BS EN 15863:2015

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# Characterization of waste — Leaching behaviour test for basic characterization — Dynamic monolithic leaching test with periodic leachant renewal, under fixed conditions

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## National foreword

This British Standard is the UK implementation of EN 15863:2015. It supersedes PD CEN/TS 15863:2012 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/508/3, Characterization of waste.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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**Compliance with a British Standard cannot confer immunity from legal obligations.**

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**EN 15863**

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English Version

# Characterization of waste - Leaching behaviour test for basic characterization - Dynamic monolithic leaching test with periodic leachant renewal, under fixed conditions

Caractérisation des déchets - Essais de comportement à la lixiviation pour la caractérisation de base - Essai de lixiviation dynamique des monolithes avec renouvellement périodique du lixiviant, dans des conditions d'essai fixes

Charakterisierung von Abfällen - Untersuchung des Elutionsverhaltens für die grundlegende Charakterisierung - Dynamisches Elutionsverfahren für monolithische Abfälle mit periodischer Erneuerung des Elutionsmittels unter festgelegten Prüfbedingungen

This European Standard was approved by CEN on 7 February 2015.

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

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# Foreword

This document (EN 15863:2015) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2015, and conflicting national standards shall be withdrawn at the latest by October 2015.

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 supersedes CEN/TS 15863:2012.

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

This document was elaborated on the basis of NEN 7345:1995.

The following significant technical changes have been implemented in this new edition of the text:

- the status of the document has been changed from a CEN/TS into a European Standard;
- performance data has been added (see Annex E).

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

# Introduction

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

This document specifies a dynamic leaching test for monolithic waste materials, to determine key parameters to address the leaching behaviour of monolithic waste materials.

For the complete characterization of the leaching behaviour of waste under specified conditions the application of other test methods is required (see EN 12920).

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples can contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction.

Consequently these samples should be handled with special care. Gases which can be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

In the different European countries, tests have been developed to characterize and assess the constituents which can be leached from waste materials. The release of soluble constituents upon contact with water is regarded as one of the main mechanism of release which results in a potential risk to the environment during life-cycle of waste materials (disposal or re-use scenario). The intent of these tests is to identify the leaching properties of waste materials. The complexity of the leaching process makes simplifications necessary. Not all of the relevant aspects of leaching behaviour can be addressed in one single standard.

Procedures to characterize the behaviour of waste materials can generally be divided into three steps, using different tests in relation to the objective. The following test hierarchy is taken from the Landfill Directive 1) and the Decision on Annex II of this Directive 2) for disposal of waste.

a) Basic characterization constitutes a full characterization of the waste by gathering all the necessary information for a safe management of the waste in the short and long term. Basic characterization may provide information on the waste (type and origin, composition, consistency, leachability, etc.), information for understanding the behaviour of waste in the considered management scenario, comparison of waste properties against limit values, and detection of key variables (critical parameters as liquid/solid (L/S) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters) for compliance testing and options for simplification of compliance testing. Characterization may deliver ratios between test results from basic characterization and results from simplified test procedures as well as information on a suitable frequency for compliance testing. In addition to the leaching behaviour, the composition of the waste should be known or determined by testing. The tests used for basic characterization should always include those to be used for compliance testing.

b) Compliance testing is used to demonstrate that the sample of today fits the population of samples tested before by basic characterization and through that, is used to carry out compliance with regulatory limit values. The compliance test should therefore always be part of the basic characterization program. The compliance test focuses on key variables and leaching behaviour identified by basic characterization tests. Parts of basic characterization tests can also be used for compliance purposes.

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1) Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste.

2) Council Decision 2003/33/EC of 19 December 2002.

c) On-site verification tests are used as a rapid check to confirm that the waste is the same as that which has been subjected to characterization or compliance tests. On-site verification tests are not necessarily leaching tests.

The test procedure described in this document is a basic characterization test and falls in category a).

According to EN 12920 the evaluation of the release of constituents from waste materials in a certain scenario involves the performance of various tests. This document describes one of the parametric test that can be used for such purposes, especially for monolithic waste.

The test procedure allows the determination of the release under dynamic conditions of constituents from a monolithic waste material, as a function of time. This release is calculated from the concentrations of the constituents measured in the solution (eluate) that is collected in a fixed number of separate fractions. Besides, the following test conditions are fixed: the type of leachant, the temperature, the liquid to surface area ratio (*L/S*), and the duration of the test.

Results of this test, combined with those from other tests (e.g. EN 14429) and the use of more or less sophisticated models, allow the identification of the main leaching mechanisms that can be distinguished, such as diffusion, dissolution of constituents, initial surface wash-off, dissolution of the matrix (see Annex A). These intrinsic properties can be used to predict the release of constituents at a given time frame, in order to assess the leaching behaviour of monolithic waste materials in practical situations or scenarios as defined in EN 12920. For assessment of the leaching under equilibrium conditions (low $L/A$ condition, pore water and/or long contact times) there are other tools available, such as the pH dependence test EN 14429, the percolation test for granular material CEN/TS 14405 (with L/S 0,1 l/kg to 0,5 l/kg) and the batch test for granular waste EN 12457-1 to EN 12457-4.

NOTE At low $L/A$ conditions, pore water conditions in monolithic specimens can be approached.

## 1 Scope

This European Standard is applicable for determining the leaching behaviour of monolithic wastes under dynamic conditions. The test is performed under fixed experimental conditions in this document. This test is aimed at determining the release as a function of time of inorganic constituents from a monolithic waste, when it is put into contact with an aqueous solution (leachant).

This dynamic monolithic leaching test (DMLT) is a parameter specific test as specified in EN 12920 and is therefore not aimed at simulating real situations. The application of this test method alone is not sufficient for the determination of the detailed leaching behaviour of a monolithic waste under specified conditions.

In the framework of EN 12920 and in combination with additional chemical information, the test results are used to identify the leaching mechanisms and their relative importance. The intrinsic properties can be used to predict the release of constituents at a given time frame, in order to assess the leaching behaviour of monolithic waste materials, placed in different situations or scenarios (including disposal and recycling scenarios).

The test method applies to regularly shaped test portions of monolithic wastes with minimum dimensions of 40 mm in all directions that are assumed to maintain their integrity over a time frame relevant for the considered scenario. The test method applies to test portions for which the geometric surface area can be determined with the help of simple geometric equations. The test method applies to low permeable monolithic materials.

Within the reproducibility ranges, the leaching results obtained with EN 15863 are expected to be equivalent to those obtained with CEN/TS 16637-2 (DMLT for construction products), because the main testing conditions are equalized in both standards. As shown in the results obtained with EN 15863 (see Annex E), they are also demonstrated to be comparable with US EPA method 1315 (SW846). These observations imply that a monolithic waste tested with this European Standard, does not need to be tested a second time, when the material proves suitable for beneficial use in construction and provided it has not undergone a treatment or other changes modifying its leaching behaviour.

NOTE 1 If, in order to comply with the requirements of regular shape, the test portion is prepared by cutting or coring, then new surfaces are exposed which can lead to change(s) in leaching properties. On the other hand if the test portion is prepared by moulding, the surface will be dependent to the type of mould and the conditions of storage. If the intention is to evaluate the behaviour of the material core, the specimen needs to be stored without any contact with air to avoid carbonation.

NOTE 2 For monolithic waste materials with a saturated hydraulic conductivity higher than $10^{-8}$ m/s water is likely to percolate through the monolith rather than flow around. In such cases relating the release to the geometric surface can lead to misinterpretation. A percolation test is more appropriate then (e.g. CEN/TS 14405).

This procedure may not be applicable to materials reacting with the leachant, leading for example to excessive gas emission or an excessive heat release.

This document has been developed to determine the release of mainly inorganic constituents from wastes. It does not take into account the particular characteristics of organic constituents, nor the consequences of microbiological processes in organic degradable wastes.

## 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 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

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)*

## 3 Terms and definitions

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

### 3.1
**eluate**
solution obtained by a leaching test

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

[SOURCE: IUPAC:1990]

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, sawing, coring, 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. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

Note 2 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When it is sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

### 3.3
**leachant**
liquid that is brought into contact with the test portion in the leaching procedure

Note 1 to entry: For the purpose of this document the leachant is water as specified in 5.1.

### 3.4
**leaching behaviour of a waste**
release and change with time in release from the waste upon contact with a *leachant* under the conditions specified in the scenario, especially within the specified time frame

[SOURCE: EN 12920:2006+A1:2008]

### 3.5
**leachant renewal**
selection of time intervals after which the *leachant* is renewed

### 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 monolith, and the surface area of the test portion ($A$)

Note 1 to entry: $L/A$ is expressed in $\mathrm{ml \cdot cm^{-2}}$.

### 3.7
**monolithic waste**

waste which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered scenario

Note 1 to entry: A monolithic stabilized or solidified waste means a waste stabilized by the application of binder(s) to form a coherent body of specified dimensions maintaining its integrity in the landfill over a specified timeframe.

### 3.8
**release**

emission of constituents from a waste, which pass through the external surface of the waste mass, as specified in the considered scenario

[SOURCE: EN 12920:2006+A1:2008]

### 3.9
**release mechanism**

physico-chemical processes that control the release of constituents from a solid into solution (leaching)

Note 1 to entry: In the case of monolithic materials, examples of these processes are diffusion, dissolution of constituents, initial surface wash-off and dissolution of the matrix.

### 3.10
**sample**

portion of material selected from a larger quantity of material

### 3.11
**test portion**

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

[SOURCE: IUPAC:1990]

### 3.12
**test portion of monolithic waste of regular shape**

test portion of monolithic waste for which the surface area of the test portion can be calculated on the basis of simple geometric equations

### 3.13
**test sample**

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

[SOURCE: IUPAC:1990]

### 3.14
**waste monolith**

waste delivered as bulky forms of specified minimum dimensions retaining its form in the landfill over a specified timeframe

Note 1 to entry: A waste monolith disposed among regular waste will not generally be required to be tested, as its contribution to landfill leachate quality is marginal.

## 4 Principle

This European Standard describes a method to determine as a function of time the release of constituents from a monolithic waste material, with a leachant in contact with its surface.

The test portion of monolithic waste is placed in a reactor / leaching vessel and 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.

The following test conditions are fixed:

— the type of leachant;

— the temperature;

— $L/A$ ratio ($\text{ml}\cdot\text{cm}^{-2}$);

— the total duration of the test;

— the number of eluates to be collected at fixed time intervals.

The eluate is collected in a fixed number of separate fractions. The eluate collection scheme is designed such that release mechanisms can be deduced from the analytical results.

The eluate fractions are filtered, and characterized physically and chemically according to existing standards (e.g. EN 16192).

The results of the test are expressed as a function of time, in terms of both mg of the constituents released per litre of eluate, and mg of constituents released cumulatively per $m^2$ of geometric surface area of the waste material exposed to leaching.

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:

— diffusion (through the pores and / or from the surface to the bulk of the leachant);

— initial surface wash-off;

— other processes (e.g. solubility control, changes in speciation, depletion).

## 5 Reagents

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

**5.1 Distilled water**, demineralized water, de-ionized water or water of equivalent purity ($5 < pH < 7,5$) with a conductivity < 0,1 mS/m according to grade 2 specified in EN ISO 3696.

**5.2 Nitric acid,** $c(HNO_3) = 0,1$ mol/l.

# 6 Equipment

## 6.1 General

Check the materials and equipment specified in 6.2.1 to 6.2.12 before use for proper operation and absence of interfering elements that may affect the result of the test (see 8.6).

Calibrate the equipment specified in 6.2.3, 6.2.7, 6.2.8, and 6.2.12.

## 6.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

**6.2.1 Leaching vessels or tanks of different size**, glass or plastics (e.g. polymethyl methacrylate (PMMA), polytetrafluoroethylene (PTFE), polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC)), which can be closed and/or sealed to avoid prolonged contact with the air, and apply vacuum if necessary.

