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BSI Standards Publication

Characterization of waste — Leaching behaviour test for basic characterization — Dynamic monolithic leaching test with continuous leachant renewal under conditions relevant for specified scenario(s)

National foreword

This Draft for Development is the UK implementation of CEN/TS15864:2015. It supersedes PD CEN/TS 15864:2012 which is withdrawn.

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

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

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Characterization of waste - Leaching behaviour test for basic characterization - Dynamic monolithic leaching test with continuous leachant renewal under conditions relevant for specified scenario(s)

Caractérisation des déchets - Essais de comportement à la lixiviation pour la caractérisation de base - Essai de lixiviation dynamique des monolithes avec renouvellement continu du lixiviant dans des conditions pertinentes pour des scénarios spécifiés

Charakterisierung von Abfällen - Untersuchung des Elutionsverhaltens für die grundlegende Charakterisierung - Dynamisches Elutionsverfahren für monolithische Abfälle mit kontinuierlicher Erneuerung des Elutionsmittels unter Bedingungen für festgelegte Szenarien

This Technical Specification (CEN/TS) was approved by CEN on 19 October 2015 for provisional application.

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

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

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European foreword

This document (CEN/TS 15864:2015) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

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

This document supersedes CEN/TS 15864:2012.

The following significant technical change has been implemented in this new edition of the text:

— the steps/fractions for eluates collection have been harmonized with EN 15863.

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

This document was elaborated on the basis of:

— AFNOR XP X30-450:2002;

— AFNOR XP X30-467:2002;

— AFNOR XP X30-469:2007.

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

Introduction

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 certain number of separate fractions.

The composition, the temperature and the renewal rate of this solution are chosen for the test in order to study the behaviour of the waste material under fixed conditions when no specific scenario is under consideration or according to the conditions defined by the disposal or utilization scenario under consideration.

Three main ranges of renewal rates can be distinguished and addressed by this document:

- high renewal rate above a so-called “critical renewal rate” for which the released elements do not influence the release (so-called “no retro-action situation”);
- “low renewal rate” which corresponds to a quasi “saturation” of the solution in the reactor in order to reach stationary conditions (i.e. close to “saturation” equilibrium);
- intermediate range for which the released elements influence the release, but the concentrations remain significantly below saturation.

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.

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

1 Scope

This Technical Specification is applicable for determining the leaching behaviour of monolithic wastes under dynamic conditions. The test is performed under experimental conditions relevant to assess the leaching behaviour in view of the considered scenario(s). 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).

In general, the composition, the temperature and the continuous renewal rate of the leachant are chosen such that the leaching behaviour of the waste material can be studied in view of the considered disposal or recovery scenario. When the release needs to be determined without any reference to a specific scenario, the leachant is demineralized water, the temperature and the continuous renewal rate are fixed.

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.

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} $m \cdot s^{-1}$, water is likely to percolate through the monolith rather than flow around it. In such cases, relating the release to the geometric surface can lead to misinterpretation. A percolation test is then more appropriate (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
critical surface-related flow rate
SF_R^C
minimum surface-related flow rate above which the no-retro action situation is fulfilled for a given temperature and composition of the *leachant*

Note 1 to entry: The critical surface-related flow rate is expressed in $ml \cdot cm^{-2} \cdot h^{-1}$ or $cm \cdot h^{-1}$.

3.2
eluate
solution obtained by a leaching test

3.3
flow rate
F_R
quantity of *leachant* passing through the reactor containing the sample holder and the test portion per time unit

Note 1 to entry: The flow rate is expressed in $ml \cdot h^{-1}$.

3.4
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*.

3.5
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.

3.6
leachant renewal
continuous addition of *leachant* that flows through the tank

3.7
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, 2.3, modified — The original NOTE to the definition was not reproduced.]

3.8
liquid volume to surface area ratio
L/A
ratio between the amount of liquid (*L*) in the reactor which, at any time 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 ml·cm^{-2}.

Note 2 to entry: *L/A* has a constant value all along the test.

3.9
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

[SOURCE: EN 12457-1:2002, 3.9]

3.10
no retro-action situation
situation in which the release of constituents into the *leachant* does not depends on the constituents already released from the test portion

3.11
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, 2.2]

3.12
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, dissolution of the matrix.

3.13
renewal rate
ratio of the flow rate to the volume *V* of the solution in the reactor containing the sample holder and *test portion*

Note 1 to entry: The renewal rate is the number of renewal per day and is expressed in h^{-1}. The renewal rate is equivalent to a residence time.

3.14
sample

portion of material representative of a larger quantity of material

3.15
surface-related flow rate

***SF*R**

ratio of the flow rate F_R to the surface area of the *test portion*

Note 1 to entry: The surface-related flow rate is expressed in $ml \cdot cm^{-2} \cdot h^{-1}$ or $cm \cdot h^{-1}$.

3.16
surface-related release rate

mass of material that is transferred into the *leachant* per surface area unit and per time unit

Note 1 to entry: This rate is calculated from the concentrations of the main constituents measured in the eluate. It can vary as a function of time.

Note 2 to entry: The release rate is expressed in $mg \cdot cm^{-2} \cdot h^{-1}$.

3.17
test portion

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

[SOURCE: IUPAC:1990, 3.17]

3.18
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

3.19
test sample

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

[SOURCE: IUPAC:1990]

3.20
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 document 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 all along the leaching process.

The following set of test conditions is chosen in order to study the leaching behaviour of the waste material in view of the considered scenario(s):

— the type of leachant;

— the leaching temperature;

— the continuous renewal rate of the leachant (in h^{-1});

— the total duration of the test;

— the time intervals for eluates collection.

Three main ranges of renewal rates can be distinguished:

— high renewal rate above a so-called “critical renewal rate” for which the released elements do not influence the release;

— “low renewal rate” which corresponds to a quasi “saturation” of the solution in the reactor in order to reach stationary conditions (i.e. close to equilibrium);

— intermediate range for which the released elements influence the release, but the concentrations remain significantly below saturation.

The test procedure addressed these three kinds of renewal rate.

NOTE For different scenarios in practice, which are characterized by different exposure conditions (e.g. specific leachant or leachant renewal rate), it is generally desirable to use different set of the test conditions listed above.

When the release shall be determined without any reference to a specific scenario (see 8.1.4), a fixed set of parameters shall be used as a basis for comparison purposes between waste materials:

— the type of leachant;

— the temperature;

— the continuous renewal surface-related flow rate (SF_R) of the leachant;

— the total duration of the test;

— the time intervals for eluates collection.

The leachant is stirred during the leaching procedure, in order to prevent concentration gradients to occur in the liquid phase.

