in dm^3.

(5) The difference between $\left(M_{\mathrm{m1}} - M_{\mathrm{m2}} + M_{\mathrm{w1}} - M_{\mathrm{w2}}\right)$ and $M_{\mathrm{ves2}} - M_{\mathrm{ves1}}$ may not be more than 5 %, where:

M_{w1} is the weighed quantity of demineralized water with which the filling procedure of the inner vessel was started, in g;

M_{w2} is the remainder of the weighed quantity of water M_{w1} after the end of the filling procedure, in g;

M_{ves1} is the mass of the empty inner vessel, in g;

M_{ves2} is the mass of the inner vessel after the end of the filling procedure, in g.

(6) Place a layer of glass beads with a height of (10 ± 2) mm on the water-saturated layer of product and place the inner vessel in the outer vessel.

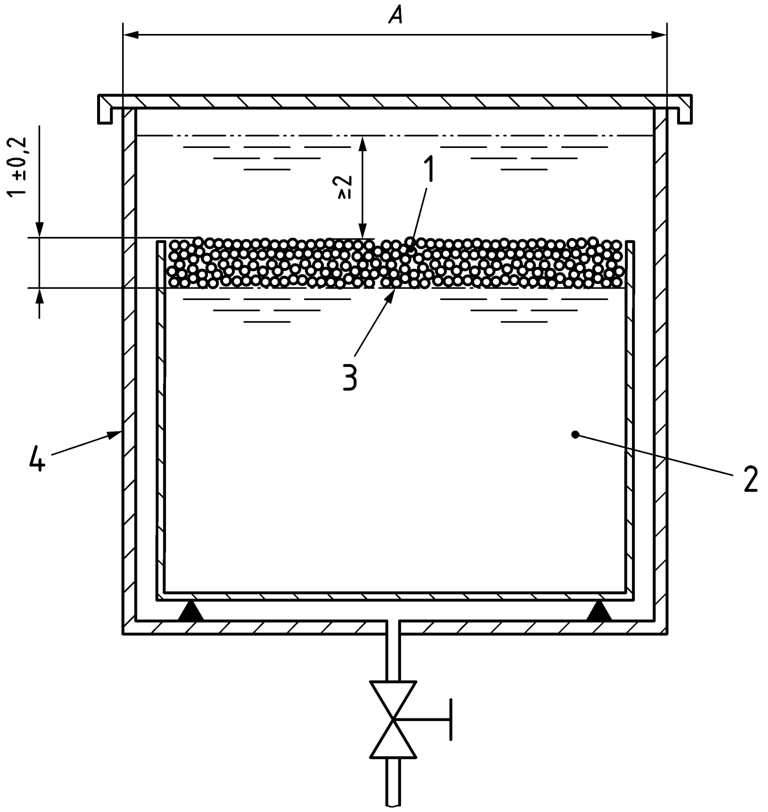
NOTE 2 The layer of glass beads serves to avoid disturbing the compacted bed when changing the fluid.

NOTE 3 Due to incomplete compaction and/or venting, start-up effects may occur that do always lead to a lower initial emission. The layer of glass beads may itself also ensure a slight start up effect, because the concentration in the pore water between the beads needs to first be built up before there is any emission to the leachant.

(7) Non-powdery materials can generally be packed as they are (moist or dry). However, powdery, dry materials should be humidified either at an arbitrary and imposed ratio or referring to the Proctor optimum humidity, if known.

(8) Materials can be too wet to pack well in the GLHC vessel. In that case, 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.

Dimensions in millimetres



Key

A	area
1	glass beads
2	material to be tested
3	diffusion
4	diffusion profile

Figure A.1 — Scheme of method for granular construction products with low hydraulic conductivity

A.6.2 Step 1 of the leaching procedure

(1) Close the leaching vessel. Note the time t_0. Allow the leaching process of this first step until (6 h ± 15 min) after the addition of the leachant $(M_{w1} - M_{w2})$.

(2) At the end of the first step, remove the eluate from the outer leaching vessel with a tap at bottom, or with an arrangement to remove leachate by hydrostatic pressure. Note the time t_1. Immediately continue with step 2 (A.6.3).

(3) If only inorganic substances are of interest, the following procedure may be used:

(4) Immediately measure pH and conductivity. Filter the eluate fraction off-line over a 0,45 µm membrane filter, and further prepare the eluate for analysis (see 9.5).

(5) If non-volatile organic substances are of interest, the following procedure should be followed:

(6) Transfer the supernatant to centrifuge tubes (7.13). There are two options for solid-liquid separation:

a) centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high speed centrifuge (7.13), or

b) centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower speed centrifuge (7.13).

NOTE Based on Stoke's law the results of both centrifugation methods are expected to be comparable.

(7) However, for clear eluates with no visible particulates or turbidity below 20 FNU, centrifugation may not be necessary.

(8) Cooling shall be applied to maintain the temperature at (19 to 25) °C (see A.6.1).

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

(10) Measure pH and conductivity of non-filtered eluate and further prepare the eluate for analysis (see 9.5).

A.6.3 Steps 2 – 8 of the leaching procedure

(1) Fill the vessel again with the leachant $(M_{\mathrm{w1}} - M_{\mathrm{w2}})$, such that the top of the test portion is at least 20 mm submerged. Close the leaching vessel.

(2) Allow the leaching process of each step until the time mentioned in Table A.1.

(3) At the end of each step remove the eluate from the leaching vessel with a tap at bottom, or with an arrangement to remove leachate by hydrostatic pressure. Note the time t_i for i being the number of the subsequent steps. Immediately continue with the next step, following the procedure described above.

(4) Centrifuge or filter the eluate fraction as described in A.6.2. Immediately measure pH and conductivity, and further prepare the eluate for analysis (see 9.5).

Table A.1 — Renewal times of the leachant

Step/fraction	Duration of the specific step	Duration from the start of the test (t_0)
1[a]	6 h ± 15 min	6 h
2	18 h ± 15 min	1 day
3	1 day and 6 h ± 45 min	2 d and 6 h
4	1 day and 18 h ± 75 min	4 d
5	5 d ± 75 min	9 d
6	7 d ± 75 min	16 d
7	20 d ± 7 h	36 d
8	28 d ± 12 h	64 d

[a] Step 1 is described in A.6.2.

NOTE 1 The liquid renewal frequency is designed and carried out such that the concentration in solution does not affect the release rate from the test portion significantly, because the concentration in the solution is far below the saturation concentration.

NOTE 2 If the full duration of the test is not required (e.g. for factory production control) a test consisting of less than eight steps can be performed as ‘indirect test’ as explained in the Introduction.

A.7 Data handling and reporting

The further steps of eluate preparation for analysis, blanks, evaluation of measurement results and reporting are the same as for the DSLT (see 9.5, 9.6, Clause 10 and Clause 11).