Clean the vessel or tank before use by filling it with nitric acid (5.2), leaving it for at least 24 h and then flushing it out with water (5.1).

The vessel or tank may have connections to allow to apply vacuum at the beginning of the leaching procedure.

NOTE 1 The contact with the air is kept limited to avoid uptake of $CO_2$ from the air in case of leaching from alkaline materials.

The minimum distance between the test portion and the walls of the vessel or tank shall be 2 cm, all around the test portion. Examples are given in Annex C.

Supports made of inert material shall be used to allow direct contact with water also on the bottom sides of the specimen. Supports shall not affect significantly the surface area of the sample exposed to the leachant.

NOTE 2 Test specimen can also be placed in the tank e.g. by attaching it to the lid.

**6.2.2 Diamond blade cutting device and/or core drilling device (dry process).**

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

**6.2.4 Device for measuring sample dimensions**, with an accuracy of at least 1 mm.

**6.2.5 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 bar).

Rinsing is compulsory.

**6.2.6 Glass or plastic bottles**, e.g. high density polyethylene (HDPE)/polypropylene (PP)/polytetrafluoroethylene (PTFE)/polyethyleneterephtalate (PET)

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 constituents HDPE/PP bottles are preferred, except for samples analysed for mercury.

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

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

**6.2.9 Thermometer.**

**6.2.10 Membrane filters**, for filtration of the eluates with a pore size of 0,45 µm.

**6.2.11 Measuring cylinders,** for volume determination with 1 % accuracy.

**6.2.12 Redox potential meter** (optional).

# 7 Sample preparation

## 7.1 General

For the performance of the DMLT a test portion is required, consisting of at least one monolithic specimen or test piece, the structure, homogeneity and composition of which shall be representative for the waste material that is to be investigated. The specimen(s) or test piece(s) shall have a minimum dimension in all directions of 40 mm.

NOTE In order to increase the representativeness of the test portion, it can be useful to test more than one specimen or test piece, together. The surface area of the test portion is, in that case, the total surface area of the individual specimens or test pieces.

Minimum dimensions are relevant, in order to ensure that, even for easily leachable constituents, no depletion occurs during the test. Minimum dimensions of 40 mm in nearly all cases suffice. However, in case of high release rates, it is recommended to justify afterwards that no depletion has taken place (see Annex A).

The laboratory sample consists of one or more specimens or test pieces.

The age of waste materials/specimens is an important factor, which can influence the leaching properties. In case the waste material results from a stabilization process (performed in the laboratory or in the practical situation) the waste material should be cured sufficiently long to avoid major variations in leaching due to ongoing changes in pore structure and in formation of release controlling mineral phases. When possible, the production date and/or curing time of the laboratory sample (at least if prepared in the laboratory) should be reported in the test report.

## 7.2 Preparation of the test portion

A test portion is obtained from the laboratory sample, by applying EN 15002.

NOTE If, in order to comply with the requirement of regular shape, the test portion is prepared by cutting or coring, new surfaces are exposed, which can lead to change(s) in leaching properties, compared with aged surfaces.

Store the test portion in accordance with EN 15002 to minimize changes due to the exposure to atmosphere (drying, carbonation, etc.).

## 7.3 Determination of the geometric surface area

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).

Determine the geometric shape of the test portion by measuring the length, width, height and/or diameter of the test portion, with an uncertainty of ± 1 mm.

Calculate the whole geometric surface area $A$ (in $\text{cm}^2$) based on normal calculation rules for the defined geometric shape.

# 8 Procedure

## 8.1 Testing conditions

The dynamic leaching test for monolithic waste shall be carried out at a temperature between 19 °C to 25 °C.

An example of installation is illustrated in Annex C.

NOTE A constant temperature 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 (6.2.1). This property is a subject for ruggedness validation.

## 8.2 Step 1 of the leaching procedure

Calculate the leachant volume $V_1$:

$$V_1 = (8 \pm 2) \times A \tag{1}$$

where

$V_1$ is the volume of the leachant, in ml;

$A$ is the surface area of the test portion, in $cm^2$.

Place the test portion in the leaching vessel (6.2.1) using the support, in order to prevent the test portion from touching the inner side of the leaching vessel. If the test portion consists of more than one specimen or test piece, the support shall be used in such a way that there is at least 2 cm space between the specimens or test pieces.

Fill the vessel with the calculated volume of leachant ($V_1$) (5.1), such that the top of the test portion is at least 2 cm submerged. Close the leaching vessel. Note the time $t_0$.

For specific scenarios in practice, it can be useful to apply another leachant (for instance ground water, sea water, $CO_2$ saturated water, etc.), and different temperature. This would constitute a deviation from the document, and shall be reported.

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

At the end of the first step remove the eluate from the leaching vessel, without removing small parts of waste material that may have fallen off the test portion. Note the time $t_1$. Immediately continue with step 2 (8.3).

Immediately measure pH and conductivity. Filter the eluate fraction off-line over a 0,45 µm membrane filter (6.2.10) and further prepare the eluate for analysis (see 8.5).

## 8.3 Steps 2 – 8 of the leaching procedure

Fill the leaching vessel again with the calculated volume of leachant ($V_1$), such that the top of the test portion is at least 2 cm submerged, and that the distance between the test portion and the wall of the vessel is at least 2 cm. Close the leaching vessel.

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

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 specimen significantly.

At the end of each step remove the eluate from the leaching vessel, without removing small parts of waste 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.

Immediately measure pH and conductivity. Filter the eluate fraction off-line over a 0,45 µm membrane filter (6.2.10) and further prepare the eluate for analysis (see 8.5).

**Table 1 — Time intervals for eluates collection**

| Step/fraction | Duration of the time interval[a] | Duration from the start of the test ($t_0$) |
|---|---|---|
| 1 | 6 h ± 15 min | 6 h ± 15 min |
| 2 | 18 h ± 45 min | 1 d ± 45 min |
| 3 | 1 d and 6 h ± 1 h | 2 d and 6 h ± 2 h |
| 4 | 1 d and 18 h ± 2 h | 4 d ± 4 h |
| 5 | 5 d ± 6 h | 9 d ± 10 h |
| 6 | 7 d ± 8 h | 16 d ± 18 h |
| 7 | 20 d ± 24 h | 36 d ± 42 h |
| 8 | 28 d ± 24 h | 64 d |

[a] The time intervals in this table allow to start the test three days per week: Monday, Tuesday, Wednesday. When the test is started on one of these days, the collection of the eluates fall in regular workdays.

NOTE 2 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 3 If the full duration of the test is not required (e.g. for factory production control) a test consisting of less than 8 steps can be performed as ‘indirect test’ as explained in the Introduction.

## 8.4 Weight loss of the monolithic waste during the test

If the specimen(s) fall(s) apart during the test, stop the test and record the failure to withstand the contact with water for prolonged time.

If it is required to check the weight loss of the specimen (for instance in order to assess the monolithic character of the waste), after step 8 of the leaching procedure, remove the test portion from the leaching vessel. Gather all solid parts and particles that have fallen off the test portion during the whole duration of the test. Dry these solid parts and particles at (40 ± 2) °C according to EN 14346 and weigh the dried material ($m_d$).

NOTE If the weight loss exceeds a certain value (to be specified in regulation or otherwise), the monolithic waste does not have enough integrity to determine the leaching behaviour of the material in monolithic form, in accordance with the procedure, described in this document.

## 8.5 Further preparation of the eluates for analysis

Measure the pH (6.2.8) and the conductivity (6.2.7) in accordance with EN 16192 (and optionally redox potential $E_h$ in mV) of each eluate fraction, just after collection. 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.

Divide the eluates into an appropriate number of sub-samples for different chemical analysis, after shaking well. 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.

Determine the concentrations of constituents of interest using the methods of eluate analysis (e.g. EN 16192). Also measure the total concentration of dissolved organic carbon (DOC) using EN 16192.

NOTE DOC (dissolved organic carbon) can be of relevance when material containing organic matter is stabilized. DOC is mobilized at high pH and is capable of mobilizing metals and organic contaminants well beyond its solubility limitation under the common alkaline conditions.

### 8.6 Blank test

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 (6.2.1) completely with leachant (5.1), 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 (5.1) and leave the leachant in the vessel for another (24 ± 1) h. Also submit this second blank eluate to the regular filtration, and measure the conductivity.

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

- in the first eluate of the blank test the concentration of each considered element shall be less than 10 % of the average concentration in the first three eluate fractions of the leaching procedure, or below the detection limit;
- the conductivity of the second blank eluate shall be < 0,2 mS/m.

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

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

## 9 Calculations

### 9.1 Expression of results

#### 9.1.1 General

The results of the leaching test shall be expressed as the cumulative amount of the constituents released, relative to the geometric surface area of the test portion (unit $mg \cdot m^{-2}$), see 9.1.2. It is also possible to express the results as the concentration of the constituents released (unit $mg \cdot l^{-1}$) in each step and/or, if done, at each instantaneous sampling (see 9.1.3).

NOTE 1 If results are expressed in $mg \cdot l^{-1}$ the results are only valid for the specimen size and shape tested and cannot be compared to results for specimens with other size and shape.

NOTE 2 When information on equilibrium based tests (e.g. pH dependence leaching test data) is available for the same material, this information can be used to identify if solubility limitations play a role (see Annex A).

NOTE 3 Keeping a graphical record of characterization and compliance test data will provide justification to adjustment of substances to be analysed as well as changes to the frequency of testing.

### 9.1.2 Expression of results in terms of area related release

The final results of the leaching tests are expressed as the cumulative amount of the constituents released, relative to the geometric surface area of the test portion (unit mg·m$^{-2}$).

Calculate the mass loss:

$$M_\mathrm{l} = \frac{m_\mathrm{d}}{A} \tag{2}$$

where

$M_\mathrm{l}$ is the mass loss during the test, in g·m$^{-2}$;

$m_\mathrm{d}$ is the weight of dried particles that have fallen off the test portion, in g;

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

Calculate the measured release of each constituent per step:

$$r_\mathrm{i} = \frac{10 \times c_\mathrm{i} \times V_1}{A} \tag{3}$$

where

$r_\mathrm{i}$ is the measured release of a constituent in Step $i$, in mg·m$^{-2}$;

$c_\mathrm{i}$ is the measured concentration of that constituent in the eluate of Step $i$, in mg·l$^{-1}$;

$V_1$ is the volume of the leachant, in ml;

$A$ is the geometric surface area of the test portion, in cm$^2$.

For analysis results below the limit of quantification for the components concerned both the lower limit and the upper limit of the release shall be calculated. For the lower limit calculate with $c_\mathrm{i}$ = 0, and for the upper limit calculate with $c_\mathrm{i}$ = the limit of quantification.

For every constituent, calculate the quantity that has been released cumulatively, up to a certain step of the leaching procedure:

$$R_\mathrm{n} = \sum_{i=1}^{n} r_\mathrm{i} \quad \text{for } n = 1 \text{ up to } 8 \tag{4}$$

where

$R_\mathrm{n}$ is the cumulative release of a constituent over a period of time, running from Step 1 until Step $n$ (inclusive), in mg·m$^{-2}$;

$r_\mathrm{i}$ is the measured release of a constituent in Step $i$, in mg·m$^{-2}$.

In the case upper and lower limit values were calculated for the release of a certain constituent, also for the cumulative release upper and lower limits shall be calculated. The lower limit value is obtained by accumulating lower limit values for $r_\mathrm{i}$ where relevant, and the upper limit value is obtained by accumulating upper limit values for $r_\mathrm{i}$ where relevant.

Plot the cumulative release of the different constituents as a function of time, corresponding with the end of the different steps. This graphical representation shall be documented on a double logarithmic scale (see Figure B.3, Type Y4). In case of compliance testing the graphical presentation may not always be necessary.