The eluate is collected in several separate fractions. The eluate collection scheme is designed such that release mechanisms can be deduced from the analytical results (8.3).

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 main leaching mechanisms that can be distinguished and identified are:

— dissolution of constituents/solubility controlled release;

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

— initial surface wash-off;

— dissolution of the matrix.

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 6.2.14 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, 6.2.9, 6.2.11 and 6.2.13.

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 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 1 cm, all around the test portion.

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.

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 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).

6.2.13 Pump, peristaltic pump or equivalent device allowing the renewal of the leachant at the considered renewal rate.

The range of flow rate covered by the pump shall be sufficiently wide so that the value of the flow rate to be applied for renewing the leachant and/or stirring the reactor is far enough the range’s boundaries.

6.2.14 Stirring device, magnetic bar (made of or coated by inert material) or pump (6.2.13) ensuring the recirculation of the leachant in the leaching vessels or tanks (6.2.1).

Stirring with a pump shall be done at a higher flow rate value than the range of leachant renewal rate, even in particular case “critical renewal rate” (8.1.2).

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 shall 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 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 cm^2) based on normal calculation rules for the defined geometric shape.

8 Procedure

8.1 Testing conditions

8.1.1 General case (intermediate range of renewal rate)

A test portion, of known mass M_i (g) and surface area A (cm^2), of the material to be tested is inserted into a reactor (leaching vessel) where it is placed in contact with, and completely submerged by all along the leaching procedure, the leachant whose composition, temperature and renewal rate are chosen for the test in order to study the behaviour of the material according to the conditions defined by the disposal or utilization scenario under consideration.

The temperature of the leachant in the reactor is maintained at a constant value (±5 °C). The leachant is renewed by a continuous supply at a constant surface related flow rate SF_R (in $\text{ml}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$) from a supply tank containing the leachant chosen for the test, so that the release of constituents of the waste material remains dynamic. The eluate output from the reactor is directed into a collection bottle for analysis purposes.

The surface-related flow rate is chosen in an intermediate range for which the released elements influence the release, but the concentrations remain significantly below saturation. This range is included in the interval between the critical flow-rate (see 8.1.2) and the low renewal rate (see 8.1.3).

When determining SF_R, both volumes of the reactor, and the tubing in case stirring by recirculation of the leachant is performed, shall be considered.

In that general case, a surface-related flow rate of the leachant of 0,5 $\text{ml}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ (0,5 $\text{cm}\cdot\text{h}^{-1}$) can be chosen as a starting point for cement-based monolithic waste or monolithic waste obtained by mixing waste with mineral reagents leading to cement-like forms. It is recommended to check it afterwards, for instance by ensuring that the measured concentrations of the considered elements in the different eluates are in the range (10 % to 90 %) of the concentrations of the same elements measured at the same pH in the pH dependence tests (either EN 14429 or EN 14997). See Figure A.1 in Annex A.

NOTE 1 In this procedure, the test portion is continuously submitted to a constant flow rate so that the leaching conditions remain dynamic and unchanged all along the duration of the test.

NOTE 2 For different scenarios in practice, which are characterized by different exposure conditions (e.g. specific leachant or leachant renewal rate), it is generally desirable to use different set of the test conditions listed above.

As the surface related flow rate SF_R can be insufficient to meet all the requirements of a stirred reactor (i.e. with homogenous concentrations in the reactor), the reactor is fitted with a stirring device either as a dedicated device (e.g. recirculation device) or as a result of the design of the reactor. It shall fulfil the following condition: the stirring action shall be sufficient to expose the entire surface area of the test portion to the same conditions, also taking into account the elements previously transferred in the solution and therefore the concentrations obtained in the solution.

The criterion to set stirring conditions is that for the selected flow rate of leachant through the reactor, the increase of the stirring intensity does not increase anymore the selected parameter (pH or constituents' concentration) at the output of the reactor.

NOTE 3 For example for a surface-related flow rate of 0,5 $cm \cdot h^{-1}$ (water flow rate of 50 $ml \cdot h^{-1}$ and geometric surface area of the test portion of 96 cm^2) and a static L/A ratio of 5 $ml \cdot cm^{-2}$, the water flow in the recirculation device used as stirring device is about 300 $ml \cdot min^{-1}$.

NOTE 4 The purpose of these requirements is to qualify an experimental setting and operating conditions. Only if a link with a previous qualification can be demonstrated, is a new qualification unnecessary.

NOTE 5 Excessive stirring can cause the test portion to be eroded.

NOTE 6 The solution specified for the aqueous solution may reflect certain scenario conditions (according to EN 12920).

The above criterion is a requirement for the main elements. In principle it may also apply to secondary or trace elements, with similar limitations.

The entire test portion is continuously immersed in the solution and the reactor is closed to prevent phenomena, such as carbonation by the CO_2 in the air.

NOTE 7 The continuous flow rate and the stirring action ensure that the test portion in contact with the leachant is not subjected to discontinuous variations (pH excursions for instance).

The eluates are collected at the output of the reactor. The total content (eluates and deposit) of the collection bottle is removed at different dates for analysis, with or without concentration by limited evaporation.

NOTE 8 For specific modelling purposes, it can be useful to also perform "instantaneous" eluates sampling by collecting small fractions at specific dates at the output of the reactor. The minimum collected volume required for the analysis is about 20 ml for most of the inorganic elements (see Annex A).

Attention should be paid that in case of concentration by heating organic substances (like DOC) could be altered and could subsequently modify the availability of the considered elements (including inorganic elements).

Two examples of installation are illustrated in Annex C.

8.1.2 Particular case "critical renewal rate"

The particular case specified in Annex B allows to put into contact a material with an aqueous solution in the same manner as in the general case, but with a constant surface related flow rate SF_R which is higher than the critical flow rate, defined and determined according to Annex A, to ensure that the released constituents cannot affect the release (so-called no retro-action situation).

NOTE When this option is used, the criterion specified in 8.1.1 above is replaced with the critical surface-related flow rate criterion specified in Annex B.

8.1.3 Particular case "low renewal rate"

The particular case specified in Annex D allows to put into contact a material with a leachant in the same manner as in the general case, but with a constant low surface related flow rate SF_R (then a low renewal rate). The experimental setting operates then in a "quasi-static" mode. This test allows to determine if a steady-state concentration, e.g. close to "saturation" equilibrium, is reached in the solution.

