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Characterisation of waste — Leaching behaviour test for basic characterisation — Dynamic Monolithic Leaching Test with continuous leachant renewal under conditions relevant for specified scenario(s)

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

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English Version

Characterisation of waste - Leaching behaviour test for basic characterisation - 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 Auslaugungsverhaltens für die grundlegende Charakterisierung - Dynamisches Auslaugungsverfahren für monolithische Abfälle mit kontinuierlicher Erneuerung des Auslaugungsmittels unter Bedingungen für festgelegte Szenarien

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

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Foreword

This document (CEN/TS 15864:2012) 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 [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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.

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 pressurise 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.

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.

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Introduction

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 shouldlways 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.

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 utilisation scenario under consideration.

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

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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.

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— 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. CEN/TS 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.

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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 is to be determined without any reference to a specific scenario, the leachant is demineralised 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 then 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 utilisation 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 area 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 transfer 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 12920:2006, *Characterization of waste — Methodology for the determination of the leaching behaviour of waste under specified conditions*

EN 16192, *Characterization of waste — Analysis of eluates*

EN 14346, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

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

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

EN ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the 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* (see Annex B)

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 from a leaching test

[SOURCE: EN 12457-1:2002, 3.2]

3.3
flow rate
F_R
quantity of *leachant*, expressed in volume unit, 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:1997, 3.4]

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 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 for the case described under 8.1.4 or a specific *leachant* for the cases described in 8.1.1, 8.1.2 and 8.1.3.

3.6
leachant renewal
continuous addition of *leachant* that flows through the tank as specified in 6.2.2 in an open curl

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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, 3.7]

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 $ml \cdot cm^{-2}$.

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, 3.11]

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
quantity that is representative of a certain larger quantity

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 $cm \cdot h^{-1}$.

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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 test

[SOURCE: IUPAC:1997, 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:1997, 3.19]

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.

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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 is to be determined without any reference to a specific scenario (see 8.1.4), a fixed set of parameters is to 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 dynamics 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, demineralised water, de-ionised 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.

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