NOTE 1 The convenient manner is to store test data electronically as a workbook with pre-defined graphical lay-out.

NOTE 2 For the identification of release controlling factors, and of leaching mechanisms, see Annex A.

### 9.1.3 Expression of results in concentrations

The analysis of the eluates produced by the leaching test provides the value of the concentration of the constituents in the eluates of the individual time steps expressed in $mg \cdot l^{-1}$.

Plot the measured concentrations of the constituents released, the pH and the conductivity, as a function of time, when the release behaviour needs to be established (see Annex A).

### 9.1.4 Calculation of release mechanism

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

## 10 Performance characteristics

The performance characteristics of the method as determined in a parallel inter-comparison validation between comparable US EPA method 1315 and this European Standard (EN 15863) are given in Annex E. Table 2 gives the resulting typical values for repeatability and reproducibility limits as their observed ranges. The typical value is derived from the data in Table E.2 by taking the median value and rounding the numbers.

**Table 2 — Typical values and observed ranges of the repeatability and reproducibility limits**

| **Results of the validation of the release of substances as a function of time from a monolithic specimen of stabilized waste expressed in mg/m$^2$** | **Typical value** % | **Observed range** % |
|---|---|---|
| Repeatability limit, $r$ | 6 | *4 - 15* |
| Reproducibility limit, $R$ | 23 | *12 – 35* |
| The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions: The tests are performed in accordance with all the requirements of this standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. The repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary field sample. | | |

NOTE The repeatability limit ($r$) and the reproducibility limit ($R$) as given in Table E.2 and in this table are indicative values of the attainable precision, if the determination of release from monolithic stabilized waste is performed in accordance with this European Standard [EN 15863].

A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the determination of release from monolithic stabilized waste. In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Table E.2 and this table.

## 11 Documentation and test report

### 11.1 General

In order to comply with the present document, the following information shall be documented in such a way that they are immediately available on request. In the following list, at least, the items marked with an asterisk (*) shall be included in the test report. All the deviation from the minimum requirements of this document shall also be documented in the test report.

### 11.2 General data

- reference to this European Standard*;
- date of receipt of the laboratory sample*;
- sampling report, according to 7.1;
- identification of the laboratory sample*;
- way in which the test portion is obtained from the laboratory sample (e.g. selection of specimens / test pieces, moulding specimens in the laboratory, cutting/sawing/coring), and justification of this way of preparation of the test portion*;
- curing time and conditions of the laboratory sample (if carried out by the laboratory)*;
- storage conditions of the laboratory sample and test portion*;
- mass and 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.

### 11.3 Leaching test conditions

- date of the test (beginning and end)*;
- identification of the test equipment and instruments used, including the dimensions of the leaching vessel*;
- volume $V_1$ of the leachant, added to the test portion each step*;
- leachant renewal times of all steps;
- temperature range during the performance of the test;
- mass loss ($M_l$) during the test;
- any deviation from the test method and the reason of this deviation together with all circumstances that may have influenced the results*.

### 11.4 Analytical report

- pH and conductivity of the eluate fractions collected (optional: redox potential)*;

- any dilutions that were carried out*;
- methods of preservation of the eluate fractions for the different elements to be analysed, in accordance with 8.5*;
- quantities of preservation fluid added in accordance with 8.5 where this is more than 1 ml per 100 ml eluate;
- all measured concentrations.

### 11.5 Results of the leaching test

- the measured concentrations per step for each constituent, in mg·l$^{-1}$, presented in a table of results and graphically presented plotted against time;
- the released quantities $r_i$ calculated per step for each constituent, in mg·m$^{-2}$ geometric surface area of the monolithic waste; for analysis results below the limit of quantification for the components concerned both the lower limit and the upper limit of the released quantity shall be indicated;
- the cumulatively released quantities $R_i$ calculated per step for each constituent, in mg·m$^{-2}$ geometric surface area of the monolithic waste, plotted against time (on a double logarithmic scale); again, if relevant, lower and upper limits shall be indicated.

NOTE For testing of the same product, it is advisable to keep a running graphical record of test data to be used for identification of unexpected deviations and data consistency in production, which may ultimately lead to a reduced number of constituents to be tested (WFT) and/or a reduced frequency of testing.

# Annex A
(informative)

# Identification of release mechanisms and use of test results

## A.1 Introduction

The main reason for developing the dynamic monolith leach test is that the release from monolithic specimen is surface area related and not, like in granular material, dictated largely by percolation and equilibrium between solid and leachant. This implies that the test is carried out on a specimen with a quantifiable surface area and of sufficient size to minimize depletion effects during the test.

Examples of factors controlling release are discussed in more detail in A.2. More detailed data can be found in various scientific publications which are cited in the Bibliography [17 – 23].

The field of application is rather wide and not limited to stabilized waste as the same release phenomena are relevant for monolithic materials as construction products (as discussed in CEN/TC 51, CEN/TC 104, CEN/TC 125, CEN/TC 229, etc.) and radioactive waste forms [21].

## A.2 Examples of factors influencing the leaching of monolithic waste

There are several factors influencing dynamic release from monolithic specimen, which obviously are not equally relevant for any one given specimen. Below the different factors are discussed briefly.

Two types of factors can be distinguished. On the one hand, those which cannot be influenced by laboratory people performing this document. Those factors are linked to the material itself (e.g. intrinsic characteristics), including the possible curing stage.

**Nature of the material** – the nature of the material is already in part addressed in view of tortuosity, porosity, dimensions, pH, etc. The nature of a product will be of relevance for the substances that are likely to be of relevance. Cement-based materials, sintered bricks, light weight concrete, bituminous materials each have their specific characteristics and most relevant substances.

**Porosity** – the porosity of a specimen is of relevance as together with tortuosity, it controls the rate of transport by diffusion within the matrix. This relates to connected porosity, as closed porosity (as in light weight concrete) does not contribute to release from the specimen.

**Tortuosity** – the tortuosity is the extended path length for diffusion due to the nonlinear nature of pores. This parameter is a property, that is unique for any one specimen as it depends on curing time, water to cement ratio (relevant for products based on hydraulic or pozzolanic properties), production conditions, degree of compaction, constituents, sintering temperature, etc.

**pH** – the pH of a monolithic specimen is important as solubility is strongly controlled by pH. In particular, cement based materials undergo carbonation with time depending on the scenario of use. In general, a pH front will develop, where in the modified zone leaching will be different from the leaching of the bulk matrix. In case of materials with a neutral pH (e.g. bitumen, sintered products) the release in a neutral pH environment is not as sensitive to pH. On the other hand materials with a very low ANC are sensitive to field imposed pH conditions (e.g. from acid rain or acidic groundwater).

**Redox** – in case of stabilized waste with reducing properties (e.g. sulphide addition), a redox front may develop, when the product is exposed to the atmosphere. This strongly affects the release behaviour of specific constituents (R.E.C.O.R.D. study on reduced metallurgical slag) [23].

**Availability** – the availability of constituents is a fraction of the total content that is relevant for modelling release and can be seen as relevant to assess the maximum released amount that might occur under the most extreme field exposed conditions. This quantity can be derived from the pH dependence leaching test (EN 14429 or EN 14997).

On the other hand, factors which can be changed and/or need to be controlled by laboratory people performing this document. Those factors are linked to the test itself (excluding the possible curing stage).

**Shape** – specimen can be tested in different shapes, e.g. block, cube, cylinder, block with open spaces. Important for expressing the results and interpreting release is the need to be able to quantify the surface area as results are expressed per unit surface area. Monolithic materials with irregular surfaces are therefore more difficult to handle. In that case the geometric surface area can be estimated, or a specimen sampled from an irregular form by cutting or coring can be used as test portion.

**Dimensions** – the dimension of specimen to be tested is important from the viewpoint of depletion. I.e. in case of small dimensions mobile species can become depleted. This depletion is a function of the porosity, tortuosity and the interaction of the constituent of interest with the solid matrix in a given period of time. For concrete in general 4 cm cubes will be adequate, for stabilized waste specimen larger specimen (10 cm diameter) are generally provide better representative results.

**Temperature** – the temperature plays a role as diffusion is a temperature controlled parameter. The Arrhenius equation applies, which in the case of testing at 10 °C and 25 °C amounts to about a factor of 2 difference in release. The temperature has also a strong effect on the solubility of some key substances. For translating effects from lab to field this factor cannot be ignored.

**Degree of water contact** – the degree of water contact is crucial for assessing impact. In dry periods, there is no transfer of substance. Depending on the degree of water saturation in the product gradient relaxation can occur from the core to the surface of the material. From some studies it has been shown that summing the wet periods provides an acceptable estimate of the actual release in case of wet–dry periods. In other cases when relaxation effect occurs, the portion of the material close to the surface is re-concentrated during each dry period and this leads to a significative increase in release versus liquid to solid ratio. Full mechanistic modelling is, however, the preferred prediction approach.

**Leachant** – the leachant used in the test is water. For standardization purposes and to ensure comparability of data, this choice was made. When assessment of a given aquatic environment is aimed at (salted, containing DOC, acidic, etc.), the composition of the leachant can be adapted in the procedure.

**Solubility, diffusion, wash off** – Different release mechanisms lead to different release patterns in the dynamic leaching test (see Annex B for a more detailed evaluation):

- solubility limitations;
- diffusion from the interior of the matrix to the surface;
- surface wash-off is a process where as a results of the process, substances may be concentrated on the surface and be released at the first contact with water.

The porosity may be extremely low in molten products, like slag or vitrified material. In case of highly soluble constituents, depletion of substance may occur. Within the matrix as well as at the surface of the specimen in contact with a solution solubility limitations and changes in solubility may occur.

Options to verify if solubility limitations are likely to control release of substances from a monolithic specimen are:

a) Comparing the measured concentrations in the dynamic leaching test eluates with the results from the pH dependence test (EN 14429 or EN 14997) at the corresponding pH. When dynamic leaching test data match with the pH dependence test results, solubility control as depicted by the pH dependence test is very likely controlling release.

b) By comparing concentration data as obtained from the first fractions of a percolation test (pore water simulation) on size reduced monolithic material with data from the pH dependence test. In case data from the percolation test on crushed monolithic material and pH dependence test data match, then that is an indication of solubility control over a wide range of pH and L/S conditions and thus will be a controlling factor in release from monolithic waste as well.

## A.3 Long term release prediction

A proper understanding of the mechanisms of release for the constituents of interest is of importance to be able to make predictions on long term release from monolithic waste. Based on this understanding simplified and more sophisticated model predictions can be made.

These calculations are to be made in the framework of the methodology described/standardized in EN 12920. Especially, the results of the dynamic leaching test cannot be used alone for the prediction/assessment of the long term release. In order to conduct properly these calculations, this mostly requires the use of results of other tests as the ones developed within CEN/TC 292, such as CEN/TS 14405, EN 14429.

A pitfall can be the use of results from this test to predict behaviour in a specific application without properly taking the mechanism of release into account. When results are expressed in $mg \cdot m^{-2}$ at a given exposure time, the direct multiplication with time is incorrect as a square root of time relationship underlies these data. Data expressed in $mg \cdot kg^{-1}$ cannot be transposed to release (only after recalculation to $mg \cdot m^{-2}$ units).

A simplified approach assumes diffusion controlled release corrected for temperature and degree of water contact. A further refinement of this approach involves a mass balance evaluation to correct for already leached material (decrease of available quantity). For non-interacting species (e.g. chlorides), this approach will provide quite reasonable estimates of release however not for other species of interest (e.g. Si, Ca, heavy metals).

In recently more sophisticated model predictions have been published that take physical form and full chemistry into account [19, 20].