Annex B
(normative)

Assessment of release mechanisms (if required)

B.1 Overview of release mechanisms

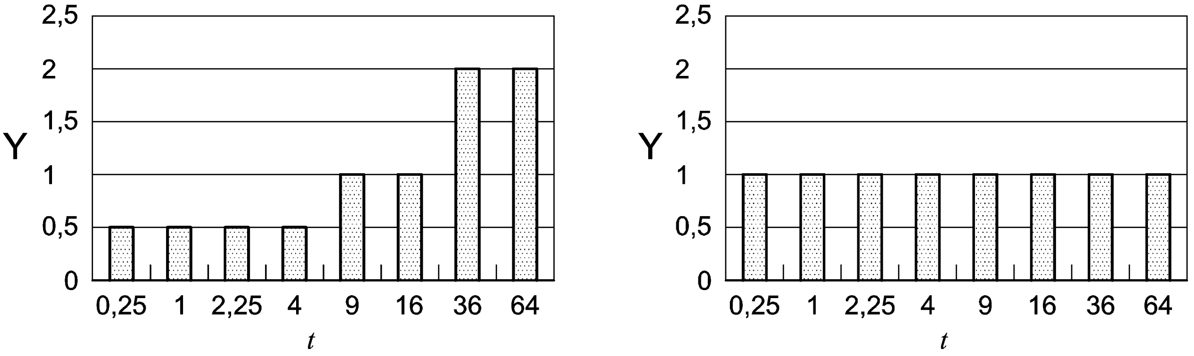
(1) Prediction of the long-term release of dangerous substances is important with respect to the evaluation of the release behaviour of a construction product during its service life in a construction. In order to be able to predict the long-term release by extrapolation, the release mechanism needs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

(2) In general, two main release mechanisms may be distinguished:

a) diffusion;

b) dissolution.

(3) Diffusion-controlled release has a linear relation with √t (time). This allows the renewal times to be chosen in such a way that a distinct pattern may be observed for diffusion-controlled release. The set of renewal times in this Technical Specification (see Table 1) produce a 3-step pattern. The concentration level of the second and third step pattern is twice the level of the previous step pattern. For dissolution-controlled release, the pattern is uniform and independent of the renewal times. The differences in the release pattern are shown in Figure B.1. The characteristics of the release pattern are used to identify the release mechanism.



Key

Y	standardized concentration
t	time in days

Figure B.1 — Release pattern of diffusion (left) and dissolution (right)

(4) In practice, the release mechanism of a construction product tends to be controlled by either one of these mechanisms or a combination. As solubility is controlled by pH the dissolution pattern may be affected by pH-changes during the test. As a result, the dissolution pattern may be less clear in practice than the diffusion pattern. Therefore the analysis of the DSLT result's primary focus is on whether the release is controlled by diffusion. If diffusion is not established it is investigated whether dissolution occurs. As dissolution is pH dependent, dissolution may only be established if pH changes during the test remain limited. In the case of substantial pH changes, dissolution cannot be established and it is concluded that release is controlled by an unidentified mechanism (which will be pH-dependent dissolution or a combination of diffusion and dissolution).

(5) If diffusion is found to be the main release mechanism it is possible to identify surface wash-off and/or depletion as possible secondary mechanisms which influence the long-term release. Under certain conditions (limited pH change), surface wash-off may also be identified in combination with an unidentified release mechanism.

(6) The product's release mechanism information shall be incorporated in the reported result. This might be required

a) to characterize the release performance;

b) to link test results from a standard test duration (normally 64 d) to shortened tests and to historic data from existing comparable tank test procedures with other duration schedules.

NOTE It is worth noting 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.

B.2 Procedure of identification of release mechanisms

The identification of the release mechanism of a substance comprises the following steps.

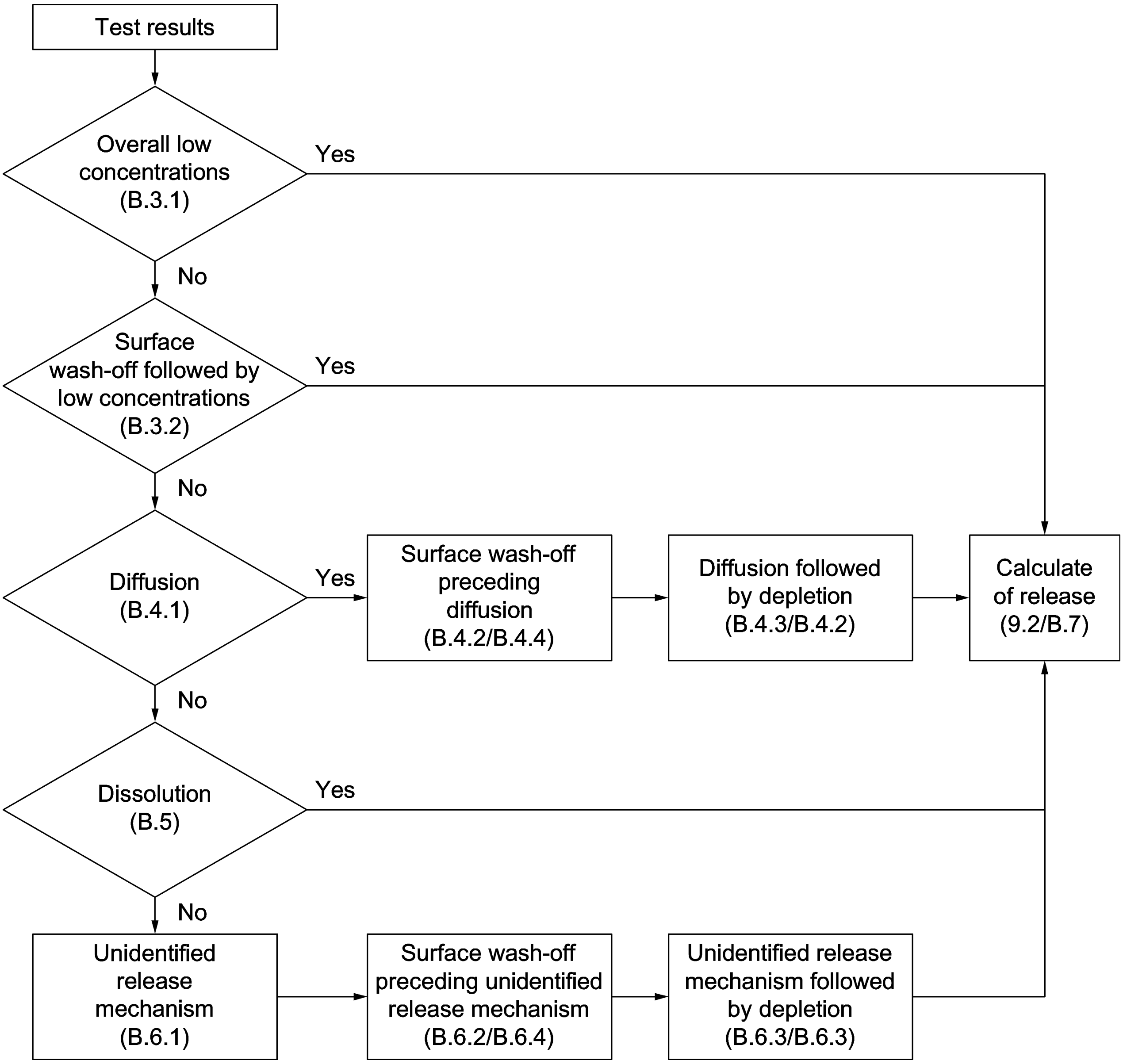


Figure B.2 — Summary of the procedure for identifying the release mechanism and specifying the level of release

B.3 Concentrations close to the limit of quantification

B.3.1 Overall low concentrations

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

$$\frac{c_{2\text{-}8}}{\text{detection limit } [\mu\text{g/l}]} < 1{,}5 \quad . \qquad \text{(B.1)}$$

where

$c_{2-8} = \frac{\sum_{i=2}^{8} c_i}{7}$ is the average concentration of the substance in eluate 2 to 8, in µg/l;

c_i is the concentration of the substance in eluate i, in µg/l.

(2) If for a specific substance concentrations are too low, sections B.3.2, B.4, B.5 and B.6 shall be skipped for this substance.