8.1.4 Particular case under fixed conditions when no specific scenario is under consideration ("not scenario-related")

When the release shall be determined without any reference to a specific scenario, the following set of parameters shall be used as a basis for comparison purposes between waste materials:

- the type of leachant: demineralized water as specified in 5.1;
- the leaching temperature: between 19 °C to 25 °C;
- the L/A ratio: (see NOTE 3);
- the continuous surface-related flow rate SF_R: (0,5 ± 0,07) cm·h^{-1};
- the total duration of the test: 36 d ± 0,25 d;
- the time intervals for eluates collection: see Table 1.

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	0,08 d ± 5 %	0,08 d ± 5 %
2	0,92 d ± 5 %	1 d ± 5 %
3	1,25 d ± 5 %	2,25 d ± 5 %
4	5,75 d ± 5 %	8 d ± 5 %
5	6 d ± 5 %	14 d ± 5 %
6	14 d ± 5 %	28 d ± 5 %
7	8 d ± 1 d	36 d ± 0,25 d
8 [b]	28 d ± 1 d	64 d ± 0,25 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.

[b] 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 (e.g. for factory production control).

NOTE 1 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.

NOTE 2 The intrinsic value of the static L/A ratio is not crucial as the key parameter is the surface-related flow rate SF_R. As soon as SF_R is maintained, the static L/A ratio can be adapted to the shape of the specimen or a specific reactor size. The target value can generally be 5 ml·cm^{-2}. However, keeping the value between 2 ml·cm^{-2} (to keep enough space for water around the specimen) and 10 ml·cm^{-2} (to limit the amount of release constituents remaining in the tank) is suitable.

8.2 Test procedure

Adjust the leachant supply pump in order to obtain the determined relevant constant surface-related flow rate SF_R according to 8.1 (in cm·h^{-1}). During this adjustment, the pump shall not be connected to the reactor.

NOTE 1 Depending on the pump, this adjustment can be time consuming: up to several days at SF_R below or equal to 0,5 cm·h^{-1}.

Assemble the equipment.

Place the weighed test portion in the empty leaching vessel (6.2.1) using the support (6.2.1), 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 1 cm space between the specimens or test pieces.

Put the reactor and the solution supply equipment to the chosen constant temperature (±5 °C).

NOTE 2 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.

Place the required volume of leachant in the supply tank.

Start up the leachant supply pump already adjusted at the determined relevant constant surface-related flow rate SF_R according to 8.1 (in cm·h^{-1}).

Fill quickly (e.g. with a beaker) the leaching vessel in so that the test portion is entirely submerged and close it. The fill in duration shall be negligible compared to the first eluate collection time (few minutes).

Start and adjust the chosen stirring system. Record this start time t_0 and date.

Remove air bubbles that can remain in the equipment (e.g. under the test portion, in the tubes).

At each time interval i specified in 8.3 or 8.1.4 depending of the considered case except the final one, do not stop the pump, change immediately the collection bottle of the eluate by another one that is suitable for the next time interval. Note the time t_i and seal the previous collection bottle.

Be aware of the carbonation of the eluate, particularly during a long step, where the eluate's volume is much smaller than the bottle's volume during a long time. Inert gas can be used to fill in the collection bottle initially.

If instantaneous sampling is performed (see 8.1.1, NOTE 8), collect a small fraction of eluate few minutes before the end of each time interval (at least 20 ml enabling the analysis of most of the inorganic elements). Note the time t_i.

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

When the time required for the test has expired, stop the supply pump and record the time t_n and date the test ended. Collect the last eluate in the last collection bottle. Collect separately the volume remaining in the leaching vessel. This last eluate will be necessary for the calculation of the total cumulated release for the considered global time duration test.

Determine weight loss of the test portion according to 8.4.

8.3 Contact periods – collection scheme

The release rate in the aqueous solution of the constituents of the material shall be determined for at least five contact periods during the scenario under consideration. The final contact period shall be such that the loss of mass from the sample to be tested at this stage is less than or equal to 10 % of its initial mass.

As a rule of thumb, the contact periods shall be shortened when the surface-related flow rate increase.

When at the final date the loss of mass of the test portion is higher or equal to 10 % of its initial mass, the formula given in Clause 8 do not apply anymore due to the corresponding evolution of the surface area.

NOTE When no background information on the leaching behaviour is available for the monolithic waste to be tested, more contact periods could be sampled. Examples of collection scheme and contact periods are given in 8.1.4 and Annexes B and C, according to the considered case.

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 1 For glassy or vitrified monolithic waste, a higher temperature such as (105 ± 3) °C can be applied to shorten the drying duration.

Calculate the mass loss M_l:

$$M_\mathrm{l} = \frac{m_\mathrm{d}}{A} \tag{1}$$

where

M_l is the mass loss during the test, in $\mathrm{g \cdot m^{-2}}$;

m_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 $\mathrm{m^2}$.

NOTE 2 If M_l 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 Eh 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.

If the concentrations are too weak, partially evaporate the sub-sample to increase the concentrations. The quantity of water evaporated shall be taken into account when calculating the concentrations of the constituents in the eluate.

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).

9.1.2 Expression of results in concentrations

The analysis of the eluates produced by the leaching test provides the value of average concentrations (or instantaneous if performed) of the constituents in the eluates during each contact period, expressed in $mg \cdot l^{-1}$.

Plot the measured concentrations of the constituents released, the pH and the conductivity, as a function of time.

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

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).

9.1.3 Expression of result in terms of surface related release

Calculate the measured release of constituent *i* per step *j*:

$$r_{\mathrm{i,j}} = \frac{10 \times c_{\mathrm{i,j}} \times V_{\mathrm{i,j}}}{A} \tag{2}$$

where

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

$c_{\mathrm{i,j}}$ is the measured concentration of constituent *i* in step *j* (excluding the volume of the water in the reactor at the final date), in mg·l^{-1};

$V_{\mathrm{i,j}}$ is the volume of eluate collected in step *j* (excluding the volume of the water in the reactor), in ml;

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

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

$$R_{\mathrm{i,n}} = \sum_{j=1}^{n} r_{\mathrm{i,j}} \quad \text{for } n = 1 \text{ up to the number of steps determined according to 8.4} \tag{3}$$

where

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

$r_{\mathrm{i,j}}$ is the measured release of a constituent *i* in step *j*, in mg·cm^{-2}.

For every constituent *i*, calculate the total cumulated release over the global duration of the test (R_{t}):

$$R_{\mathrm{i,t}} = R_{\mathrm{i,n}} + r_{\mathrm{i,f}} \tag{4}$$

where

$r_{\mathrm{i,f}}$ is the measured release of the constituent in the remaining volume of eluate in the reactor after the last eluate collection interval.

Plot the calculated 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 linear or semi-logarithmic scale.