# Annex B
(informative)

# Assessment of release mechanisms (if required)

## B.1 Overview of release mechanisms

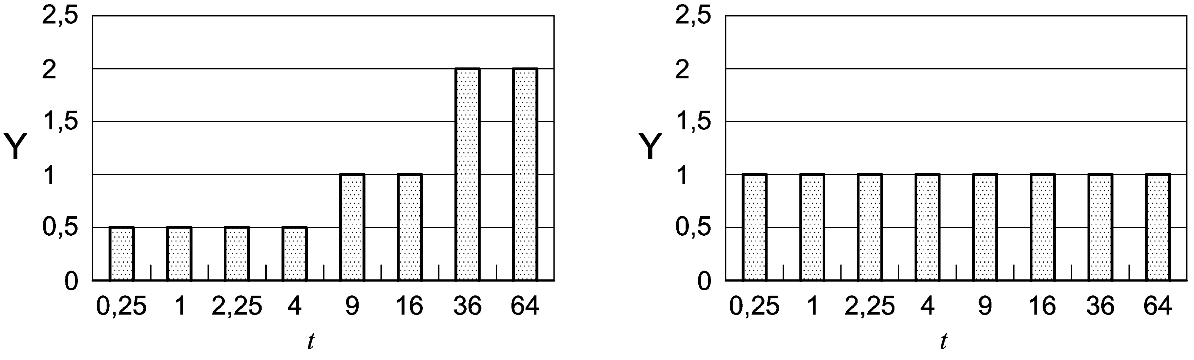
Prediction of the long term release of dangerous substances is important with respect to the evaluation of the release behaviour of a construction product during its service life in a construction. In order to be able to predict the long term release by extrapolation, the release mechanism needs to be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

In general two main release mechanisms may be distinguished:

a) diffusion;

b) dissolution.

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 European Standard (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)

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).

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.

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

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

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

e) to link test results from a standard test duration (normally 64 days) to shortened factory production control (FPC) 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.

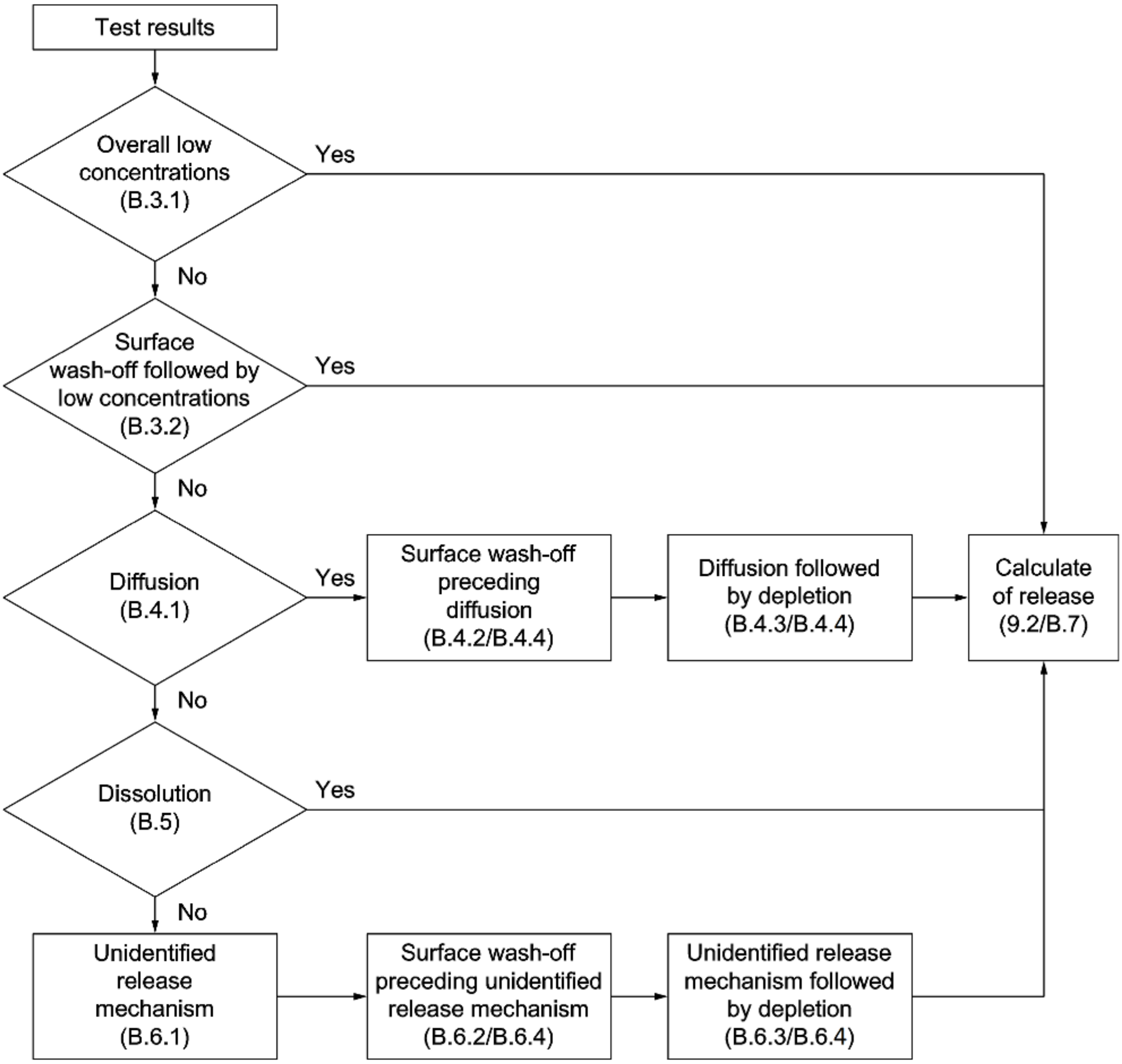


**Figure B.2 — Summary of the procedure for identifying the release mechanism and specifying the level of release**

## B.3 Concentrations close to the limit of quantification

### B.3.1 Overall low concentrations

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:

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

where

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

$c_\mathrm{i}$ is the concentration of the substance in Eluate i, in µg/l.

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

### B.3.2 Surface wash-off followed by low concentrations

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.

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

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

and

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

where

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

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

$c_\mathrm{i}$ is the concentration of the substance in Eluate i, in µg/l.

If for a specific substance surface wash-off is followed by low concentrations, 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

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:

$$\sqrt{MSE} < 0,40 \qquad \text{(B.4)}$$

where

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

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

$c_\mathrm{i}$ is the concentration of the substance in Eluate i, in µg/l;

$SE_\mathrm{i}$ is the square error of the concentration of the substance in Eluate i;

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

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.

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

$c_{3-4} = \frac{\sum_{i=3}^{4} c_i}{2}$ is the average concentration of the substance in Eluates 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

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:

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

and

$$\frac{\sigma_c}{c_{1-8}} < 0{,}25 \tag{B.8}$$

where

$\sigma_{\mathrm{pH}} = \sqrt{\frac{\sum_{i=1}^{8}(\mathrm{pH}_\mathrm{i} - \mathrm{pH}_{1-8})^2}{8}}$ is the standard deviation of the pH of Eluates 1 to 8;

$\mathrm{pH}_{1-8} = \frac{\sum_{i=1}^{8}\mathrm{pH}_\mathrm{i}}{8}$ is the average pH of Eluates 1 to 8, in µg/l;

$\mathrm{pH}_i$ is the pH of Eluate i;

$\sigma_\mathrm{C} = \sqrt{\frac{\sum_{i=1}^{8}(c_\mathrm{i} - c_{1-8})^2}{8}}$ is the standard deviation of the concentrations of the substance in Eluates 1 to 8;

$c_{1-8} = \frac{\sum_{i=1}^{8}c_\mathrm{i}}{8}$ is the average concentration of the substance in Eluates 1 to 8, in µg/l;

$c_\mathrm{i}$ is the concentration of the substance in Eluate i, in µg/l.

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

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

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:

$$\left|pH_1 - pH_{2-8}\right| < 0{,}5 \tag{B.9}$$

and

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

where

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

$\mathrm{pH}_i$ 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 Eluates 2 to 4, in µg/l;

$c_\mathrm{i}$ is the concentration of the substance in Eluate i, in µg/l.

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

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.

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

$c_\mathrm{i}$ is the concentration of the substance in Eluate i, in µg/l;

$\sigma_\mathrm{pH} = \sqrt{\frac{\sum_{i=1}^{8}\left(pH_\mathrm{i} - pH_{1-8}\right)^2}{8}}$ is the standard deviation of the pH of Eluates 1 to 8;

$\text{pH}_{1-8} = \frac{\sum_{i=1}^{8} pH_\text{i}}{8}$ is the average pH of Eluates 1 to 8, in µg/l;

$\text{pH}_\text{i}$ is the pH of Eluate i.

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 9.1.2 for the calculation of the area release $r_\text{i}$ 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:

$$R_\text{SWO} = R_2 - r_3 - r_4 \tag{B.16}$$

where

$R_\text{swo}$ 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 9.1.2);

$r_3$ is the area release of the substance for fraction 3, in mg/m$^2$ (see 9.1.2);

$r_4$ is the area release of the substance for fraction 4, in mg/m$^2$ (see 9.1.2).

### B.7.3 Release in the case of dissolution

In the case of dissolution the cumulative release, $R_\text{n}$ 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 days**

| 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 | $R_{T_E} = R_2 + \left(R_8 - R_2\right) \frac{\sqrt{T_E} - \sqrt{1}}{\sqrt{64} - \sqrt{1}}$ | (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 | $R_{T_E} = R_2 + \left(R_8 - R_2\right) \frac{\sqrt{T_E} - \sqrt{1}}{\sqrt{64} - \sqrt{1}}$ | (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 | $R_{T_E} = R_2 + \left(R_8 - R_2\right) \frac{\sqrt{T_E} - \sqrt{1}}{\sqrt{64} - \sqrt{1}}$ | (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] | 2 800 |
| $c_2$ [µg/l] | 940 |
| $c_3$ [µg/l] | 540 |
| $c_4$ [µg/l] | 470 |
| $c_5$ [µg/l] | 1 300 |
| $c_6$ [µg/l] | 1 300 |
| $c_7$ [µg/l] | 2 100 |
| $c_8$ [µg/l] | 2 300 |

| | |
|---|---|
| $pH_1$ | 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 |

**Sulfate**

| **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** | |
|---|---|
| $\sigma_{\mathrm{pH}}$ | |
| $\sigma_{\mathrm{pH}} < 0,25$ | |
| $\sigma_{\mathrm{c}}$ | |
| $c_{1–8}$ | |
| $\sigma_{\mathrm{c}}/c_{1–8} < 0,25$ | |