B.3.2 Surface wash-off followed by low concentrations

(1) If the check on overall low concentrations for a substance is negative, it is checked if initial surface wash-off has occurred followed by low concentrations. Surface wash-off is a form of dissolution giving a relatively high initial release in the first (and second) eluate followed by substantially lower concentrations in the following eluates. If the concentrations of a substance in the following eluates are overall low (B.1), it is not possible to determine the main release mechanism of this substance.

(2) Surface wash-off followed by low concentrations is established if:

$$\frac{c_1}{c_{3-7}} > 1{,}8 \qquad \text{(B.2)}$$

and

$$\frac{c_{5-8}}{\text{detection limit } [\mu\text{g/l}]} < 1{,}5 \qquad \text{(B.3)}$$

where

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

$c_{5-8} = \frac{\sum_{i=5}^{8} c_i}{4}$ is the average concentration of the substance in eluate 5 to 8, in µg/l;

c_i is the concentration of the substance in eluate i, in µg/l.

(3) If for a specific substance surface wash-off is followed by low concentrations, sections B.4, B.5 and B.6 shall be skipped for this substance.

B.4 Diffusion controlled release of a substance

B.4.1 Identification of diffusion controlled release

(1) If overall low concentrations or surface wash-off followed by low concentrations are not established it is checked if diffusion may be the main release mechanism. The rate of diffusion depends on the porosity and pore structure (tortuosity) of the matrix. Diffusion is established if:

$$\sqrt{MSE} < 0{,}40 \tag{B.4}$$

where

MSE is the mean square error of the concentration in eluates 2 to 7 or 8.

(2) The mean square error MSE is calculated as follows.

If $\frac{c_8}{c_7} \geq 0{,}9$ (no depletion):	If $\frac{c_8}{c_7} < 0{,}9$ (depletion):
$c_{2-8} = \frac{\sum_{i=2}^{8} c_\mathrm{i}}{7}$,	$c_{2-7} = \frac{\sum_{i=2}^{7} c_\mathrm{i}}{6}$,
$SE_2 = \left(\frac{c_2}{c_{2-8}} - 0{,}467\right)^2$,	$SE_2 = \left(\frac{c_2}{c_{2-7}} - 0{,}545\right)^2$,
$SE_3 = \left(\frac{c_3}{c_{2-8}} - 0{,}467\right)^2$,	$SE_3 = \left(\frac{c_3}{c_{2-7}} - 0{,}545\right)^2$,
$SE_4 = \left(\frac{c_4}{c_{2-8}} - 0{,}467\right)^2$,	$SE_4 = \left(\frac{c_4}{c_{2-7}} - 0{,}545\right)^2$,
$SE_5 = \left(\frac{c_5}{c_{2-8}} - 0{,}933\right)^2$,	$SE_5 = \left(\frac{c_5}{c_{2-7}} - 1{,}091\right)^2$,
$SE_6 = \left(\frac{c_6}{c_{2-8}} - 0{,}933\right)^2$,	$SE_6 = \left(\frac{c_6}{c_{2-7}} - 1{,}091\right)^2$,
$SE_7 = \left(\frac{c_7}{c_{2-8}} - 1{,}867\right)^2$,	$SE_7 = \left(\frac{c_7}{c_{2-7}} - 2{,}182\right)^2$,
$SE_8 = \left(\frac{c_8}{c_{2-8}} - 1{,}867\right)^2$,	
$MSE = \frac{\sum_{i=2}^{8} SE_\mathrm{i}}{7}$.	$MSE = \frac{\sum_{i=2}^{7} SE_\mathrm{i}}{6}$.

where

c_i is the concentration of the substance in eluate *i*, in µg/l;

SE_i is the square error of the concentration of the substance in eluate *i*;

c_{2-n} is the average concentration of the substance in eluate 2 to *n* (n = 7 or 8), in µg/l.

(3) If for a specific substance diffusion is identified as the main release mechanism, it is checked whether surface wash-off and/or depletion may be present as a secondary effect for this substance. Surface wash-off is checked according to B.4.2 and depletion is checked according to B.4.3.

(4) If for a specific substance diffusion is established as the main release mechanism, it is checked whether surface-wash-off and/or depletion occurs according to B.4.2, B.4.3 and B.4.4, while B.5 and B.6 shall be skipped for this substance.

B.4.2 Surface wash-off preceding diffusion-controlled release

Surface wash-off is characterized by a relatively high concentration level in the first one or two fractions. If diffusion is identified as the main release mechanism diffusion preceded by surface wash-off occurs if:

$$\frac{c_1}{c_{3-4}} > 1{,}8 \tag{B.5}$$

where

$c_{3-4} = \frac{\sum_{i=3}^{4} c_\mathrm{i}}{2}$ is the average concentration of the substance in eluate 3 to 4, in µg/l;

c_i is the concentration of the substance in eluate *i*, in µg/l.

B.4.3 Diffusion-controlled release followed by depletion

Depletion is a function of the porosity, tortuosity and the interaction of the constituent of interest with the solid matrix and/or other substances in the eluate. Depletion may be identified by a decrement of the concentration level in the last fraction(s). If diffusion is identified as the main release mechanism, diffusion followed by depletion occurs if:

$$\frac{c_8}{c_7} < 0{,}9 \tag{B.6}$$

where

c_i is the concentration of the substance in eluate *i*, in µg/l.

B.4.4 Surface wash-off preceding diffusion-controlled release followed by depletion

If diffusion is identified as the main release mechanism, surface wash-off preceding diffusion followed by depletion occurs if both conditions of B.4.2 and B.4.3 are satisfied.

B.5 Dissolution controlled release of a substance

(1) If diffusion is not established as the main release mechanism, it is checked whether dissolution may be the main release mechanism of the substance. Dissolution occurs if:

$$\sigma_\mathrm{pH} < 0{,}25 \tag{B.7}$$

and

$$\frac{\sigma_{\mathrm{c}}}{c_{1-8}} < 0{,}25 \qquad \text{(B.8)}$$

where

$\sigma_{\mathrm{pH}} = \sqrt{\dfrac{\sum_{i=1}^{8} \left(pH_{\mathrm{i}} - pH_{1-8}\right)^2}{8}}$	is the standard deviation of the pH of eluates 1 to 8;
$pH_{1-8} = \dfrac{\sum_{i=1}^{8} pH_{\mathrm{i}}}{8}$	is the average pH of eluates 1 to 8, in µg/l;
pH_{i}	is the pH of eluate *i*;
$\sigma_{\mathrm{c}} = \sqrt{\dfrac{\sum_{i=1}^{8} \left(c_{\mathrm{i}} - c_{1-8}\right)^2}{8}}$	is the standard deviation of the concentrations of the substance in eluates 1 to 8;
$c_{1-8} = \dfrac{\sum_{i=1}^{8} c_{\mathrm{i}}}{8}$	is the average concentration of the substance in eluate 1 to 8, in µg/l;
c_{i}	is the concentration of the substance in eluate *i*, in µg/l.

(2) If for a specific substance dissolution is established as the main release mechanism, B.6 shall be skipped for this substance.

(3) As pH-changes during the test may affect the release of a substance, dissolution may only be identified if there are only small differences in the pH value of the eluates.

B.6 Other release mechanism

B.6.1 Unidentified release mechanism

If neither diffusion nor dissolution is established as the main release mechanism, the release mechanism of that substance remains unidentified.