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

9.2 Average surface-related release rate

The average surface-related release rate into the leachant at time interval *j* calculated from the concentration of a main constituent *i* in the eluate collected at time interval *j* is defined by a mass balance at the interface solid/solution.

This leads to the following expressions for the average release rate of constituent *i* at date *j* (in g·m^{-2}·d^{-1}):

$$TR_{\mathrm{i,j}} = 10 \times 24 \times SF_{\mathrm{R\_i,j}} \times C_{\mathrm{i,j}} \tag{5}$$

where

$TR_{\mathrm{i,j}}$ is the average surface-related release rate of constituent *i*, in mg·m^{-2}·d^{-1};

$SF_{R\_i,j}$ is the surface-related flow rate used in the test, in $ml \cdot cm^{-2} \cdot h^{-1}$;

$C_{i,j}$ is the measured concentration of constituent i in the collected eluate at collection period j (including the volume of the water in the reactor at the final date), in $mg \cdot l^{-1}$.

A graphical representation of the results can be documented as follows: $TR_{i,j} = f(t)$.

The calculation of the average surface-related release rate is mainly relevant for waste material of which the release is controlled by surface reactions, in particular surface dissolution. It may be used for comparison purposes between different waste materials.

10 Performance characteristics

The test specified in this Technical Specification is not validated and no data were available on robustness, repeatability and reproducibility.

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 document*;
- 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 of the solution in the reactor containing the sample holder and test portion*;

- identification to the case under consideration (8.1.1 or 8.1.2 or 8.1.3 or 8.1.4) with mention of the test conditions including flow rate, renewal rate, temperature, composition of the leachant*;
- eluate collection scheme including contact period duration*;
- stirring system/action description and characteristics*;
- 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 for each contact time for each constituent, in $mg \cdot l^{-1}$, presented in a table of results and graphically presented plotted against time*;
- the released quantities $r_{i,j}$ calculated for each contact time for each constituent, in $mg \cdot m^{-2}$ 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 $R_{i,j}$ calculated for each contact time for each constituent, in $mg \cdot m^{-2}$ geometric surface area of the monolithic waste, plotted against time. Again, if relevant, lower and upper limits shall be indicated*;
- the measured released quantities $r_{i,f}$, $mg \cdot m^{-2}$, determine for each constituent in the remaining volume of eluate in the reactor after the last eluate collection interval;
- the total cumulated release of each constituent over the global duration of the test $R_{i,t}$, $mg \cdot m^{-2}$*;
- the average surface-related release rate $TR_{i,j}$, in $mg \cdot m^{-2} \cdot d^{-1}$, for each main constituent if relevant*.

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 of this document in Bibliography [13 – 20].

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 [18].

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) [20].

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.

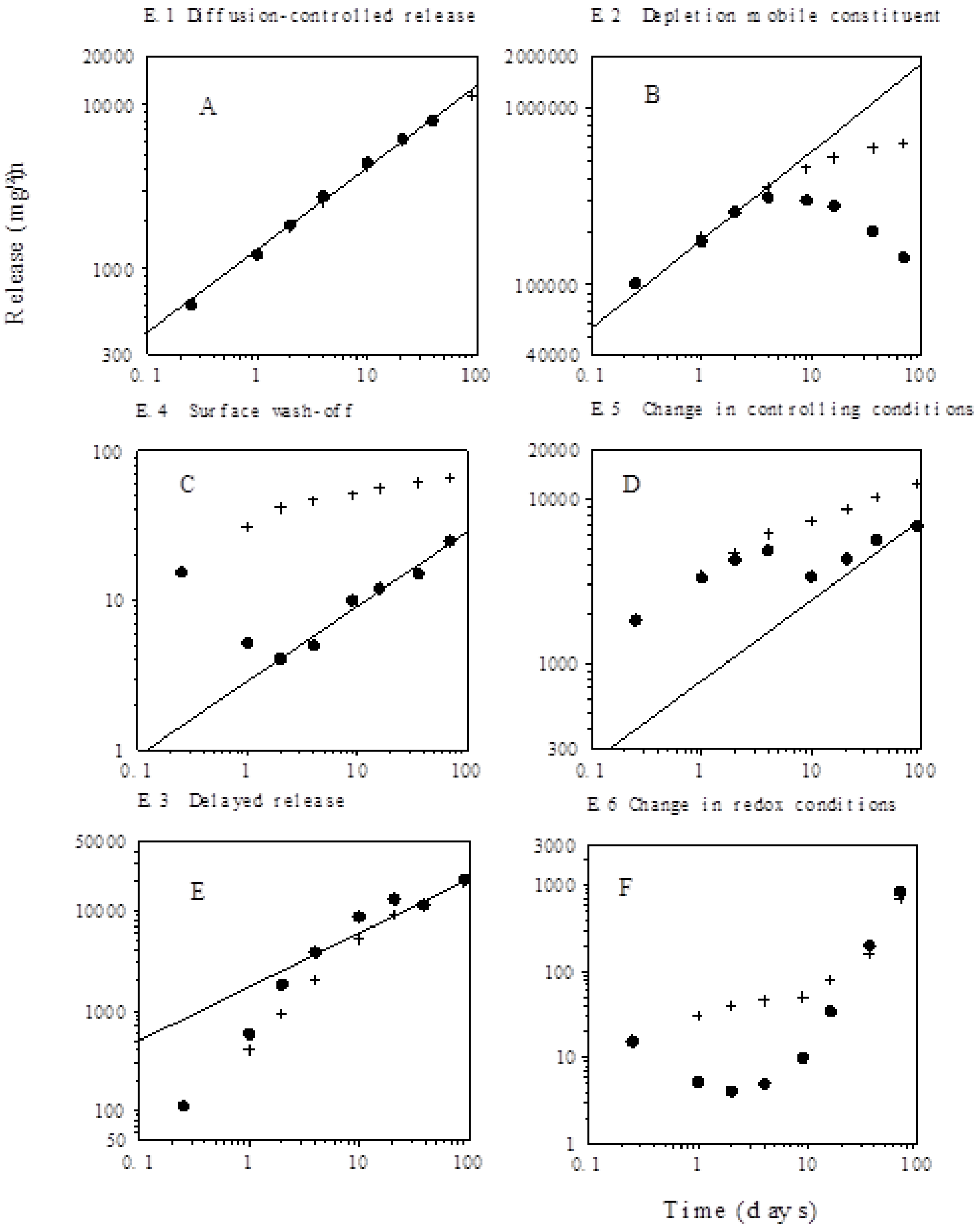
Degree of water contact – the degree of water contact is crucial for assessing impact. In dry periods, there is no transfer of substance. Depending on the degree of water saturation in the product gradient relaxation can occur from the core to the surface of the material. From some studies it has been 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 demineralized 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:

- solubility limitation;
- 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. In Figure A.1, typical patterns are shown as examples.