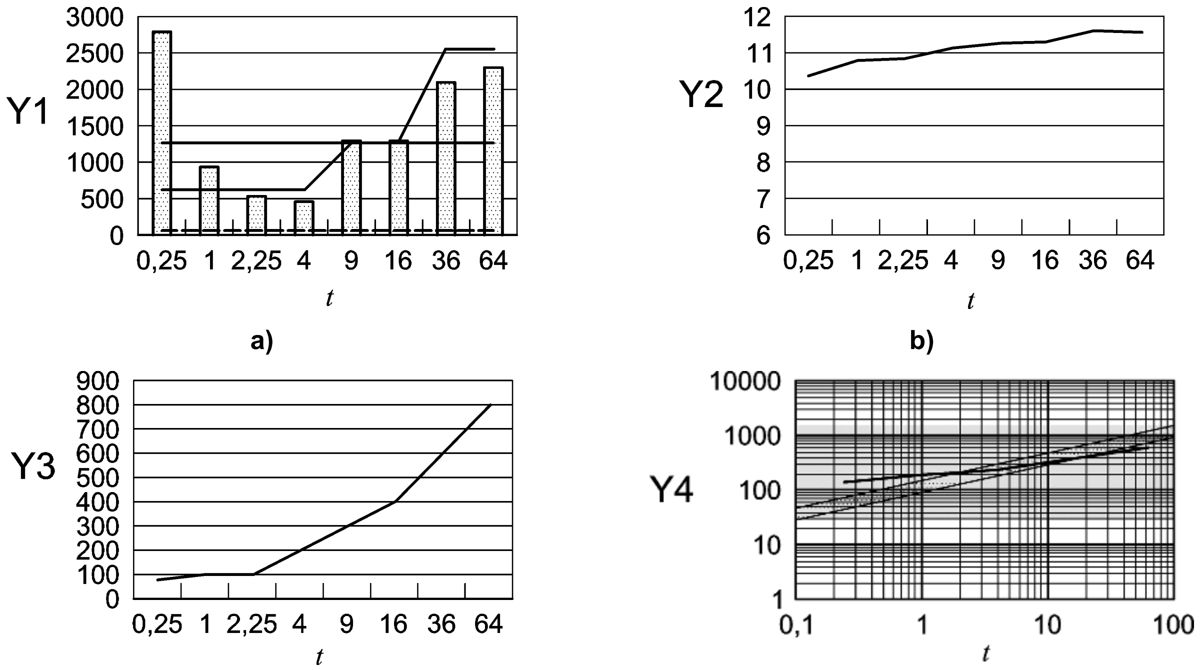
| **Surface wash-off preceding unidentified mechanism** | |
|---|---|
| $\Delta \mathrm{pH}_1$ | |
| $\Delta \mathrm{pH}_1 < 0,50$ | |
| $c_1/c_{3–4} > 1,8$ | |

| **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 | [m²] | $m_{\mathrm{s1–2}}$ | 3 | [g] |
| Volume eluate ($V$) | 25,254 | [l] | $m_{\mathrm{s3–8}}$ | 2 | [g] |
| $r_1$ | 140 | [mg/m²] | $R_1$ | 140 | [mg/m²] |
| $r_2$ | 47 | [mg/m²] | $R_2$ | 190 | [mg/m²] |
| $r_3$ | 27 | [mg/m²] | $R_3$ | 210 | [mg/m²] |
| $r_4$ | 23 | [mg/m²] | $R_4$ | 240 | [mg/m²] |
| $r_5$ | 65 | [mg/m²] | $R_5$ | 300 | [mg/m²] |
| $r_6$ | 65 | [mg/m²] | $R_6$ | 370 | [mg/m²] |
| $r_7$ | 100 | [mg/m²] | $R_7$ | 470 | [mg/m²] |
| $r_8$ | 110 | [mg/m²] | $R_8$ | 590 | [mg/m²] |
| $R_{\mathrm{swo}}$ | 140 | [mg/m²] | | | |
| $m_{\mathrm{a}}$ | 9,9 | [g/m²] | | | |

| **Cumulative release over 64 d:** | 590 | [mg/m²] |
|---|---|---|



a) b) c) d)

**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** | | | |
|---|---|---|---|
| $c_{2–8}$ | 16 | $c_{2–8}/DL$ | 1,6 |
| $c_{2–8}/DL < 1,5$ | no | | |

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

| **Diffusion** | |
|---|---|
| $c_8/c_7$ | |
| $c_8/c_7 < 0,9$ | |
| $c_{2-8}$ | |
| $SE_2$ | 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** |
|---|
| $c_{3-4}$ |
| $c_1/c_{3-4} > 1,8$ |

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

| **Dissolution** |
|---|
| $\sigma_{pH}$ |
| $\sigma_{pH} < 0,25$ |
| $\sigma_c$ |
| $c_{1-8}$ |
| $\sigma_c/c_{1-8} < 0,25$ |

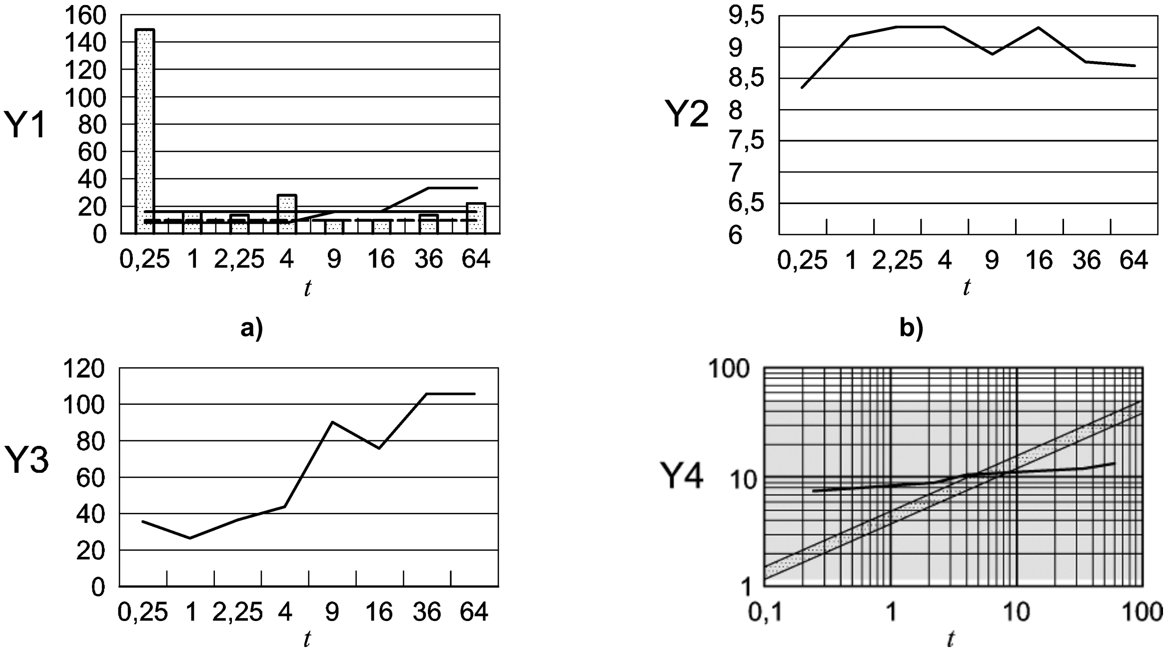
| **Surface wash-off preceding unidentified mechanism** |
|---|
| $\Delta pH_1$ |
| $\Delta pH_1 < 0,50$ |
| $c_1/c_{3-4} > 1,8$ |

| **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$^2$] | $m_{\mathrm{s1-2}}$ | 0,5 | [g] |
| Volume eluate ($V$) | 2,007 | [l] | $m_{\mathrm{s3-8}}$ | 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$^2$] |
| $R_{\mathrm{swo}}$ | 6,3 | [mg/m$^2$] | | | |
| $m_{\mathrm{a}}$ | 20 | [g/m$^2$] | | | |

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



a) b) c) d)

**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** | |
|---|---|
| $\sigma_{pH}$ | |
| $\sigma_{pH} < 0,25$ | |
| $\sigma_c$ | |
| $c_{1-8}$ | |
| $\sigma_c/c_{1-8} < 0,25$ | |

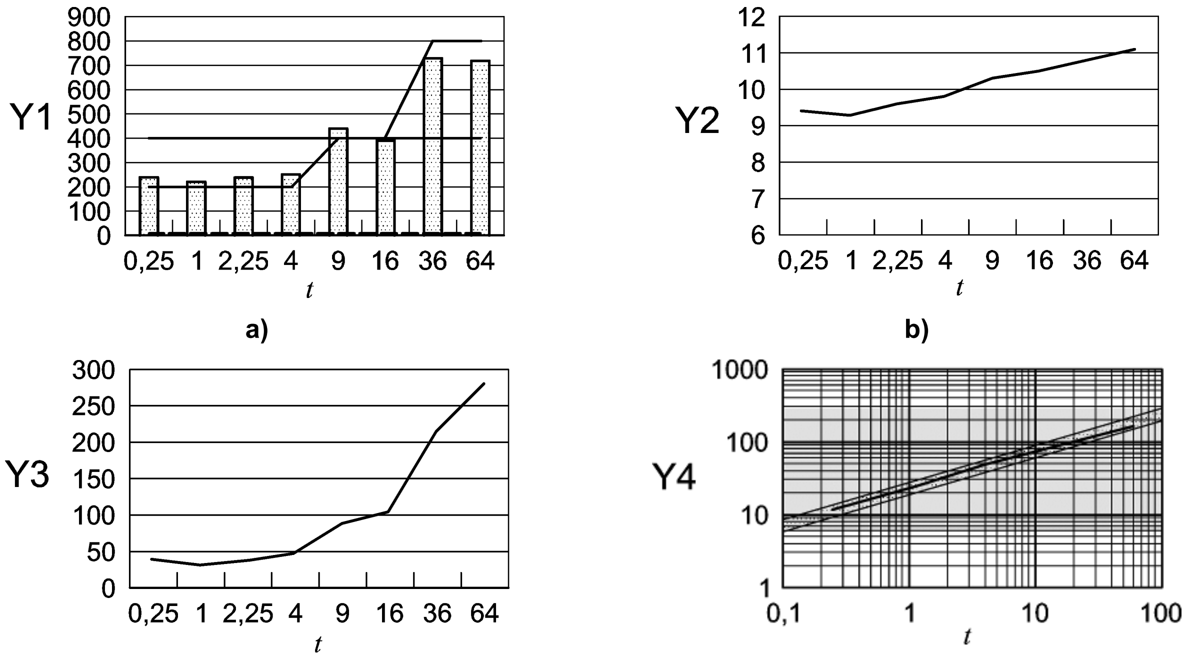
| **Surface wash-off preceding unidentified mechanism** | |
|---|---|
| $\Delta pH_1$ | |
| $\Delta pH_1 < 0,50$ | |
| $c_1/c_{3-4} > 1,8$ | |

| **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 | |
| $\sigma_{pH}$ < 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$^2$] | $m_{s1-2}$ | 0,5 | [g] |
| Volume eluate ($V$) | 22,850 | [l] | $m_{s3-8}$ | 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$^2$] |
|---|---|---|



a) b) c) d)

**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** | |
|---|---|
| $\sigma_{pH}$ | 0,07 |
| $\sigma_{pH} < 0,25$ | yes |
| $\sigma_c$ | 130 |
| $c_{1–8}$ | 1 100 |
| $\sigma_c/c_{1–8} < 0,25$ | yes |

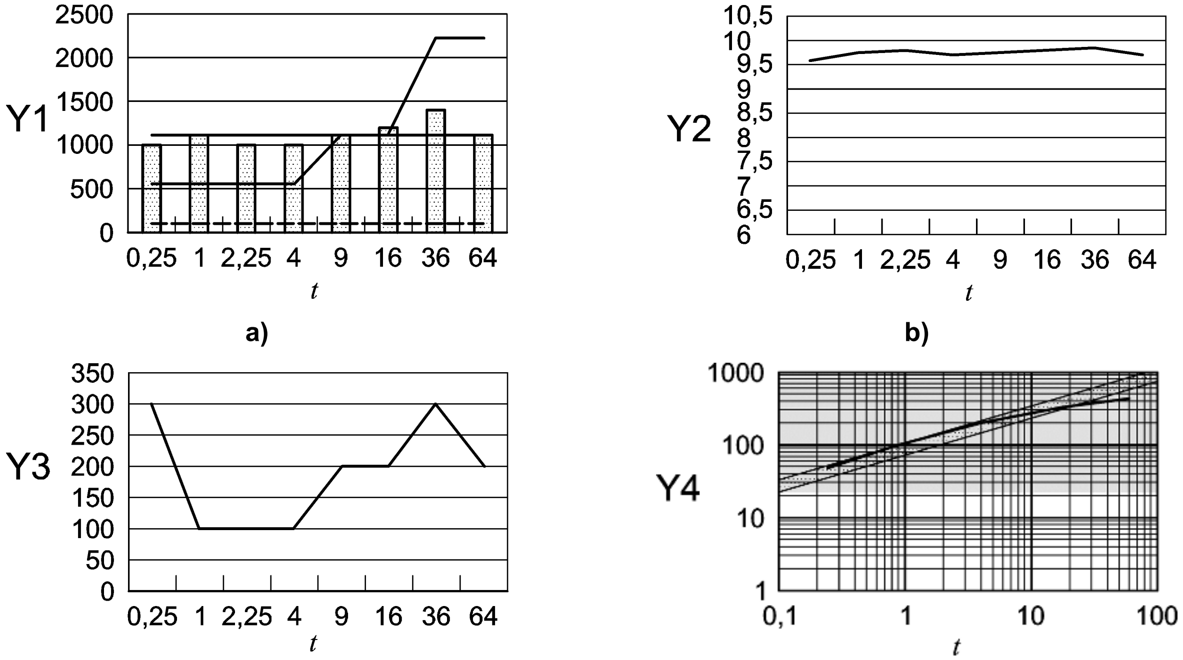
| **Surface wash-off preceding unidentified mechanism** |
|---|
| $\Delta pH_1$ |
| $\Delta pH_1 < 0,50$ |
| $c_1/c_{3–4} > 1,8$ |