B.6.2 Surface wash-off of a substance

(1) In the case of an unidentified release mechanism, it is checked whether surface wash-off influences the release of the substance. Surface wash-off is established if:

$$\left|pH_1 - pH_{2-8}\right| < 0{,}5 \qquad \text{(B.9)}$$

and

$$\frac{c_1}{c_{2-4}} > 1{,}8 \qquad \text{(B.10)}$$

where

$pH_{2-8} = \frac{\sum_{i=2}^{8} pH_{\mathrm{i}}}{7}$ is the average pH value of eluates 2 to 8;

pH_{i} is the pH value of eluate i;

$c_{2-4} = \frac{\sum_{i=2}^{4} c_{\mathrm{i}}}{3}$ is the average concentration of the substance in eluate 2 to 4, in µg/l;

c_{i} is the concentration of the substance in eluate *i*, in µg/l.

(2) As pH-changes may affect the release of a substance, surface wash-off can only be identified if the difference in pH value between the first eluate and the following eluates is limited.

B.6.3 Depletion of a substance

(1) If the release mechanism of a substance is not influenced by the pH of the eluate or if pH changes during the test remain limited and the release mechanism remains unidentified, it is also checked whether depletion influences the release of the substance.

(2) Some substances are not sensitive for pH changes of the eluate during the test, e.g. bromide and chloride. Depletion for inert substances is established if:

$$\frac{c_8}{c_7} < 0{,}9 \qquad \text{(B.11)}$$

and

$$\frac{c_6}{c_5} < 0{,}9 \qquad \text{(B.12)}$$

For other substances, depletion is established if:

$$\sigma_{\mathrm{pH}} < 0{,}25 \qquad \text{(B.13)}$$

and

$$\frac{c_8}{c_7} < 0{,}9 \qquad \text{(B.14)}$$

and

$$\frac{c_6}{c_5} < 0{,}9 \qquad \text{(B.15)}$$

where

c_i is the concentration of the substance in eluate *i*, in µg/l;

$\sigma_\text{pH} = \sqrt{\dfrac{\sum_{i=1}^{8}\left(pH_\text{i} - pH_{1\text{–}8}\right)^2}{8}}$ is the standard deviation of the pH of eluates 1 to 8;

$pH_{1\text{–}8} = \dfrac{\sum_{i=1}^{8} pH_\text{i}}{8}$ is the average pH of eluates 1 to 8, in µg/l;

pH_i is the pH of eluate *i*.

(3) As pH-changes during the test may affect the release of a substance, depletion may only be identified if there are only small differences in the pH value of the eluates.

B.6.4 Surface wash-off and depletion

In the case of an unidentified mechanism, surface wash-off and depletion occur if both conditions B.6.2 and B.6.3 are satisfied.

B.7 Calculation of release

B.7.1 Area release

See 10.2 for the calculation of the area release r_i and cumulative area release R for each period.

B.7.2 Surface wash-off

If surface wash-off precedes any of the mechanisms, the release by surface wash-off equals:

$$R_\text{SWO} = R_2 - r_3 - r_4 \qquad \text{(B.16)}$$

where

R_swo is the cumulative area release of the substance by surface wash-off, in mg/m^2;

R_2 is the cumulative area release of the substance for period 2, in mg/m^2 (see 10.2);

r_3 is the area release of the substance for fraction 3, in mg/m^2 (see 10.2);

r_4 is the area release of the substance for fraction 4, in mg/m^2 (see 10.2).

B.7.3 Release in the case of dissolution

In the case of dissolution the cumulative release, R_n shall be multiplied by 2.

NOTE The multiplication factor of 2 is a correction for the long-term leaching behaviour of the construction product. Dissolution leads to a higher release in time compared to diffusion.

B.7.4 Extrapolation of the cumulative area release for larger time values

A proper understanding of the release mechanism of a substance is of importance to be able to make predictions on long term release from monolithic, plate- or sheet-like construction products. For the different mechanisms, formulae for extrapolation of the cumulative area release are given in Table B.1.

NOTE The use of these formulae implies that the pH of the product matrix does not largely change in time. For some construction products this may not be the case as carbonation or redox changes may influence the pH of the product in time.

Table B.1 — Calculation of the area release for time values greater than 64 d

Release mechanism	Section	Formula for extrapolation	
Overall low concentrations	B.3.1	$R_{T_E} = R_8 \sqrt{\frac{T_E}{64}}$	(B.17)
Surface wash-off followed by low concentrations	B.3.2	$R_{T_E} = R_2 + (R_8 - R_2) \frac{\sqrt{T_E} - \sqrt{1}}{\sqrt{64} - \sqrt{1}}$	(B.18)
Diffusion	B.4.1	$R_{T_E} = R_8 \sqrt{\frac{T_E}{64}}$	(B.19)
Diffusion preceded by surface wash-off	B.4.2	$R_{T_E} = R_2 + (R_8 - R_2) \frac{\sqrt{T_E} - \sqrt{1}}{\sqrt{64} - \sqrt{1}}$	(B.18)
Diffusion (preceded by surface wash-off and) followed by depletion	B.4.3 and B.4.4	$R_{T_E} = R_7 + (R_8 - R_7) \cdot \frac{\sqrt{T_E} - \sqrt{36}}{\sqrt{64} - \sqrt{36}}$	(B.20)
Dissolution	B.5	$R_{T_E} = 2 \cdot R_8 \sqrt{\frac{T_E}{64}}$	(B.21)
Unidentified mechanism	B.6.1	$R_{T_E} = R_8 \sqrt{\frac{T_E}{64}}$	(B.22)
Unidentified mechanism preceded by surface wash-off	B.6.2	$R_{T_E} = R_2 + (R_8 - R_2) \frac{\sqrt{T_E} - \sqrt{1}}{\sqrt{64} - \sqrt{1}}$	(B.18)
Unidentified mechanism (preceded by surface wash-off and) followed by depletion	B.6.3 and B.6.4	$R_{T_E} = R_7 + (R_8 - R_7) \cdot \frac{\sqrt{T_E} - \sqrt{36}}{\sqrt{64} - \sqrt{36}}$	(B.23)

where

R_{T_E} is the extrapolated release over T_E days, in mg/m^2;

R_{swo} is the cumulative area release of the substance by surface wash-off, in mg/m^2;

R_7 is the cumulative area release over period 7, in mg/m^2;

R_8 is the cumulative area release over period 8, in mg/m^2;

T_E is the time value for extrapolation, in days.

B.8 Examples

The following pages show examples of different release mechanisms.

EXAMPLE 1 Surface wash-off preceding diffusion controlled release.

Eluate data	
c_1 [µg/l]	2800
c_2 [µg/l]	940
c_3 [µg/l]	540
c_4 [µg/l]	470
c_5 [µg/l]	1300
c_6 [µg/l]	1300
c_7 [µg/l]	2100
c_8 [µg/l]	2300

pH_1	10,40
pH_2	10,80
pH_3	10,85
pH_4	11,15
pH_5	11,25
pH_6	11,30
pH_7	11,60
pH_8	11,55

Detection limit (DL)	100

Sulphate

Overall low concentrations			
$c_{2–8}$	1 300	$c_{2–8}/DL$	13,0
$c_{2–8}/DL < 1{,}5$	no		

Surface wash-off followed by low concentrations			
$c_{3–7}$	1 100	$c_1/c_{3–7}$	2,55
$c_{5–8}$	1 800	$c_{5–8}/DL$	18,0
$c_1/c_{3–7} > 1{,}8$ and $c_{5–8}$/DL < 1,5	no		

Diffusion			
c_8/c_7	1,10		
$c_8/c_7 < 0{,}9$	no		
$c_{2–8}$	1300		
SE_2	0,072	Criterion SE_2 to SE_4	0,467
SE_3	0,002	Criterion SE_5 to SE_6	0,933
SE_4	0,010	Criterion SE_7 to SE_8	1,867
SE_5	0,007		
SE_6	0,007		
SE_7	0,050		
SE_8	0,005		
$RMSE$	0,148		
$RMSE < 0{,}40$	yes		

