SLOVENSKI STANDARD
SIST-TS CEN/TS 15862:2013

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Karakterizacija odpadkov - Preskus skladnosti izluževanja - Enostopenjski šaržni preskus za monolitne odpadke pri določenem razmerju med prostornino tekočine in površino odpadka (L/A) za preskusne vzorce z določenimi najmanjšimi merami

Characterization of waste - Compliance leaching test - One stage batch leaching test for monoliths at fixed liquid to surface area ratio (L/A) for test portions with fixed minimum dimensions

Charakterisierung von Abfällen - Auslaugung zur Übereinstimmungsuntersuchung - Einstufiges Schüttelverfahren für monolithische Abfälle bei festgelegtem Flüssigkeit/Oberfläche-Verhältnis (L/A) für Prüfproben mit festgelegten Mindestabmessungen

Caractérisation des déchets - Essai de lixiviation de conformité - Essai de lixiviation en bâchée unique pour des monolithes avec un rapport liquide/ surface (L/A) fixe, pour des prises d'essai de dimensions minimales fixes

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This Technical Specification (CEN/TS) was approved by CEN on 6 December 2010 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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Foreword

This document (CEN/TS 15862:2012) 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 compliance testing within the EU and EFTA countries.

This document was elaborated on the basis of:

— NF X 31-211:2000;

— NEN 7345:1995;

— ÖNORM S 2116-4.

This document specifies compliance tests. For basic characterization a methodology for the determination of the leaching behaviour of waste has been developed and formulated in 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.

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 a main mechanism of release which results in a potential risk to the environment during the disposal of waste materials. 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 standard.

Procedure 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.

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 procedure described in this document is a compliance leaching test and falls in category b).

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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.

1 Scope

This Technical Specification specifies a compliance test for routine testing providing information on the leaching of monolithic waste which can be obtained under the experimental conditions specified hereafter with a single batch leaching test at a specified liquid to surface area ratio (L/A) of 12 ($cm^3 \cdot cm^{-2}$). It applies to test portion of monolithic waste of regular shape, with a minimum dimension of 40 mm in all directions, obtained e.g. by cutting, coring or moulding.

This document is not applicable if the surface area of the test portion cannot be determined by simple geometrical means.

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.

The test procedure specified in this document produces an eluate which subsequently need to be characterized physically and chemically, according to appropriate standard methods.

NOTE 1 If, in order to comply with the requirement 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.

NOTE 2 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 leaching test does not provide information by itself on dynamic leaching behaviour, as specified in EN 12920. It does not give information on equilibrium conditions. For specific situations or basic characterization, other tests are available in the toolbox of CEN/TC 292 *"Characterization of waste"*.

This document does not address issues related to health and safety.

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, *Characterisation of waste — Calculation of dry matter by determination of dry residue or water content*

EN 15002, *Characterisation 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
eluate
solution obtained from a leaching test

[SOURCE: EN 12457-1:2002, 3.1]

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

[SOURCE: IUPAC:1997, 3.2]

Note 1 to entry: When the *laboratory sample* is further prepared (reduced) by subdividing, mixing, grinding 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 sample collection 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.

3.3
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 test
test during which a material is put into contact with a leachant and some constituents of the material are extracted

[SOURCE: EN 12457-1:2002, 3.4]

3.5
liquid volume to surface area ratio
L/A
ratio between the amount of liquid (*L*), which, at any time of the test, is in contact with the waste, and the surface area of the *test portion* (*A*)

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

3.6
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 (see EN 12457-1:2002)

3.7
release
emission of constituents from waste which pass through the external surface of a waste mass as specified in the considered scenario

[SOURCE: EN 12920:2006, 3.7]

3.8
single batch leaching test
leaching test in which a fixed amount of material is leached in one step with a fixed amount of *leachant*

[SOURCE: EN 12457-1:2002, 3.8]

3.9
test portion
amount or volume of the *test sample* taken for analysis, usually of known weight or volume

[SOURCE: IUPAC, 1997, 3.9]

3.10
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

3.11
test sample
sample, prepared from the *laboratory sample,* from which *test portions* are removed for testing or for analysis

[SOURCE: IUPAC:1997, 3.11]

4 Principle

This compliance leaching test for monolithic waste consists in a single batch leaching test with a L/A ratio of 12 $cm^3 \cdot cm^{-2}$. The test portion, obtained originally or after pre-treatment, has a minimum dimension of 40 mm in all directions. The test portion is brought into contact with water as specified in 5.1 under defined conditions for a duration of 24 h. After filtration, the properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluate.