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

| **Conclusion:** | dissolution controlled release |
|---|---|

| **Calculations** | | | | | |
|---|---|---|---|---|---|
| Area test specimen ($A$) | 0,3690 | [m²] | $m_{s1-2}$ | 4 | [g] |
| Volume eluate ($V$) | 18,450 | [l] | $m_{s3-8}$ | 2 | [g] |
| $r_1$ | 50 | [mg/m²] | $R_1$ | 50 | [mg/m²] |
| $r_2$ | 55 | [mg/m²] | $R_2$ | 110 | [mg/m²] |
| $r_3$ | 50 | [mg/m²] | $R_3$ | 160 | [mg/m²] |
| $r_4$ | 50 | [mg/m²] | $R_4$ | 210 | [mg/m²] |
| $r_5$ | 55 | [mg/m²] | $R_5$ | 260 | [mg/m²] |
| $r_6$ | 60 | [mg/m²] | $R_6$ | 320 | [mg/m²] |
| $r_7$ | 70 | [mg/m²] | $R_7$ | 390 | [mg/m²] |
| $r_8$ | 55 | [mg/m²] | $R_8$ | 450 | [mg/m²] |
| $R_{swo}$ | 0 | [mg/m²] | | | |
| $m_a$ | 16 | [g/m²] | | | |

| **Cumulative release over 64 d:** | 890 | [mg/m²] |
|---|---|---|



c) d)

**Key**

Y1 concentration in mg/l

Y2 pH

Y3 electrical conductivity in µS/cm

Y4 cumulative area release rate in mg/m²

*t* time in days

**Figure B.6 — Interpretation of shortened DSL-tests for factory production control**

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<br>Diffusion<br>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 | $R_8 = R_7\sqrt{\frac{64}{36}} = R_7 \times 1{,}33$ |
| Surface wash-off followed by low concentrations | 2,25 | $R_8 = R_2 + \left(R_3 - R_2\right)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{2.25}-\sqrt{1}} = R_2 + \left(R_3 - R_2\right) \times 14$ |
| | 4 | $R_8 = R_2 + \left(R_4 - R_2\right)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{4}-\sqrt{1}} = R_2 + \left(R_4 - R_2\right) \times 7$ |
| | 9 | $R_8 = R_2 + \left(R_5 - R_2\right)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{9}-\sqrt{1}} = R_2 + \left(R_5 - R_2\right) \times 3{,}5$ |
| | 16 | $R_8 = R_2 + \left(R_6 - R_2\right)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{16}-\sqrt{1}} = R_2 + \left(R_6 - R_2\right) \times 2{,}33$ |
| | 36 | $R_8 = R_2 + \left(R_7 - R_2\right)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{36}-\sqrt{1}} = R_2 + \left(R_7 - R_2\right) \times 1{,}4$ |
| Diffusion preceded by surface wash-off (and followed by depletion)<br>Unidentified mechanism preceded by surface wash-off (and followed by depletion) | 2,25 | Not applicable |
| | 4 | Not applicable |
| | 9 | $R_8 = R_2 + \left(R_5 - R_2\right)\frac{\sqrt{64}-\sqrt{1}}{\sqrt{9}-\sqrt{1}} = R_2 + \left(R_5 - R_2\right) \times 3{,}5$ |

| **Release mechanism** | **Time (days)** | **Formula for extrapolating shortened test data to 64 d** |
|---|---|---|
| | 16 | $R_8 = R_2 + \left(R_6 - R_2\right)\dfrac{\sqrt{64}-\sqrt{1}}{\sqrt{16}-\sqrt{1}} = R_2 + \left(R_6 - R_2\right)\times 2{,}33$ |
| | 36 | $R_8 = R_2 + \left(R_7 - R_2\right)\dfrac{\sqrt{64}-\sqrt{1}}{\sqrt{36}-\sqrt{1}} = R_2 + \left(R_7 - R_2\right)\times 1{,}4$ |
| Diffusion followed by depletion<br>Unidentified mechanism followed by depletion | 2,25 | $R_8 = R_3\sqrt{\dfrac{64}{2.25}} = R_3 \times 5{,}33$ |
| | 4 | $R_8 = R_4\sqrt{\dfrac{64}{4}} = R_3 \times 4$ |
| | 9 | $R_8 = R_5\sqrt{\dfrac{64}{9}} = R_5 \times 2{,}67$ |
| | 16 | $R_8 = R_6\sqrt{\dfrac{64}{16}} = R_3 \times 2$ |
| | 36 | $R_8 = R_7\sqrt{\dfrac{64}{36}} = R_7 \times 1{,}33$ |
| Dissolution | 2,25 | $R_8 = 2 \cdot R_3\sqrt{\dfrac{64}{2.25}} = R_3 \times 10{,}67$ |
| | 4 | $R_8 = 2 \cdot R_4\sqrt{\dfrac{64}{4}} = R_3 \times 8$ |
| | 9 | $R_8 = 2 \cdot R_5\sqrt{\dfrac{64}{9}} = R_5 \times 5{,}33$ |
| | 16 | $R_8 = 2 \cdot R_6\sqrt{\dfrac{64}{16}} = R_3 \times 4$ |
| | 36 | $R_8 = 2 \cdot R_7\sqrt{\dfrac{64}{36}} = R_7 \times 2{,}67$ |

NOTE 1 In the case of a shortened test period of 2,25 d it is not possible to determine $R_{\text{swo}}$. 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 data about the *L/A* ratio

In the table below for some specimen types (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 6.2.1, stating that the minimum distance between the test portion and the walls shall be 2 cm. The maximum diameter is calculated in order to fulfil the criterion, mentioned in 8.2, stating that the top of the test portion shall be at least 2 cm submerged.

**Table C.1 — Vessel dimensions in relation with specimen of different sizes and/or shapes (based on *L/A* = 8)**

| Specimen | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Type | Height cm | Diameter cm | Length cm | Width cm | Volume $V$ ml | Surface $A$ $cm^2$ | $V_{total}$ [a] ml | Distance between vessel and specimen [b] cm | Distance between specimen and water level [b] cm | Diameter cylindrical vessel [c] *min. to max.* cm |
| cube | 10 | - | 10 | 10 | 1 000 | 600 | 5 800 | 2,0 | 10,4 | 18 to 23 |
| | | | | | | | | 4,4 | 2,0 | |
| cube | 4 | - | 4 | 4 | 64 | 96 | 832 | 2,0 | 5,4 | 9,7 to 11,5 |
| | | | | | | | | 2,9 | 2,0 | |
| cylinder | 10 | 10 | - | - | 785 | 471 | 4 553 | 2,0 | 17,6 | 14 to 20 |
| | | | | | | | | 5,2 | 2,0 | |
| rectangular | 5 | - | 20 | 10 | 1 000 | 700 | 6 600 | 2,0 | 5,1 | 26 to 30 |
| | | | | | | | | 4,1 | 2,0 | |
| rectangular | 16 | - | 4 | 4 | 256 | 288 | 2 560 | 2,0 | 17,0 | 9,7 to 12,8 |
| | | | | | | | | 3,6 | 2,0 | |

[a] $V_{total}$ is the sum of the volume of the specimen and leachant volume as calculated according to Formula (1).

[b] The distance of 2,0 cm is fixed, the non-fixed distance is calculated based on size and/or shape of the specimen type.

[c] The minimum diameter is calculated in order to fulfil the criterion, mentioned in 6.2.1, stating that the minimum distance between the test portion and the walls shall be 2 cm. The maximum diameter is calculated in order to fulfil the criterion, mentioned in 8.2, stating that the top of the test portion shall be at least 2 cm submerged.

If the most adapted calculated size (e.g. height) of the vessel cannot be bought easily, or if several test portions with slightly different sizes shall be tested, it is recommended to acquire a slightly bigger vessel. The volume is then corrected by adding inert material (e.g. glass balls) to comply with the *L*/*A* ratio of 8 ml·$cm^{-2}$. The minimum distance between test portion and inert material still need to be equal to or greater than 2 cm.