Surface wash-off preceding diffusion	
$c_{3–4}$	510
$c_1/c_{3–4} > 1{,}8$	yes

Diffusion followed by depletion	
$c_8/c_7 < 0{,}9$	no

Dissolution	
σ_{pH}	
$\sigma_{\mathrm{pH}} < 0{,}25$	
σ_{c}	
$c_{1–8}$	
$\sigma_{\mathrm{c}}/c_{1–8} < 0{,}25$	

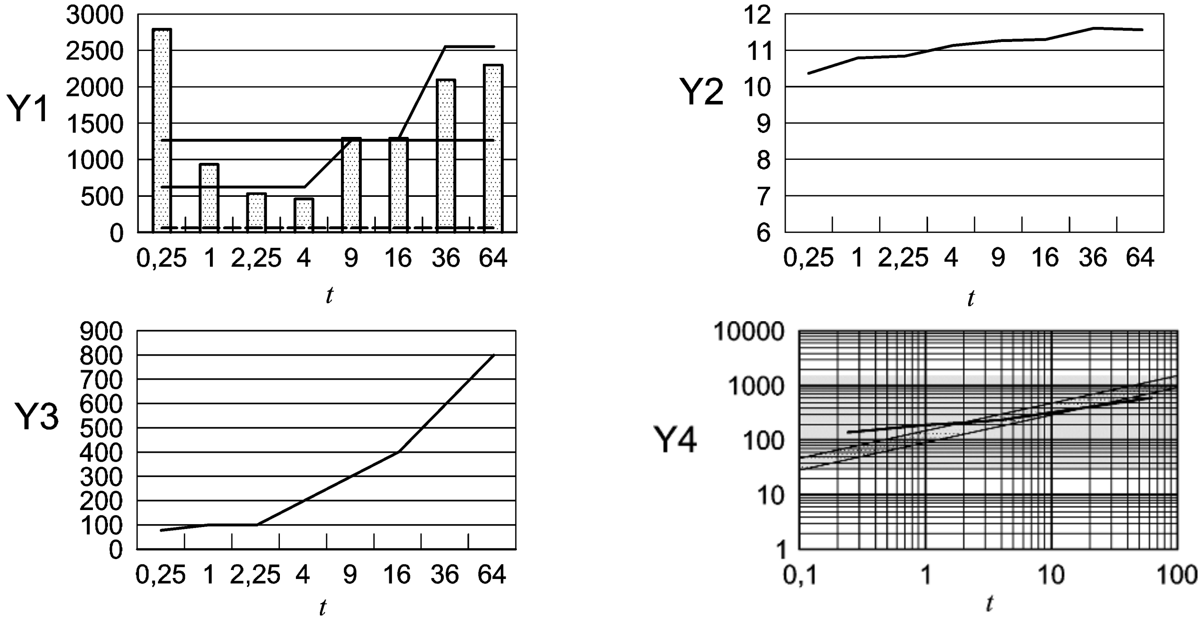
Surface wash-off preceding unidentified mechanism	
ΔpH_1	
$\Delta pH_1 < 0{,}50$	
$c_1/c_{3–4} > 1{,}8$	

Unidentified mechanism followed by depletion	
Inert substance	no
c_6/c_5	
$c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	
$\sigma_{\mathrm{pH}} < 0{,}25$ und $c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	

Conclusion:	Surface wash-off preceding diffusion controlled release

Calculations					
Area test specimen (A)	0,505 1	[m^2]	$m_{\mathrm{s}1-2}$	3	[g]
Volume eluate (V)	25,254	[l]	$m_{\mathrm{s}3-8}$	2	[g]
r_1	140	[mg/m^2]	R_1	140	[mg/m^2]
r_2	47	[mg/m^2]	R_2	190	[mg/m^2]
r_3	27	[mg/m^2]	R_3	210	[mg/m^2]
r_4	23	[mg/m^2]	R_4	240	[mg/m^2]
r_5	65	[mg/m^2]	R_5	300	[mg/m^2]
r_6	65	[mg/m^2]	R_6	370	[mg/m^2]
r_7	100	[mg/m^2]	R_7	470	[mg/m^2]
r_8	110	[mg/m^2]	R_8	590	[mg/m^2]
R_{swo}	140	[mg/m^2]			
m_{a}	9,9	[g/m^2]			

Cumulative release over 64 d:	590	[mg/m^2]



Key

Y1	concentration in mg/l
Y2	pH
Y3	electrical conductivity in µS/cm
Y4	cumulative area release rate in mg/m^2
t	time in days

Figure B.3

EXAMPLE 2 Surface wash-off followed by low concentrations

Eluate data	
c_1 [µg/l]	150
c_2 [µg/l]	17
c_3 [µg/l]	14
c_4 [µg/l]	28
c_5 [µg/l]	< 10
c_6 [µg/l]	< 10
c_7 [µg/l]	14
c_8 [µg/l]	22

pH_1	8,35
pH_2	9,15
pH_3	9,30
pH_4	9,30
pH_5	8,88
pH_6	9,30
pH_7	8,75
pH_8	8,70

Detection limit (DL)	10

Bromide

Overall low concentrations			
$c_{2\text{–}8}$	16	$c_{2\text{–}8}/DL$	1,6
$c_{2\text{–}8}/DL < 1,5$	no		

Surface wash-off followed by low concentrations			
$c_{3\text{–}7}$	15	$c_1/c_{3\text{–}7}$	10,00
$c_{5\text{–}8}$	14	$c_{5\text{–}8}/DL$	1,4
$c_1/c_{3\text{–}7} > 1,8$ and $c_{5\text{–}8}/DL < 1,5$	yes		

Diffusion	
c_8/c_7	
$c_8/c_7 < 0,9$	
$c_{2\text{–}8}$	
SE_2	Criterion SE_2 to SE_4
SE_3	Criterion SE_5 to SE_6
SE_4	Criterion SE_7 to SE_8
SE_5	
SE_6	
SE_7	
SE_8	
$RMSE$	
$RMSE < 0,40$	

Surface wash-off preceding diffusion
$c_{3\text{–}4}$
$c_1/c_{3\text{–}4} > 1,8$

Diffusion followed by depletion
$c_8/c_7 < 0,9$

Dissolution
σ_{pH}
$\sigma_{\text{pH}} < 0,25$
σ_{c}
$c_{1\text{–}8}$
$\sigma_{\text{c}}/c_{1\text{–}8} < 0,25$