Key

● ● ● ● = main release behaviour, which represent release per time interval, expressed in mg·m^{-2};

+ + + + = cumulative release, expressed in mg·m^{-2};

––––– = line with slope of 0,5, representing release by diffusion (in case, or in phases of, release by diffusion the release per time interval (black dots) is parallel to the dotted line).

E1 leaching is dominated by diffusion;

E2 leaching, after short period of full diffusion ending with depletion;

E3 release by diffusion, but in the first steps far dominated by wash-off;

E4 release starts with fast release of mobile species of a substance, followed by release (by diffusion) of less-mobile species of that substance;

E5 release (by diffusion) starts delayed;

E6 change in release with time due to major changes in matrix chemistry.

NOTE Explanation:

- graphs are in a double logarithmic presentation (both X- and Y-axes);
- in double logarithmic graphs diffusion is theoretically represented by a line with a slope 0,5;
- the cumulative graph shows the total release from the start; it is possible, but less easy to derive relevant mechanism from this curve;
- in case of full solubility control, the concentration in all time intervals is the same. If a time schedule with a square root of time relation is applied this may lead wrongly to a conclusion of diffusion control. The time intervals have been adjusted to prevent this problem.

Figure A.1 — Overview of characteristic release curves for monolithic specimen, each figure showing a specific release mechanism

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 shall 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 $mg\ kg^{-1}$ 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 [16, 17].

Annex B
(normative)

Particular case "renewal rate without retro-action"

B.1 Introduction

This annex describes a particular case for the test. In this particular case, the continuous renewal is performed at a surface-related flow rate at least equal to a critical surface-related flow rate (SF_R^C) specified below.

It is then possible to study the release into the leachant of the elements without being influenced by the elements transferred into the leachant and coming from the test portion.

The minimum requirements of the main body of the document are generally applicable to this option. However, modifications and/or additions have been made to several of these minimum requirements, as described below.

The principle of this option is to use a higher surface-related flow rate than the critical surface-related flow rate, so that the elements transferred into the aqueous solution cannot affect the release. The critical surface-related flow rate is determined during a preliminary test run on a test portion, over a 24 h test period as described below.

NOTE 1 This high surface-related flow rate is not directly linked to the majority of exposure scenarios. However it could be useful to address scenarios such as monolithic waste used as construction material for a pillar placed into a canal.

NOTE 2 For vitrified waste, this high surface-related flow rate allows to define and to determine the "initial transfer rate" i.e. the constant transfer rate obtained when a first stable regime is observed when putting the test portion in contact with the aqueous solution continuously renewed according to the conditions specified in the present annex. This variable is commonly used in alteration and release models for vitreous matrices.

B.2 Determination of the critical surface-related flow rate

The temperature of the aqueous solution in the reactor is maintained at a constant value (±5 °C). The continuous renewal of the aqueous solution is secured by a continuous supply at constant surface-related flow rate SF_R (±10 % - average flow rate measured at least over 10 min) from a supply tank containing the leachant having the composition chosen for the test. At the outlet of the reactor, the eluate is collected in a collection tank in view of analysis.

To be considered as above the critical surface-related flow rate, SF_R shall fulfil the following condition: the average surface-related flow rate SF_R (see above) shall be sufficient to prevent the components released into the leachant of affecting the release, due to the concentrations obtained in the eluate. The average surface-related flow rate SF_R is deemed to be sufficient – and corresponds to the critical surface-related flow rate SF_R^C – when the quantity of each main element recovered from the collection tank at rate $SF_R = 2\ SF_R^C$ does not differ by more than 20 % from the quantity of main element recovered from the collection tank at rate $SF_R = SF_R^C$. It does not apply to those main elements for which the quantity m (mg) recovered from the collection tank corresponds to a low concentration of the leachant in contact with the test portion being tested. The concentration is deemed to be low when the m/SF_R ratio is less than 0,000 1 ($mg \cdot cm^{-2} \cdot h^{-1}$).

It shall be noted that this requirement describes the conditions under which the retro-action from the elements coming from the test portion and released into the leachant is considered to be negligible.

NOTE 1 The very nature of low-solubility compounds means that their release is extremely limited at the practicable renewal rates. They are therefore not considered in the above-mentioned requirement.

NOTE 2 The purpose of these requirements is to qualify an experimental device and operating conditions. Only if a link with a previous qualification can be demonstrated is a new qualification unnecessary.

NOTE 3 The content specified for the leachant can reflect certain scenario conditions (according to EN 12920). A combination of tests above the critical surface-related rate and at surface-related flow rates according to the scenario can be used to deconvoluate the various effects by separating the basic processes from the limiting effects that can be generated by the aqueous environment.

NOTE 4 In this preliminary test, the choice of the initial flow rate in view of determining the critical flow rate is greatly facilitated by the availability of test results on identical or similar material. Examples are given below.

— A value of 2 cm/h at constant temperature between 19 °C to 25 °C with demineralized water as leachant, has been identified as above the critical surface-related flow rate for an “artificial” cement-based monolithic waste made of sand, cement and chemical compounds as pollutants. At (30 ± 5) °C with demineralized water as leachant, a value of 10 cm/h has been identified as above the critical surface-related flow rate for the same “artificial” monolithic waste.

— For vitrified municipal solid waste incineration (MSWI) fly ash, a value of 12 cm/h at (100 ± 5) °C with demineralized water as leachant has been identified as above the critical surface-related flow rate.

The set of contact periods required to determine the leaching behaviour of the monolithic waste is specific to this option: the determination shall be performed over four or five dates (for example: 1 d, 3 d, 7 d, 10 d and 14 d). The duration until the first date shall be equivalent to at least five renewals (see 8.3, contact periods). The final date shall be such that the loss of mass from the test portion at this stage is less than or equal to 10 % of its initial mass.

The contact periods are adjusted according to the test temperature.

NOTE 5 The following dates are proposed for vitrified waste that could be reasonably tested in the 50 °C to 90 °C range. These dates could be taken as a basis for cement-based matrices.

— at 90 °C: the collection tank could be changed every 90 min, 6 times over a minimum period of 9 h on the first day (if it can be changed after 12 h, this is advisable), then again after 24 h, with the test being completed after 48 h.

— at 70 °C: the collection tank could be changed after 2 h, 4 h, 8 h, (and again, if possible, after 12 h) and then after 1 d, 2 d, 3 d, 4 d and 7 d.