# Annex D
(informative)

# Process map for EN 15863

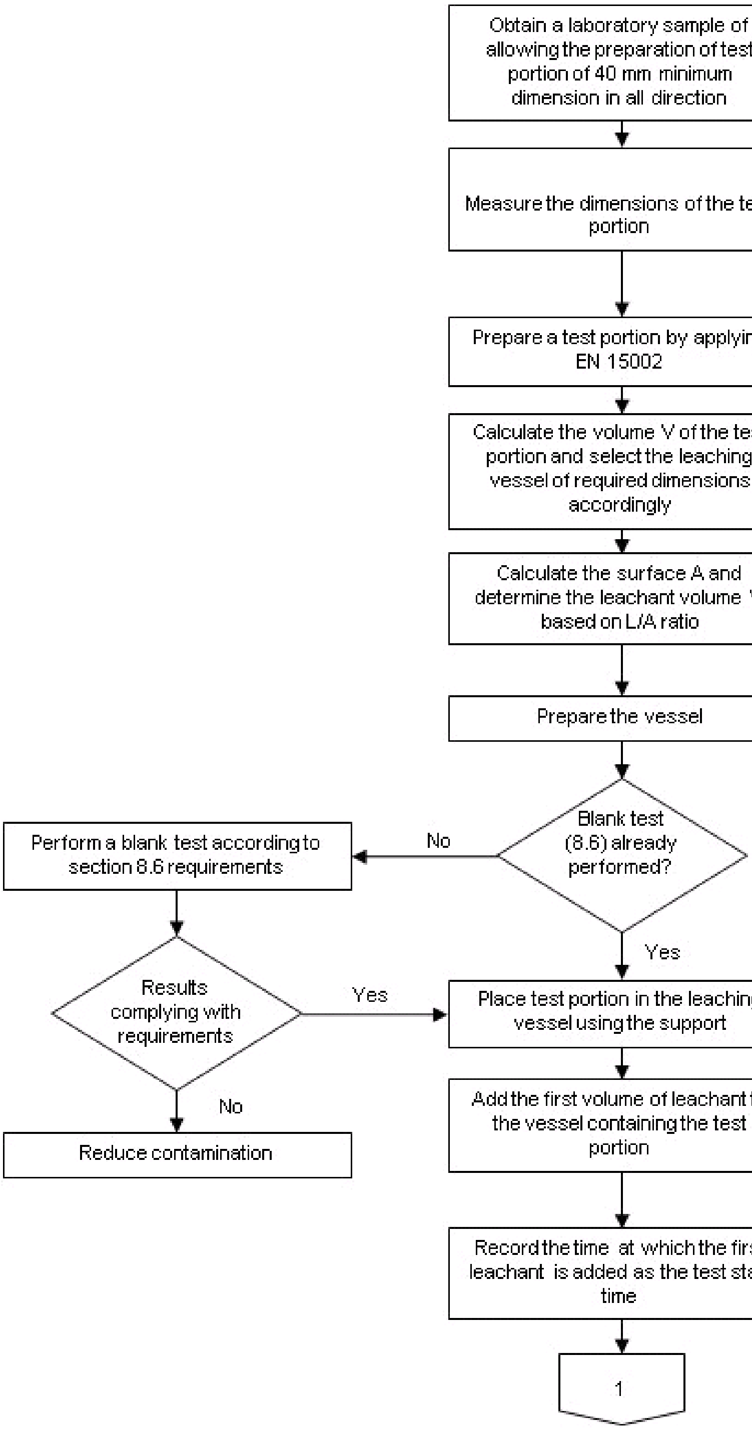


Figure D.1

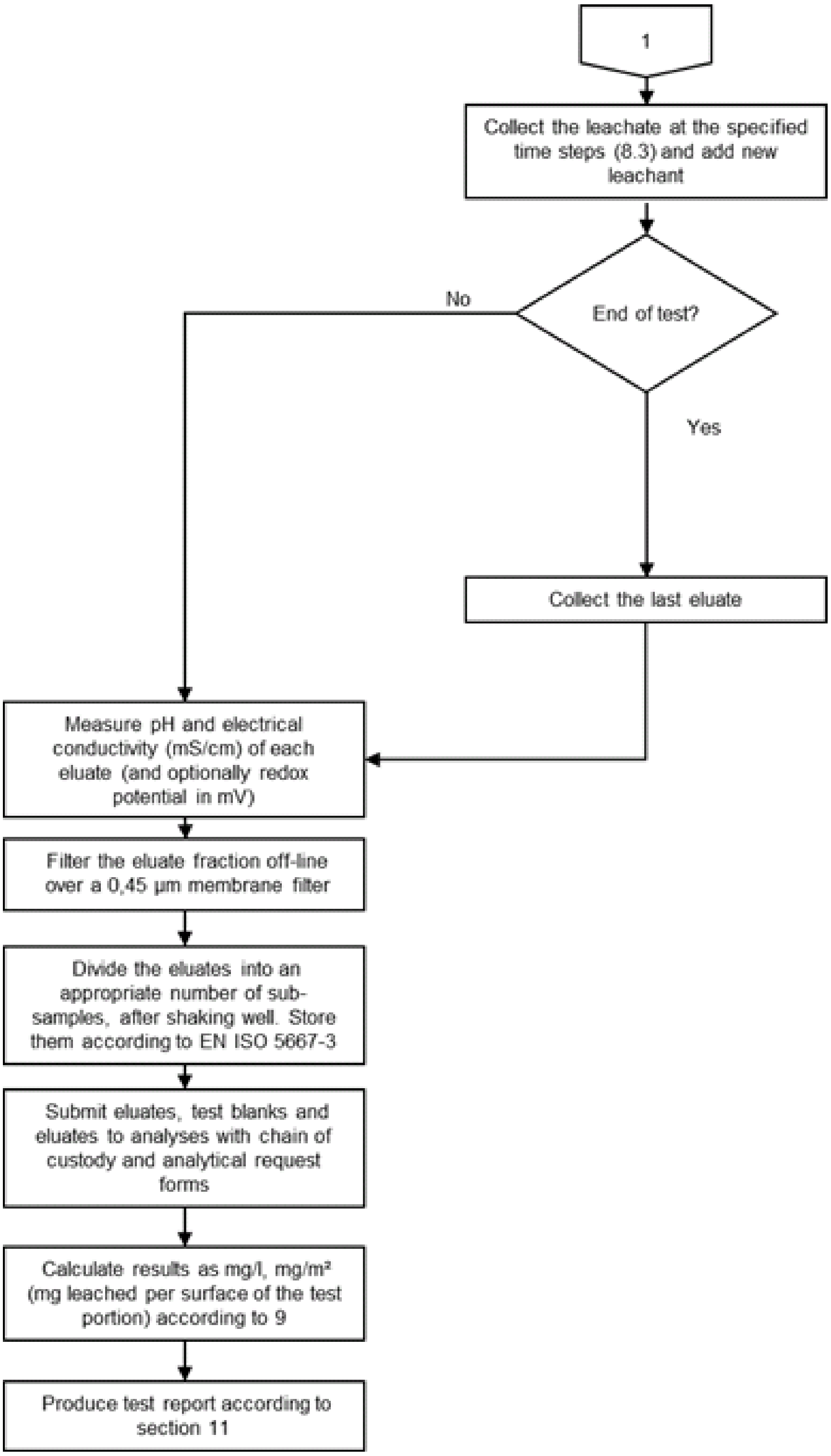

1
Collect the leachate at the specified time steps (8.3) and add new leachant
End of test?
No
Yes
Collect the last eluate
Measure pH and electrical conductivity (mS/cm) of each eluate (and optionally redox potential in mV)
Filter the eluate fraction off-line over a 0,45 µm membrane filter
Divide the eluates into an appropriate number of sub-samples, after shaking well. Store them according to EN ISO 5667-3
Submit eluates, test blanks and eluates to analyses with chain of custody and analytical request forms
Calculate results as mg/l, mg/m² (mg leached per surface of the test portion) according to 9
Produce test report according to section 11

**Figure D.2**

# Annex E
(informative)

# Repeatability and reproducibility data

## E.1 Material used in the interlaboratory comparison study

The interlaboratory comparison of release of substances from stabilized waste was carried out with ten American and one European laboratory on one monolithic material. The material selected for the interlaboratory comparison was chosen to represent stabilized waste (detailed information can be found in the final report on the interlaboratory comparison study [24, 25]).

Table E.1 provides the types of material chosen for testing and the selected substances for analysis.

**Table E.1 — Material type tested and substances analysed in the interlaboratory comparison of - Determination of release of substances from monolithic stabilized waste**

| Grain size class | Sample code | Material type tested |
|---|---|---|
| Monolithic | SWA | Cement stabilized waste |

## E.2 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2 using a log normal distribution of test data. The average values, the repeatability standard deviation ($RSD_r$) and the reproducibility standard deviation ($RSD_R$) were obtained (Table E.2).

## Table E.2 — Results of the interlaboratory comparison studies of - Determination of the release of substances from monolithic stabilized waste

| **CUMULATIVE RELEASE** | | | | **Concentration range** | | | | **Labs** | | **Collected** | **Filtered** | **Used** | **CUMULATIVE RELEASE** | | **Concentration range** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **EPA Method** | **Matrix** | **Para-meter** | Time days | Minimum $mg/m^2$ | Maximum $mg/m^2$ | $RSD_r$ % | $RSD_R$ % | $p$ | Outliers | Total number | Number < DLT | Total used | EN | Time days | Minimum $mg/m^2$ | Maximum $mg/m^2$ | $RSD_r$ % |
| 1315 | SWA | Al | 0,08 | 26,6 | 155,2 | 24 | 58 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 41,3 | 43,6 | 3 |
| | | | 1,0 | 239,5 | 548,1 | 10 | 29 | 9 | | 30 | 0 | 30 | | 1 | 195,7 | 221,3 | 6 |
| | | | 2,0 | 374,5 | 860,7 | 8 | 27 | 9 | | 30 | 0 | 30 | | 2,25 | 389,5 | 445,6 | 7 |
| | | | 7,0 | 621,9 | 1 347 | 7 | 24 | 9 | | 30 | 0 | 30 | | 8 | 616,4 | 701,5 | 7 |
| | | | 14,0 | 852,9 | 1 738 | 7 | 21 | 9 | | 30 | 0 | 30 | | 14 | 801,8 | 881,9 | 5 |
| | | | 28,0 | 1 140 | 2 213 | 6 | 30 | 9 | | 30 | 0 | 30 | | 15 | 846,9 | 926,6 | 5 |
| | | | 42,0 | 1 386 | 2 618 | 6 | 19 | 9 | | 30 | 0 | 30 | | 28 | 1 187 | 1 233 | 2 |
| | | | 49,0 | 1 551 | 2 925 | 6 | 20 | 9 | | 30 | 0 | 30 | | 36 | 1 410 | 1 472 | 2 |
| | | | 63,0 | 1 755 | 3 241 | 5 | 21 | 9 | | 30 | 0 | 30 | | | | | |
| 1315 | SWA | Sb | 0,08 | 4,1 | 24,6 | 21 | 64 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 4,15 | 4,55 | 5 |
| | | | 1,0 | 25,8 | 67,9 | 9 | 24 | 9 | | 30 | 0 | 30 | | 1 | 17,9 | 18,6 | 2 |
| | | | 2,0 | 37,4 | 90,6 | 8 | 21 | 9 | | 30 | 0 | 30 | | 2,25 | 36,9 | 39,4 | 3 |
| | | | 7,0 | 56,7 | 132,6 | 8 | 20 | 9 | | 30 | 0 | 30 | | 8 | 55,6 | 58,3 | 3 |
| | | | 14,0 | 76,2 | 166,3 | 8 | 18 | 9 | | 30 | 0 | 30 | | 14 | 70,6 | 73,5 | 2 |
| | | | 28,0 | 100,6 | 201,2 | 8 | 16 | 9 | | 30 | 0 | 30 | | 15 | 72,5 | 75,0 | 2 |
| | | | 42,0 | 120,7 | 228,2 | 7 | 15 | 9 | | 30 | 0 | 30 | | 28 | 95,4 | 95,9 | 1 |
| | | | 49,0 | 135,1 | 245,1 | 7 | 15 | 9 | | 30 | 0 | 30 | | 36 | 110,6 | 111,8 | 1 |
| | | | 63,0 | 152,2 | 265,7 | 7 | 15 | 9 | | 30 | 0 | 30 | | | | | |

| **CUMULATIVE RELEASE** | | | | **Concentration range** | | | | **Labs** | | **Collected** | **Filtered** | **Used** | **CUMULATIVE RELEASE** | | **Concentration range** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **EPA Method** | **Matrix** | **Parameter** | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % | $RSD_R$ % | $p$ | Outliers | Total number | Number < DLT | Total used | EN | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % |
| 1315 | SWA | As | 0,08 | 4,2 | 28,6 | 23 | 71 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 7,42 | 9,19 | 11 |
| | | | 1,0 | 13,8 | 48,0 | 15 | 41 | 9 | | 30 | 0 | 30 | | 1 | 15,9 | 16,8 | 3 |
| | | | 2,0 | 19,3 | 61,7 | 17 | 38 | 9 | | 30 | 0 | 30 | | 2,25 | 22,7 | 23,7 | 2 |
| | | | 7,0 | 31,3 | 100,5 | 20 | 31 | 9 | | 30 | 0 | 30 | | 8 | 35,4 | 37,1 | 2 |
| | | | 14,0 | 41,0 | 121,5 | 20 | 28 | 9 | | 30 | 0 | 30 | | 14 | 45,3 | 47,1 | 2 |
| | | | 28,0 | 50,9 | 139,5 | 18 | 25 | 9 | | 30 | 0 | 30 | | 15 | 48,4 | 50,3 | 2 |
| | | | 42,0 | 59,1 | 152,1 | 17 | 23 | 9 | | 30 | 0 | 30 | | 28 | 63,3 | 65,0 | 1 |
| | | | 49,0 | 63,9 | 158,9 | 17 | 22 | 9 | | 30 | 0 | 30 | | 36 | 72,1 | 74,5 | 2 |
| | | | 63,0 | 72,9 | 167,8 | 16 | 21 | 9 | | 30 | 0 | 30 | | | | | |
| 1315 | SWA | Ba | 0,08 | 0,3 | 9,6 | 55 | 105 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 2,58 | 3,48 | 15 |
| | | | 1,0 | 5,5 | 42,4 | 37 | 62 | 9 | | 30 | 0 | 30 | | 1 | 15,7 | 16,4 | 2 |
| | | | 2,0 | 15,8 | 68,2 | 24 | 41 | 9 | | 30 | 0 | 30 | | 2,25 | 37,7 | 38,7 | 1 |
| | | | 7,0 | 26,9 | 109,6 | 16 | 36 | 9 | | 30 | 0 | 30 | | 8 | 66,2 | 67,7 | 1 |
| | | | 14,0 | 49,7 | 156,1 | 12 | 31 | 9 | | 30 | 0 | 30 | | 14 | 90,3 | 93,0 | 2 |
| | | | 28,0 | 74,8 | 213,1 | 10 | 31 | 9 | | 30 | 0 | 30 | | 15 | 99,0 | 101,9 | 2 |
| | | | 42,0 | 92,7 | 277,2 | 8 | 32 | 9 | | 30 | 0 | 30 | | 28 | 130,0 | 133,5 | 1 |
| | | | 49,0 | 99,9 | 330,6 | 8 | 35 | 9 | | 30 | 0 | 30 | | 36 | 156,9 | 162,2 | 2 |
| | | | 63,0 | 111,9 | 387,5 | 7 | 36 | 9 | | 30 | 0 | 30 | | | | | |