Surface wash-off preceding unidentified mechanism

ΔpH_1

$\Delta pH_1 < 0{,}50$

$c_1/c_{3–4} > 1{,}8$

Unidentified mechanism followed by depletion

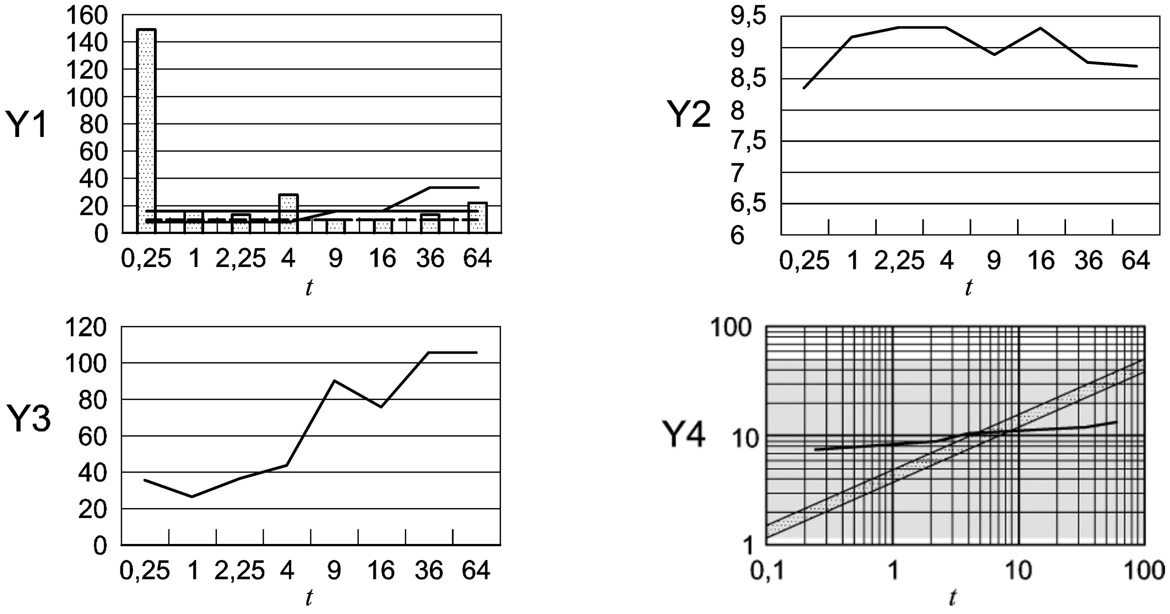
Inert substance	yes
c_6/c_5	
$c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	
$\sigma_{\mathrm{pH}} < 0{,}25$ und $c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	

Conclusion: Surface wash-off followed by low concentrations

Calculations

Area test specimen (A)	0,039 8	[m^2]	$m_{s1–2}$	0,5	[g]
Volume eluate (V)	2,007	[l]	$m_{s3–8}$	0,3	[g]
r_1	7,6	[mg/m^2]	R_1	7,6 – 7,6	[mg/m^2]
r_2	0,86	[mg/m^2]	R_2	8,4 – 8,4	[mg/m^2]
r_3	0,71	[mg/m^2]	R_3	9,1 – 9,1	[mg/m^2]
r_4	1,4	[mg/m^2]	R_4	11 – 11	[mg/m^2]
r_5	0 – 0,5	[mg/m^2]	R_5	11 – 11	[mg/m^2]
r_6	0 – 0,5	[mg/m^2]	R_6	11 – 12	[mg/m^2]
r_7	0,71	[mg/m^2]	R_7	11 – 12	[mg/m^2]
r_8	1,1	[mg/m^2]	R_8	12 – 13	[mg/m^2]
R_{swo}	6,3	[mg/m^2]			
m_a	20	[g/m^2]			

Cumulative release over 64 d: 12 – 13 [mg/m^2]



Key

Y1	concentration in mg/l
Y2	pH
Y3	electrical conductivity in µS/cm
Y4	cumulative area release rate in mg/m^2
t	time in days

Figure B.4

EXAMPLE 3 Diffusion controlled release

Eluate data	
c_1 [µg/l]	240
c_2 [µg/l]	220
c_3 [µg/l]	240
c_4 [µg/l]	250
c_5 [µg/l]	440
c_6 [µg/l]	390
c_7 [µg/l]	730
c_8 [µg/l]	720

pH_1	9,40
pH_2	9,30
pH_3	9,60
pH_4	9,80
pH_5	10,30
pH_6	10,50
pH_7	10,80
pH_8	11,10

Detection limit (DL)	10

Vanadium

Overall low concentrations

$c_{2–8}$	430	$c_{2–8}/DL$	43,0
$c_{2–8}/DL < 1,5$	no		

Surface wash-off followed by low concentrations

$c_{3–7}$	410	$c_1/c_{3–7}$	0,59
$c_{5–8}$	570	$c_{5–8}/DL$	57,0
$c_1/c_{3–7} > 1,8$ and $c_{5–8}/DL < 1,5$	no		

Diffusion

c_8/c_7	0,99		
$c_8/c_7 < 0,9$	no		
$c_{2–8}$	430		
SE_2	0,002	Criterion SE_2 to SE_4	0,467
SE_3	0,009	Criterion SE_5 to SE_6	0,933
SE_4	0,014	Criterion SE_7 to SE_8	1,867
SE_5	0,009		
SE_6	0,000		
SE_7	0,025		
SE_8	0,033		
$RMSE$	0,115		
$RMSE < 0,40$	yes		

Surface wash-off preceding diffusion

$c_{3–4}$	250
$c_1/c_{3–4} > 1,8$	no

Diffusion followed by depletion

$c_8/c_7 < 0,9$	no

Dissolution

σ_{pH}	
$\sigma_{pH} < 0,25$	
σ_c	
$c_{1–8}$	
$\sigma_c/c_{1–8} < 0,25$	

Surface wash-off preceding unidentified mechanism

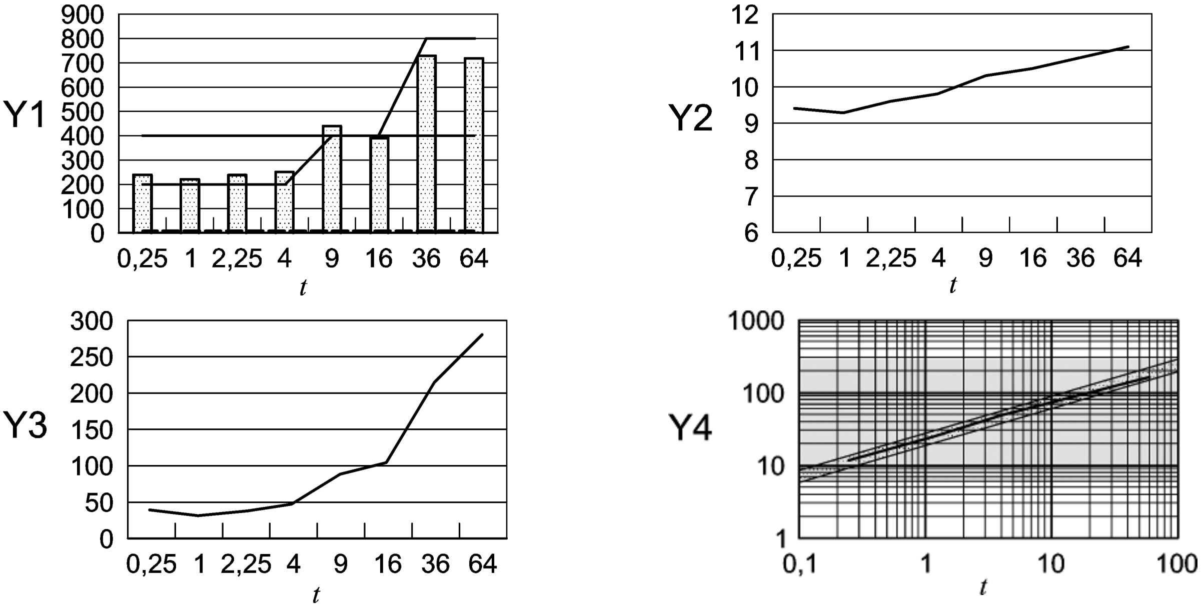
ΔpH_1	
$\Delta pH_1 < 0,50$	
$c_1/c_{3–4} > 1,8$	

Unidentified mechanism followed by depletion	
Inert substance	no
c_6/c_5	
$c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	
$\sigma_{\mathrm{pH}} < 0{,}25$ und $c_8/ c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	