— at 50 °C: the collection tank could be changed after 4 h, 10 h and then after 1 d, 2 d, 3 d, 4 d, 7 d and 10 d.

In addition to the information mentioned in Clause 11, the report shall contain the following data:

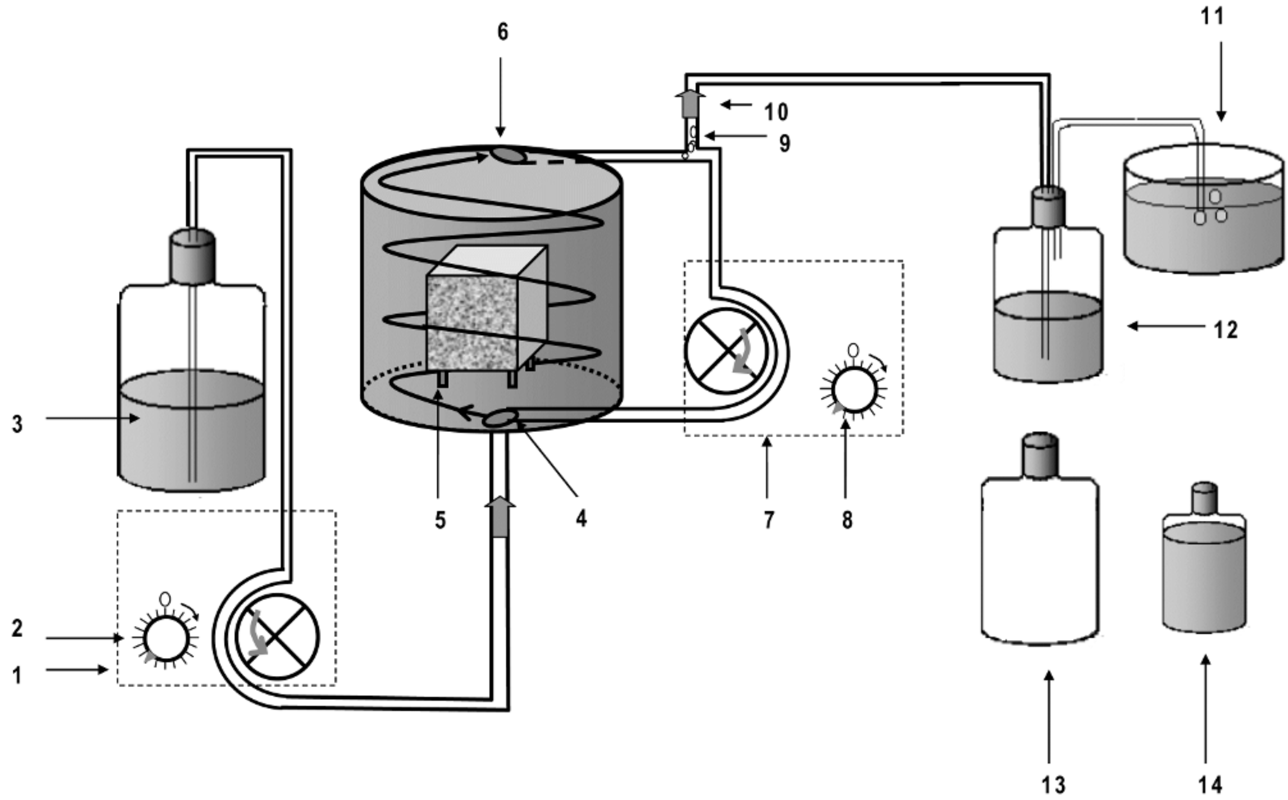
— reference to the present annex;

— surface-related flow rate values tested during the preliminary test run;

— critical surface-related flow rate SF_R^C.

Annex C
(informative)

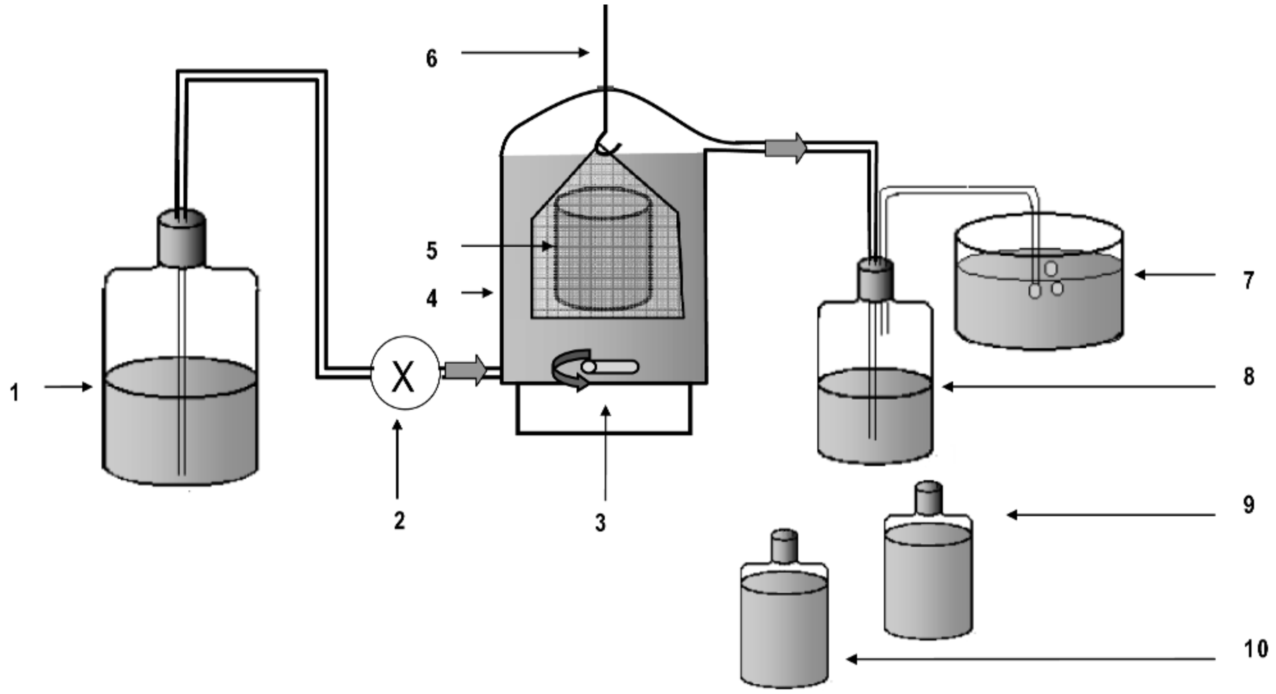
Examples of scheme of installation



Key

1	supply pump
2	flow rate adjust
3	leachant
4	peripheral input
5	stalk support
6	peripheral output
7	stirring pump
8	stirring adjust
9	bubble trap
10	eluate output
11	air lock (optional)
12	eluate 2
13	eluate 3
14	eluate 1

Figure C.1 — Installation with recirculation



Key

1	leachant
2	supply pump
3	stirring (magnetic device)
4	leaching vessel
5	test portion (in a net)
6	adjustable hook
7	air lock (optional)
8	eluate 3
9	eluate 1
10	eluate 2

Figure C.2 — Installation with magnetic stirring

Annex D
(normative)

Particular case “low renewal rate”

D.1 Introduction

This annex specifies certain minimum requirements for the particular case “low renewal rate”.