NOTE Different factors (see Annex C) can influence the results linked to the dimension-shape of the test portion (e.g. sharp edges, dimensions tolerance acceptance) or linked to the quality of the material (e.g. quality of the surface, density).

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 $mS.m^{-1}$ according to grade 2 specified in EN ISO 3696.

5.2 Nitric acid, $c(HNO_3)$ = 0,1 $mol.l^{-1}$.

6 Equipment

6.1 General

Since low concentrations of leached components in the eluate can occur, the question of contamination of and adsorption on the equipment has to be addressed. Materials in contact with the eluate in 6.2.1, 6.2.6, 6.2.10, 6.2.11, shall be of such quality that contamination and adsorption are minimized. The blank test addresses the issue of contamination. The following materials are generally considered as suitable: glass, plastics (polyethylene (PE), polytetrafluoroethylene (PTFE), polyvinylchloride (PVC), polyethyleneterephtalate (PET)) according to EN ISO 5667-3.

6.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

6.2.1 Leaching vessel or tank, glass or plastic (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, if necessary, to apply vacuum.

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).

NOTE 1 The vessel or tank may have connections to allow to apply vacuum at the beginning of the leaching procedure.

NOTE 2 The contact with the air is kept limited to avoid uptake of CO_2 from the air in case 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 B.

Supports made of inert material shall be used to allow direct contact with water on all 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 accuracy of at least 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 bars). Rinsing is compulsory.

NOTE 1 Water ejection pump generally operates in the vicinity of 50 kPa.

When volatile components are to be analysed, vacuum filtration should not be used.

6.2.6 Bottles made in material, that will not affect the analytical results or the assessment of the results in accordance with EN ISO 5667-3, with an appropriate volume (500 ml to 2 000 ml) to minimise headspace; glass bottles having caps of inert materials, for example PTFE.

NOTE For inorganic constituents high density polyethylene/polypropylene (HDPE/PP) bottles are preferred, except for samples analysed for the mercury.

6.2.7 Conductivity meter, with an accuracy of at least 0,1 $mS.m^{-1}$.

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

6.2.9 Thermometer.

6.2.10 Stirring device, magnetic bar, propeller or other (made of or coated by inert material).

6.2.11 Pre-rinsed or similarly membrane filters for filtration of the eluates, with a pore size of 0,45 µm. (e.g. rinsed with 0,1 $mol.l^{-1}$ HNO_3 (5.2) and water (5.1)).

Depending on the test requirements, PTFE filter material can be required. Filter made of PTFE are hydrophobic. Before filtration of eluate, they should be wetted using ethanol or methanol provided it doesn't influence the results of analysis.

6.2.12 Vacuum pump or a water suction device, reaching a residual pressure of 50 mm Hg (6 700 Pa) or less.

6.2.13 Redox potential meter (optional).

7 Sample preparation

7.1 Laboratory sample

For the performance of this procedure 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 C).

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 on 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. 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 Test portion

7.2.1 Preparation of the test portion:

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

NOTE 1 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.).

The choice of the procedure used shall be justified and described in the test report.

The preparation of the test portion shall prevent leaching (e.g. use of cooling fluid shall be excluded).

When the waste material results from a stabilisation process, 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.

NOTE 2 The test portion could be a laboratory prepared sample, which is prepared under conditions similar to the field situation. It could also be a part of a waste material, unless cutting results in significant differences in surface properties influencing the leaching behaviour.

7.2.2 Influence of curing time and ageing of the test portion

The date of preparation of the test portion (if prepared in the laboratory) and the date of the performing of the test shall be reported in the test report.

NOTE The age of product is an important factor that can influence the leaching properties.