Conclusion:	diffusion controlled release

Calculations					
Area test specimen (A)	0,457 0	[m^2]	$m_{\mathrm{s1-2}}$	0,5	[g]
Volume eluate (V)	22,850	[l]	$m_{\mathrm{s3-8}}$	0,5	[g]
r_1	12	[mg/m^2]	R_1	12	[mg/m^2]
r_2	11	[mg/m^2]	R_2	23	[mg/m^2]
r_3	12	[mg/m^2]	R_3	35	[mg/m^2]
r_4	13	[mg/m^2]	R_4	48	[mg/m^2]
r_5	22	[mg/m^2]	R_5	70	[mg/m^2]
r_6	20	[mg/m^2]	R_6	89	[mg/m^2]
r_7	37	[mg/m^2]	R_7	130	[mg/m^2]
r_8	36	[mg/m^2]	R_8	160	[mg/m^2]
R_{swo}	0	[mg/m^2]			
m_{a}	2,2	[g/m^2]			

Cumulative release over 64 d:	160	[mg/m^2]



Key

Y1	concentration in mg/l
Y2	pH
Y3	electrical conductivity in µS/cm
Y4	cumulative area release rate in mg/m^2
t	time in days

Figure B.5

EXAMPLE 4 Dissolution controlled release

Eluate data	
c_1 [µg/l]	1 000
c_2 [µg/l]	1 100
c_3 [µg/l]	1 000
c_4 [µg/l]	1 000
c_5 [µg/l]	1 100
c_6 [µg/l]	1 200
c_7 [µg/l]	1 400
c_8 [µg/l]	1 100

pH_1	9,60
pH_2	9,75
pH_3	9,80
pH_4	9,70
pH_5	9,75
pH_6	9,80
pH_7	9,85
pH_8	9,70

Detection limit (DL)	100

Fluoride

Overall low concentrations			
c_{2-8}	1 100	c_{2-8}/DL	11,0
$c_{2-8}/DL < 1{,}5$	no		

Surface wash-off followed by low concentrations			
c_{3-7}	1 100	c_1/c_{3-7}	0,91
c_{5-8}	1 200	c_{5-8}/DL	12,0
$c_1/c_{3-7} > 1{,}8$ and $c_{5-8}/DL < 1{,}5$	no		

Diffusion			
c_8/c_7	0,79		
$c_8/c_7 < 0{,}9$	yes		
c_{2-8}	1100		
SE_2	0,181	Criterion SE_2 to SE_4	0,545
SE_3	0,114	Criterion SE_5 to SE_6	1,091
SE_4	0,114	Criterion SE_7 to SE_8	2,182
SE_5	0,014		
SE_6	0,001		
SE_7	0,896		
SE_8			
$RMSE$	0,469		
$RMSE < 0{,}40$	no		

Surface wash-off preceding diffusion	
c_{3-4}	
$c_1/c_{3-4} > 1{,}8$	

Diffusion followed by depletion	
$c_8/c_7 < 0{,}9$	

Dissolution	
σ_{pH}	0,07
$\sigma_{\mathrm{pH}} < 0{,}25$	yes
σ_{c}	130
c_{1-8}	1 100
$\sigma_{\mathrm{c}}/c_{1-8} < 0{,}25$	yes

Surface wash-off preceding unidentified mechanism

ΔpH_1

$\Delta pH_1 < 0{,}50$

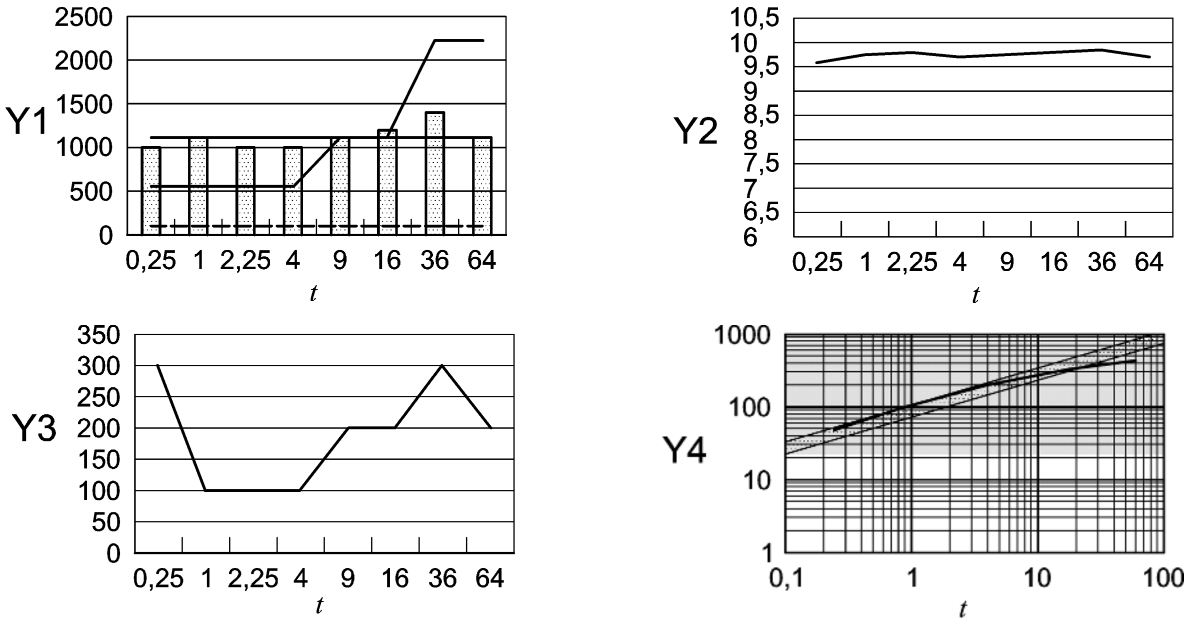
$c_1/c_{3\text{–}4} > 1{,}8$

Unidentified mechanism followed by depletion	
Inert substance	no
c_6/c_5	
$c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	
$\sigma_{\mathrm{pH}} < 0{,}25$ und $c_8/c_7 < 0{,}90$ and $c_6/c_5 < 0{,}90$	

Conclusion: dissolution controlled release

Calculations					
Area test specimen (A)	0,369 0	[m^2]	$m_{\mathrm{s1–2}}$	4	[g]
Volume eluate (V)	18,450	[l]	$m_{\mathrm{s3–8}}$	2	[g]
r_1	50	[mg/m^2]	R_1	50	[mg/m^2]
r_2	55	[mg/m^2]	R_2	110	[mg/m^2]
r_3	50	[mg/m^2]	R_3	160	[mg/m^2]
r_4	50	[mg/m^2]	R_4	210	[mg/m^2]
r_5	55	[mg/m^2]	R_5	260	[mg/m^2]
r_6	60	[mg/m^2]	R_6	320	[mg/m^2]
r_7	70	[mg/m^2]	R_7	390	[mg/m^2]
r_8	55	[mg/m^2]	R_8	450	[mg/m^2]
R_{swo}	0	[mg/m^2]			
m_{a}	16	[g/m^2]			

Cumulative release over 64 d: 890 [mg/m^2]



Key

Y1 concentration in mg/l
Y2 pH
Y3 electrical conductivity in µS/cm
Y4 cumulative area release rate in mg/m^2
t time in days

Figure B.6 - Interpretation of shortened DSL-Tests for FPC

As the cumulative release over 64 d is taken as standard to characterize the release of a substance, data which have been obtained with shortened DSL tests need to be corrected. Formulae for the extrapolation of shortened test data to 64 d are given in Table B.2. The validity of the formulae for the tested material should be checked with the reference test.