In this particular case “low renewal rate”, it is possible to study the release under “quasi-static” conditions over time with strong retro-action as a result of the low surface-related flow rate, under the conditions specified in the body of this document and in this annex.

The minimum requirements of the main body of the document are generally applicable to this option. However, modifications and/or additions have been made to several of these minimum requirements, as described below.

The principle of this option is to use the experimental system in “quasi-static” mode. The leachant in he reactor is continuously stirred. This option is used to determine whether a steady-state concentration is obtained in the leachant (which is slowly renewed), particularly for main elements. It ensures that an advanced stage of alteration is achieved under the selected conditions.

D.2 Procedure

The temperature of the aqueous solution in the reactor is maintained at a constant value (±5 °C). The continuous renewal of the aqueous solution is secured by a continuous supply at low and constant surface-related flow rate SF_R (±10 % - average flow rate measured at least over 10 min) from a supply tank containing the leachant having the composition chosen for the test. At the outlet of the reactor, the eluate is collected in a collection tank in view of analysis.

NOTE 1 A value of 0,05 cm/h at a constant temperature between 19 °C to 25 °C with demineralized water as leachant, has been identified as low surface-related flow rate for an “artificial” cement-based monolithic waste made of sand, cement and chemical compounds as pollutants.

NOTE 2 The content specified for the leachant can reflect certain scenario conditions (according to EN 12920). A combination of tests above the critical surface-related rate and at surface-related flow rates according to the scenario can be used to deconvoluate the various effects by separating the basic processes from the limiting effects that can be generated by the aqueous environment.

The set of contact periods required to determine the leaching behaviour of the monolithic waste is specific to this option: the determination shall be performed over four or five dates. The final date shall be such that the loss of mass from the test portion at this stage is less than or equal to 10 % of its initial mass.

The contact periods are adjusted according to the test temperature.

NOTE 3 The following dates are proposed for vitrified waste at (90 ± 5) °C: 1 d, 3 d, 7 d, 14 d, 28 d, 56 d and 91 d. These dates could be taken as a basis for cement-based matrices.

Perform the following operations on each eluate:

— if the test is performed at higher temperature than (30 ± 5) °C, weigh the reactor to determine the amount of solution lost by evaporation. If the total solution loss (evaporation plus total samples) exceeds 10 %, the test is cancelled;

- stop the agitation and measure the pH at the test temperature;
- take a 2 ml sample of the eluate with a syringe;
- ultra-filtrate this sample at ambient temperature. After centrifugation, dilute the sample solution with 2 ml of nitric acid 1 mol/l, then store it in an airtight container until it is analysed according to 8.5;
- weigh the reactor, replace it in the thermostatically controlled enclosure if relevant, and restart the agitation until the next date of sample collection.

NOTE 4 As it takes less than two minutes to check the pH and to take a sample of the solution, cooling or precipitate formation is avoided when the test is performed at high temperature.

The achievement of steady-state condition is checked with both pH and concentration deviation.

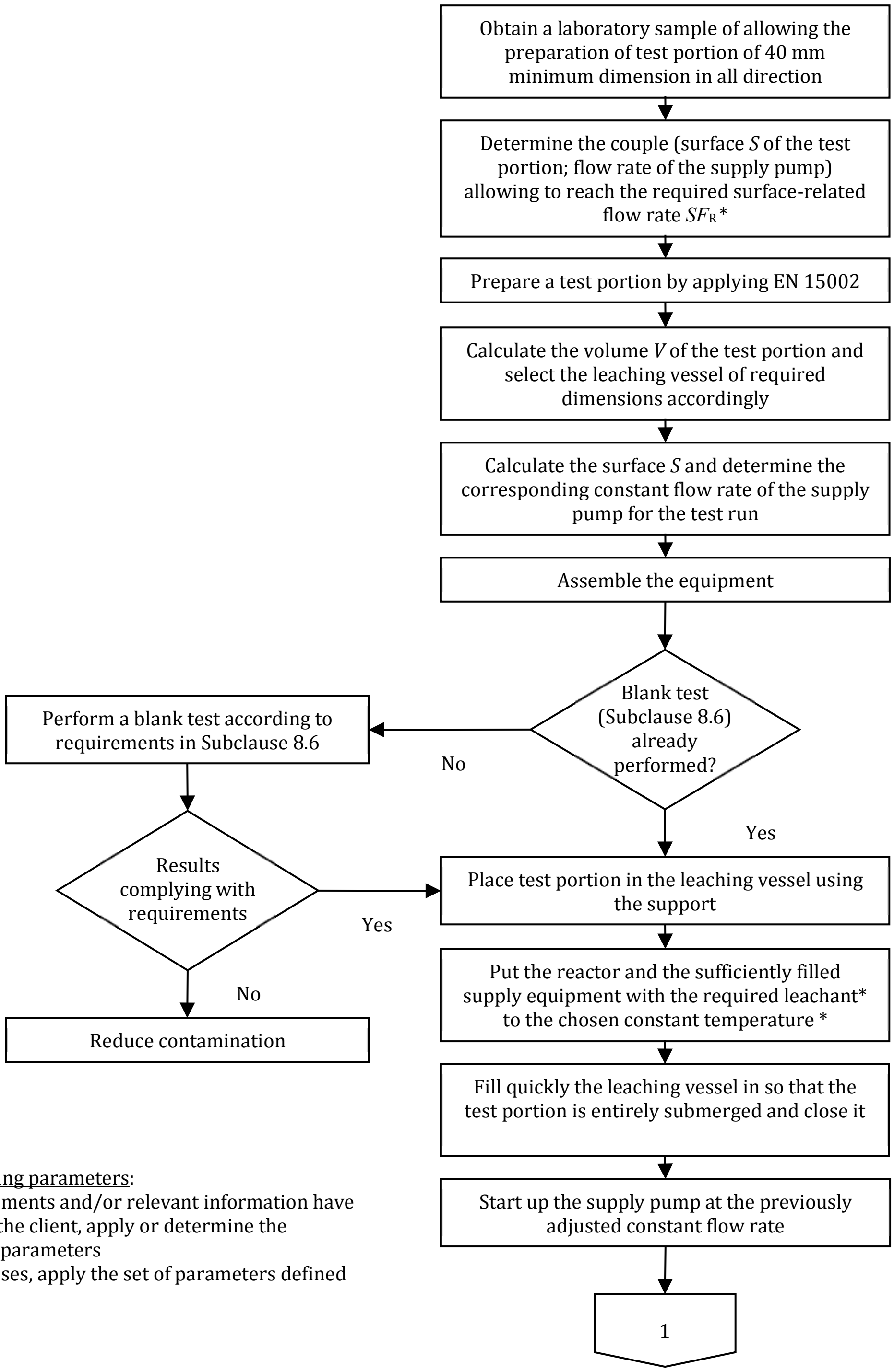
- pH value are plotted as a function of time. A statistical method is employed to determine if a steady-state condition is achieved according to pH. If this condition is achieved, record the value pH_{stat}. This statistical method consists in calculating arithmetic mean of three pH values obtained at three successive contact period. Steady-state condition is considered to be achieved if three successive pH values are included in the range [mean pH – 0,1 ; mean pH + 0,1], the subsequent pH values being included in this interval as well. The time of achievement of pH_{stat} is the lower sampling date for which this condition is achieved.
- Concentrations of the constituents of concern, especially main elements are plotted as a function of time. A statistical method is employed to determine if a steady-state condition is achieved according to constituents' concentration. If this condition is achieved, record the value for each element *i* Ci_{stat}. For each element *i*, this statistical method consists in calculating arithmetic mean of three concentration obtained at three successive contact period. For each element *i*, steady-state condition is considered to be achieved if three successive concentrations Ci are included in the range [mean Ci – 0,1 ; mean Ci + 0,1], the subsequent concentrations being included in this interval as well. The time of achievement of Ci_{stat} is the lower sampling date for which this condition is achieved.