| **CUMULATIVE RELEASE** | | | | **Concentration range** | | | | **Labs** | | **Collected** | **Filtered** | **Used** | **CUMULATIVE RELEASE** | | **Concentration range** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **EPA Method** | **Matrix** | **Para-meter** | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % | $RSD_R$ % | $p$ | Outliers | Total number | Number < DLT | Total used | EN | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % |
| 1315 | SWA | B | 0,08 | 1,3 | 4,8 | 18 | 40 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 1,93 | 2,30 | 9 |
| | | | 1,0 | 4,4 | 13,8 | 17 | 29 | 9 | | 30 | 0 | 30 | | 1 | 5,84 | 5,98 | 1 |
| | | | 2,0 | 7,3 | 18,5 | 14 | 25 | 9 | | 30 | 0 | 30 | | 2,25 | 10,2 | 10,2 | 1 |
| | | | 7,0 | 10,7 | 27,8 | 12 | 23 | 9 | | 30 | 0 | 30 | | 8 | 14,5 | 14,9 | 1 |
| | | | 14,0 | 13,5 | 35,1 | 11 | 22 | 9 | | 30 | 0 | 30 | | 14 | 18,1 | 18,4 | 1 |
| | | | 28,0 | 17,7 | 43,2 | 10 | 20 | 9 | | 30 | 0 | 30 | | 15 | 20,0 | 20,4 | 1 |
| | | | 42,0 | 21,8 | 49,6 | 9 | 19 | 9 | | 30 | 0 | 30 | | 28 | 25,4 | 25,7 | 1 |
| | | | 49,0 | 24,8 | 53,9 | 9 | 19 | 9 | | 30 | 0 | 30 | | 36 | 29,5 | 30,1 | 1 |
| | | | 63,0 | 29,0 | 59,6 | 8 | 18 | 9 | | 30 | 0 | 30 | | | | | |
| 1315 | SWA | Ca | 0,08 | 39,6 | 194,7 | 24 | 60 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 21,6 | 45,9 | 43 |
| | | | 1,0 | 593,8 | 1 564 | 8 | 36 | 9 | | 30 | 0 | 30 | | 1 | 464,2 | 482,2 | 2 |
| | | | 2,0 | 1 319 | 3 300 | 6 | 31 | 9 | | 30 | 0 | 30 | | 2,25 | 1 402 | 1 503 | 4 |
| | | | 7,0 | 2 534 | 5 335 | 6 | 24 | 9 | | 30 | 0 | 30 | | 8 | 2 587 | 2 751 | 3 |
| | | | 14,0 | 3 846 | 8 828 | 6 | 24 | 9 | | 30 | 0 | 30 | | 14 | 3 701 | 3 930 | 3 |
| | | | 28,0 | 5 550 | 11 675 | 5 | 22 | 9 | | 30 | 0 | 30 | | 15 | 4 219 | 4 505 | 3 |
| | | | 42,0 | 7 219 | 14 004 | 5 | 20 | 9 | | 30 | 0 | 30 | | 28 | 5 779 | 6 179 | 3 |
| | | | 49,0 | 8 103 | 16 091 | 4 | 21 | 9 | | 30 | 0 | 30 | | 36 | 7 307 | 7 769 | 3 |
| | | | 63,0 | 9 088 | 18 296 | 5 | 21 | 9 | | 30 | 0 | 30 | | | | | |

| **CUMULATIVE RELEASE** | | | | **Concentration range** | | | | **Labs** | | **Collected** | **Filtered** | **Used** | **CUMULATIVE RELEASE** | | **Concentration range** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **EPA Method** | **Matrix** | **Para-meter** | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % | $RSD_R$ % | $p$ | Outliers | Total number | Number < DLT | Total used | EN | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % |
| 1315 | SWA | Cr | 0,08 | 1,1 | 4,3 | 28 | 40 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 1,1 | 1,2 | 7 |
| | | | 1,0 | 4,0 | 9,4 | 17 | 26 | 9 | | 30 | 0 | 30 | | 1 | 4,0 | 4,3 | 4 |
| | | | 2,0 | 5,7 | 11,9 | 15 | 21 | 9 | | 30 | 0 | 30 | | 2,25 | 7,4 | 7,7 | 2 |
| | | | 7,0 | 8,7 | 16,4 | 13 | 18 | 9 | | 30 | 0 | 30 | | 8 | 10,5 | 10,8 | 2 |
| | | | 14,0 | 11,7 | 19,7 | 12 | 15 | 9 | | 30 | 0 | 30 | | 14 | 12,2 | 12,5 | 1 |
| | | | 28,0 | 14,7 | 23,0 | 10 | 14 | 9 | | 30 | 0 | 30 | | 15 | 12,7 | 13,0 | 1 |
| | | | 42,0 | 17,4 | 26,9 | 9 | 14 | 9 | | 30 | 0 | 30 | | 28 | 15,4 | 16,0 | 2 |
| | | | 49,0 | 19,4 | 30,0 | 8 | 13 | 9 | | 30 | 0 | 30 | | 36 | 17,9 | 18,6 | 2 |
| | | | 63,0 | 21,0 | 33,8 | 8 | 14 | 9 | | 30 | 0 | 30 | | | | | |
| 1315 | SWA | K | 0,08 | 305,0 | 1 360 | 15 | 47 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 344 | 370 | 4 |
| | | | 1,0 | 1 541 | 4 106 | 11 | 32 | 9 | | 30 | 0 | 30 | | 1 | 1 182 | 1 223 | 2 |
| | | | 2,0 | 2 410 | 5 829 | 12 | 29 | 9 | | 30 | 0 | 30 | | 2,25 | 2 473 | 2 536 | 1 |
| | | | 7,0 | 4 295 | 9 613 | 13 | 25 | 9 | | 30 | 0 | 30 | | 8 | 4 426 | 4 618 | 2 |
| | | | 14,0 | 6 055 | 12 758 | 13 | 24 | 9 | | 30 | 0 | 30 | | 14 | 5 591 | 5 805 | 2 |
| | | | 28,0 | 8 271 | 16 714 | 12 | 22 | 9 | | 30 | 0 | 30 | | 15 | 5 814 | 6 033 | 2 |
| | | | 42,0 | 9 862 | 20 026 | 11 | 22 | 9 | | 30 | 0 | 30 | | 28 | 7 953 | 8 288 | 2 |
| | | | 49,0 | 10 564 | 21 386 | 11 | 22 | 9 | | 30 | 0 | 30 | | 36 | 9 092 | 9 466 | 2 |
| | | | 63,0 | 11 589 | 23 556 | 11 | 22 | 9 | | 30 | 0 | 30 | | | | | |

| CUMULATIVE RELEASE | | | | Concentration range | | | | Labs | | Collected | Filtered | Used | CUMULATIVE RELEASE | | Concentration range | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **EPA Method** | **Matrix** | **Para-meter** | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % | $RSD_R$ % | $p$ | Outliers | Total number | Number < DLT | Total used | EN | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % |
| 1315 | SWA | Se | 0,08 | 43,5 | 224,9 | 14 | 54 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 47,7 | 50,8 | 3 |
| | | | 1,0 | 187,1 | 544,7 | 7 | 36 | 9 | | 30 | 0 | 30 | | 1 | 150,9 | 152,2 | 1 |
| | | | 2,0 | 278,4 | 743,5 | 7 | 33 | 9 | | 30 | 0 | 30 | | 2,25 | 293,7 | 304,5 | 2 |
| | | | 7,0 | 480,8 | 1 334 | 9 | 27 | 9 | | 30 | 0 | 30 | | 8 | 497,8 | 531,7 | 3 |
| | | | 14,0 | 667,2 | 1 711 | 9 | 23 | 9 | | 30 | 0 | 30 | | 14 | 620,9 | 660,7 | 3 |
| | | | 28,0 | 905,5 | 2 134 | 9 | 19 | 9 | | 30 | 0 | 30 | | 15 | 637,6 | 678,4 | 3 |
| | | | 42,0 | 1 061 | 2 387 | 9 | 18 | 9 | | 30 | 0 | 30 | | 28 | 885,4 | 949,8 | 4 |
| | | | 49,0 | 1 126 | 2 477 | 9 | 17 | 9 | | 30 | 0 | 30 | | 36 | 1 026 | 1 099 | 4 |
| | | | 63,0 | 1 229 | 2 632 | 9 | 17 | 9 | | 30 | 0 | 30 | | | | | |
| 1315 | SWA | V | 0,08 | 0,3 | 1,4 | 17 | 51 | 9 | | 30 | 0 | 30 | 15863 | 0,08 | 0,37 | 0,41 | 5 |
| | | | 1,0 | 1,2 | 3,1 | 9 | 27 | 9 | | 30 | 0 | 30 | | 1 | 1,05 | 1,09 | 2 |
| | | | 2,0 | 1,7 | 4,3 | 8 | 24 | 9 | | 30 | 0 | 30 | | 2,25 | 1,97 | 2,01 | 1 |
| | | | 7,0 | 2,6 | 5,9 | 8 | 23 | 9 | | 30 | 0 | 30 | | 8 | 3,02 | 3,13 | 2 |
| | | | 14,0 | 3,5 | 7,5 | 7 | 20 | 9 | | 30 | 0 | 30 | | 14 | 3,78 | 3,89 | 2 |
| | | | 28,0 | 4,7 | 9,3 | 7 | 18 | 9 | | 30 | 0 | 30 | | 15 | 3,91 | 4,03 | 2 |
| | | | 42,0 | 5,6 | 10,8 | 6 | 17 | 9 | | 30 | 0 | 30 | | 28 | 5,17 | 5,36 | 2 |
| | | | 49,0 | 6,3 | 12,0 | 6 | 17 | 9 | | 30 | 0 | 30 | | 36 | 6,09 | 6,29 | 2 |
| | | | 63,0 | 7,1 | 13,3 | 6 | 17 | 9 | | 30 | 0 | 30 | | | | | |

| **CUMULATIVE RELEASE** | | | | **Concentration range** | | | | **Labs** | | **Collected** | **Filtered** | **Used** | **CUMULATIVE RELEASE** | | **Concentration range** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **EPA Method** | **Matrix** | **Para-meter** | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % | $RSD_R$ % | $p$ | Outliers | Total number | Number < DLT | Total used | EN | Time days | Minimum mg/m$^2$ | Maximum mg/m$^2$ | $RSD_r$ % |

$RSD_r$ is repeatability standard deviation

$RSD_R$ is reproducibility standard deviation

$p$ is the number of laboratories remaining in statistical evaluation

NOTE 1 All laboratories conducted 3 replicates of each test with one of the laboratories (the reference laboratory) conducting 6 replicates.

NOTE 2 No outlier analysis was performed.

NOTE 3 No data were removed from analysis (i.e. “filtered”) due to blank concentrations.

NOTE 4 No data were “filtered for cause”.

NOTE 5 For the CEN test only $RSD_r$ can be quantified.

# Bibliography

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