Table B.2 - Calculation of the area release for shortened DSL tests

Release mechanism	Time (days)	Formula for extrapolating shortened test data to 64 d
Overall low concentrations Diffusion Unidentified mechanism	2,25	$R_8 = R_3\sqrt{\frac{64}{2.25}} = R_3 \times 5{,}33$
	4	$R_8 = R_4\sqrt{\frac{64}{4}} = R_3 \times 4$
	9	$R_8 = R_5\sqrt{\frac{64}{9}} = R_5 \times 2{,}67$
	16	$R_8 = R_6\sqrt{\frac{64}{16}} = R_3 \times 2$
	36	$R_8 = R_7\sqrt{\frac{64}{36}} = R_7 \times 1{,}33$

Release mechanism	**Time (days)**	**Formula for extrapolating shortened test data to 64 d**
Surface wash-off followed by low concentrations	2,25	$R_8 = R_2 + (R_3 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{2.25}-\sqrt{1}} = R_2 + (R_3 - R_2)\times 14$
	4	$R_8 = R_2 + (R_4 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{4}-\sqrt{1}} = R_2 + (R_4 - R_2)\times 7$
	9	$R_8 = R_2 + (R_5 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{9}-\sqrt{1}} = R_2 + (R_5 - R_2)\times 3{,}5$
	16	$R_8 = R_2 + (R_6 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{16}-\sqrt{1}} = R_2 + (R_6 - R_2)\times 2{,}33$
	36	$R_8 = R_2 + (R_7 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{36}-\sqrt{1}} = R_2 + (R_7 - R_2)\times 1{,}4$
Diffusion preceded by surface wash-off (and followed by depletion) Unidentified mechanism preceded by surface wash-off (and followed by depletion)	2,25	Not applicable
	4	Not applicable
	9	$R_8 = R_2 + (R_5 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{9}-\sqrt{1}} = R_2 + (R_5 - R_2)\times 3{,}5$
	16	$R_8 = R_2 + (R_6 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{16}-\sqrt{1}} = R_2 + (R_6 - R_2)\times 2{,}33$
	36	$R_8 = R_2 + (R_7 - R_2)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{36}-\sqrt{1}} = R_2 + (R_7 - R_2)\times 1{,}4$
Diffusion followed by depletion Unidentified mechanism followed by depletion	2,25	$R_8 = R_3\sqrt{\frac{64}{2.25}} = R_3 \times 5{,}33$
	4	$R_8 = R_4\sqrt{\frac{64}{4}} = R_3 \times 4$
	9	$R_8 = R_5\sqrt{\frac{64}{9}} = R_5 \times 2{,}67$
	16	$R_8 = R_6\sqrt{\frac{64}{16}} = R_3 \times 2$

Release mechanism	Time (days)	Formula for extrapolating shortened test data to 64 d
	36	$R_8 = R_7\sqrt{\frac{64}{36}} = R_7 \times 1{,}33$
Dissolution	2,25	$R_8 = 2 \cdot R_3\sqrt{\frac{64}{2.25}} = R_3 \times 10{,}67$
	4	$R_8 = 2 \cdot R_4\sqrt{\frac{64}{4}} = R_3 \times 8$
	9	$R_8 = 2 \cdot R_5\sqrt{\frac{64}{9}} = R_5 \times 5{,}33$
	16	$R_8 = 2 \cdot R_6\sqrt{\frac{64}{16}} = R_3 \times 4$
	36	$R_8 = 2 \cdot R_7\sqrt{\frac{64}{36}} = R_7 \times 2{,}67$

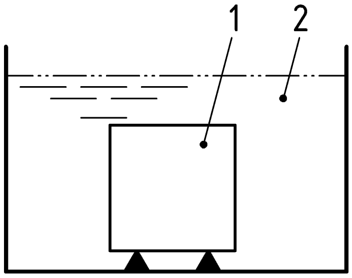
NOTE 1 In the case of a shortened test period of 2,25 d it is not possible to determine R_{swo}. In that specific situation, extrapolation based on the third step only is not likely to give a reliable estimation of the cumulative release after 64 d. Only in the case of low concentrations is such extrapolation possible as surface wash-off dominates the total release.

NOTE 2 The effect of depletion cannot be taken into account and extrapolation is based on the assumption of diffusion up to 64 d.

Annex C
(informative)

Examples of the test set up for construction products (DSLT)

Figure C.1 shows a set up for testing of a cubic concrete test piece.

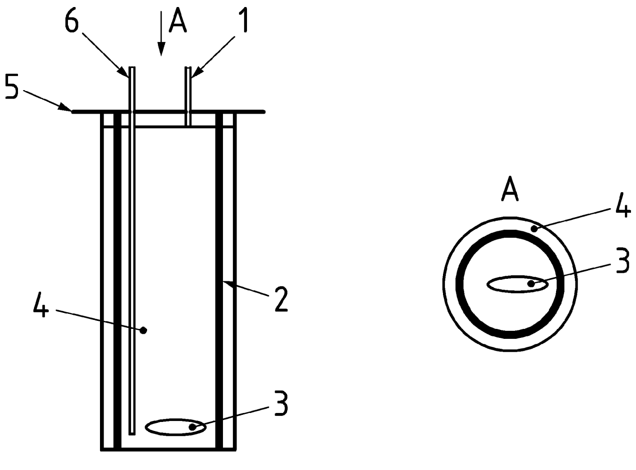


Key

1	concrete cube (surface area 6 dm^2)
2	leachant

Figure C.1 — Set up for testing of a concrete test piece

Figure C.2 represents a method for performing the DSLT on roofing felt which is affixed to the inner vertical walls of a glass cylinder. In this set-up a stirrer is used.



Key

1	nitrogen in and out
2	bitumen coated roofing felt
3	magnetic stirrer
4	glass cylinder
5	glass cover
6	eluate out and clean water in

Figure C.2 — Set up for testing of roofing felt

NOTE For narrow cylinders it might be advantageous to apply occasional stirring by means of a magnetic stirrer, e.g. 15 min per hour.

Annex D
(informative)

Examples of data about the L/A ratio

In the table below for some test pieces (cube, rectangular and cylinder) and the most common dimensions minimum and maximum vessel diameters are calculated, in the case cylindrical vessels are used. The minimum diameter is calculated in order to fulfil the criterion, mentioned in 7.2, stating that the minimum distance between the test portion and the walls should be 20 mm. The maximum diameter is calculated in order to fulfil the criterion, mentioned in 9.2, stating that the top of the test portion should be at least 20 mm submerged.

Table D.1 – Vessel dimensions in relation with test pieces of different sizes and/or dimensions (L/A = 80)

Test portion	**Height**	**Diameter**	**Length**	**Width**	**Volume (V)**	**Surface (A)**	V_{total}	**Vessel diameter range (min. and max.)**	**Minimum distance between vessel and test piece**	**Distance between test piece and water level**
	mm	**mm**	**mm**	**mm**	**cm^3**	**cm^2**	**cm^3**	**mm**	**mm**	**mm**
Cube	100		100	100	1 000	600	5 800	**181**	20	104
		-						**230**	44	20
	40		40	40	64	96	832	**97**	20	54
								115	29	20
Cylinder	100	100			785	471	4 553	**140**	20	176
								204	52	20
Rectangular	50		200	100	1 000	700	6 600	**264**	20	51
		-						**306**	41	20
	160		40	40	256	288	2 560	**97**	20	170
								128	36	20

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1) Under development.

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