In addition to the information mentioned in Clause 11, the report shall contain the following data:

- reference to the present annex;
- pH measured in each eluate fraction at test temperature;
- if steady-state conditions are achieved, pH_{stat} value and Ci_{stat} values and time of achievement.

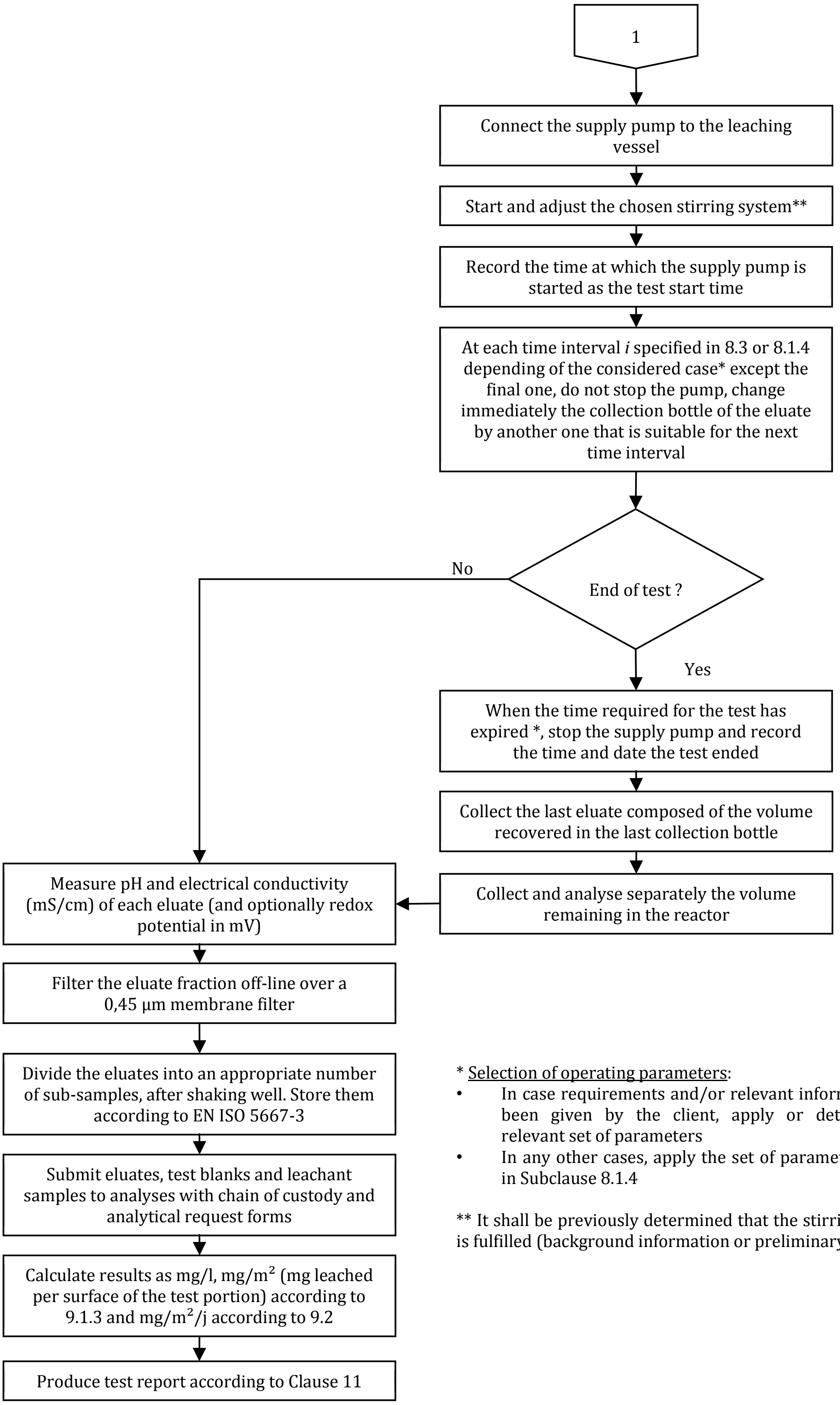
Annex E
(informative)

Process map for CEN/TS 15864



* Selection of operating parameters:

- In case requirements and/or relevant information have been given by the client, apply or determine the relevant set of parameters
- In any other cases, apply the set of parameters defined in 8.1.4



* Selection of operating parameters:

- In case requirements and/or relevant information have been given by the client, apply or determine the relevant set of parameters
- In any other cases, apply the set of parameters defined in Subclause 8.1.4

** It shall be previously determined that the stirring criterion is fulfilled (background information or preliminary test run)

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