SLOVENSKI STANDARD
SIST EN 15863:2015

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Karakterizacija odpadkov - Izluževalni preskus za osnovno karakterizacijo - Dinamični izluževalni preskus pri določenih pogojih za izluževanje monolitnih odpadkov z občasnim obnavljanjem izluževalnega medija

Characterisation of waste - Leaching behaviour test for basic characterisation - Dynamic monolithic leaching test with periodic leachant renewal, under fixed conditions

Charakterisierung von Abfällen - Untersuchung des Auslaugungsverhaltens für die grundlegende Charakterisierung - Dynamisches Auslaugungsverfahren für monolithische Abfälle mit periodischer Erneuerung des Auslaugungsmittels unter festgelegten Prüfbedingungen

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

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13.030.10	Trdni odpadki	Solid wastes

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

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Contents

Page

Foreword

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

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of an identical text or by endorsement, at the latest by October 2015, and conflicting national standards shall be withdrawn at the latest by October 2015.

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

This document supersedes CEN/TS 15863:2012.

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

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

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

— the status of the document has been changed from a CEN/TS into a European Standard;

— performance data has been added (see Annex E).

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

Introduction

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

1 Scope

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

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

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

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

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

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

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

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

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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

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

EN ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples (ISO 5667-3)*

3 Terms and definitions

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

3.1
eluate
solution obtained by a leaching test

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

[SOURCE: IUPAC:1990]

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, sawing, coring, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

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

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

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

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

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

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

3.6
liquid volume to surface area ratio
L/A
ratio between the amount of liquid (L) which in a given step of the test is in contact with the monolith, and the surface area of the test portion (A)

Note 1 to entry: L/A is expressed in ml·cm^{-2}.

3.7
monolithic waste
waste which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered scenario

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

3.8
release
emission of constituents from a waste, which pass through the external surface of the waste mass, as specified in the considered scenario

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

3.9
release mechanism
physico-chemical processes that control the release of constituents from a solid into solution (leaching)

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

3.10
sample
portion of material selected from a larger quantity of material

3.11
test portion
amount or volume of the test sample taken for analysis, usually of known weight or volume

[SOURCE: IUPAC:1990]

3.12
test portion of monolithic waste of regular shape
test portion of monolithic waste for which the surface area of the test portion can be calculated on the basis of simple geometric equations

3.13
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: IUPAC:1990]

3.14
waste monolith
waste delivered as bulky forms of specified minimum dimensions retaining its form in the landfill over a specified timeframe

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

4 Principle

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

The test portion of monolithic waste is placed in a reactor / leaching vessel and completely submerged in a leachant. The leachant is introduced in the reactor up to a given volume of liquid to surface area ratio (L/A ratio), at a given temperature and renewed at predetermined time intervals.

The following test conditions are fixed:

— the type of leachant;

— the temperature;

— L/A ratio (ml·cm^{-2});

— the total duration of the test;

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

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

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

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

The test conditions (L/A ratio, leachant renewal scheme) have been designed such, that the identification of leaching mechanisms and their relative importance is enabled. The main leaching mechanisms that can be distinguished and identified are:

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

— initial surface wash-off;

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

5 Reagents

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

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

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

6 Equipment

6.1 General

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

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

6.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

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

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

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

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

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

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

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

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

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

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

6.2.5 Filtering device, either a vacuum filtration device (between 30 kPa and 70 kPa) (300 mbar to 700 mbar) or a high-pressure filtration apparatus (<0,5 MPa) (5 bar).

Rinsing is compulsory.

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

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

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

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

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

6.2.9 Thermometer.

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

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

6.2.12 Redox potential meter (optional).

7 Sample preparation

7.1 General

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

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

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

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

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

7.2 Preparation of the test portion

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

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

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

7.3 Determination of the geometric surface area

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

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

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

8 Procedure

8.1 Testing conditions

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

An example of installation is illustrated in Annex C.

NOTE A constant temperature in the test can be achieved by either controlling the temperature of the lab, or by controlling the temperature of the leachant and insulating the leaching vessel (6.2.1). This property is a subject for ruggedness validation.

8.2 Step 1 of the leaching procedure

Calculate the leachant volume V_1:

$$V_1 = (8 \pm 2) \times A \quad (1)$$

where

V_1 is the volume of the leachant, in ml;

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

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

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

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

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

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

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

8.3 Steps 2 – 8 of the leaching procedure

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

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

NOTE 1 The liquid renewal frequency is designed and carried out such that the concentration in solution does not affect the release rate from the specimen significantly.