SLOVENSKI STANDARD
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Karakterizacija odpadkov - Izluževalni preskus - Vpliv pH na izluževanje z uravnavanjem pH

Characterisation of waste - Leaching behaviour test - Influence of pH on leaching with continuous pH control

Charakterisierung von Abfällen - Untersuchung des Auslaugungsverhaltens - Einfluss des pH-Wertes auf die Auslaugung bei kontinuierlicher pH-Wert-Kontrolle

Caractérisation des déchets - Essais de comportement à la lixiviation - Influence du pH sur la lixiviation avec contrôle continu du pH

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Characterization of waste - Leaching behaviour test - Influence of pH on leaching with continuous pH control

Caractérisation des déchets - Essais de comportement à la lixiviation - Influence du pH sur la lixiviation avec contrôle continu du pH

Charakterisierung von Abfällen - Untersuchung des Elutionsverhaltens - Einfluss des pH-Wertes auf die Elution bei kontinuierlicher pH-Wert-Kontrolle

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 14997:2015) 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of an identical text or by endorsement, at the latest by September 2015, and conflicting national standards shall be withdrawn at the latest by September 2015.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 14997:2006.

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

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 an 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 test method for the determination of the influence of pH on the leachability of inorganic constituents from 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 will 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. The test described in this European Standard is carried out by continuous automated adjustment of pH. Size reduction is performed to facilitate approaching of equilibrium.

This test is different from the "Influence of pH on leaching with initial acid/base addition" (see EN 14429) in which the pH is controlled by addition of pre-determined amounts of acid or base to reach desired end pH values. The test is aiming at approaching equilibrium at the end of the procedure.

NOTE In Annex B specific uses of both the pH dependence test with initial acid/ base addition and the pH dependence test with continuous pH control are indicated.

1 Scope

This European Standard specifies a method for the determination of the influence of pH on the leachability of inorganic constituents from a waste material. Approaching equilibrium as defined in this European Standard is established by continuous adjustment of the pH by addition of acid or base to reach desired pH values. This test method produces eluates, which are subsequently characterized physically and chemically.

This European Standard is a parameter specific test as specified in EN 12920. The application of this test method alone is not sufficient for the determination of the detailed leaching behaviour of a waste under specified conditions.

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 14346:2006, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

EN 14899, *Characterization of waste — Sampling of waste materials — Framework for the preparation and application of a Sampling Plan*

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
dry residue
w_{dr}
remaining mass fraction of a sample after a drying process at 105 °C

[SOURCE: EN 14346:2006]

3.2
eluate
solution obtained by a leaching test

3.3
equilibrium
condition achieved when the acid or base consumption during a checking period at the last 4 h of the test is less than 2 % of the total acid or base consumption during the entire test

3.4
laboratory sample
sample or subsample(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, crushing, 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

3.6
liquid to solid-ratio
L/*S*
ratio between the amount of liquid (L) and of solid (S) in the test

Note 1 to entry: *L*/*S* is expressed in l/kg dry matter.

3.7
suspension
mixture of leachant and test portion

3.8
test portion
amount or volume of the test sample taken for analysis, usually of known weight or volume

[SOURCE: IUPAC, 1990]

3.9
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: IUPAC, 1990]

4 Symbols and abbreviations

For the purposes of this document, the following symbols and abbreviations apply.

ANC	acid neutralization capacity
BNC	base neutralization capacity
DM	dry matter
DOC	dissolved organic carbon
L/*S*	liquid to solid-ratio
M_d	dried mass of the test portion
m_d	mass after drying

M_w	un-dried mass of the test portion
m_r	mass before drying
t_0	time at the start of the leaching test
$V_{A/B}$	volume of acid or base used in leachant
V_{demin}	volume of demineralized water used in leachant
V_L	volume of added leachant
w_{dr}	dry residue of the sample

5 Principle

This European Standard describes a method to determine the influence of pH on the leachability of inorganic constituents from a waste material.

Separate test portions are leached at a fixed *L*/*S* ratio with leachants where the pH is adjusted and controlled at pre-set pH values (pH static titration procedure). At least 8 final pH values are required, covering at the minimum the range pH 2 - pH 12 (both included i.e. the lowest value ≤ 2 and the highest value ≥ 12). The amounts of acid or base needed to cover the pH range can be derived from the results of a preliminary titration, from available experimental data on the material to be tested or from an arbitrary division of the predetermined maximum consumption of acid and base. The tests are carried out at a fixed contact time at the end of which equilibrium can be assumed to be approached for most constituents in most waste materials to be characterized. The approaching of equilibrium as defined in the standard is verified at the end of the leaching procedure.

The results are expressed in mg/l of constituents for each final pH value. For each final pH value also the quantity of acid that is added is expressed in mol H^+/kg dry matter and the quantity of base that is added is expressed as mol OH^-/kg DM (for graphical presentation mol OH^-/kg DM is expressed as - mol H^+/kg DM).

NOTE Other expression of results is possible (including mg leached /kg dry matter). Since this test is aiming at approaching equilibrium i.e. solubility controlled, the results alone cannot be used to quantify the soluble mass fraction.

The ANC or BNC of the waste is also determined. The ANC or BNC is defined as the amount of acid or base (+/- mol H^+/kg dry matter) needed to reach a given user-defined end-pH. It is graphically [6] or numerically derived from a curve representing each end-pH obtained as a function of the amounts of acid or base added.

The pH range covered by the test may be restricted to a pH range relevant for the specific material and the considered problem (see 9.2).

6 Reagents

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

6.1 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.

6.2 Nitric acid, $c(HNO_3)$ = 0,1 mol/l to 5 mol/l.

6.3 Sodium hydroxide, c(NaOH) = 0,1 mol/l to 5 mol/l.

Sodium hydroxide is unstable due to possible uptake of CO_2. Therefore it is recommended to prepare a fresh solution.

7 Equipment

7.1 General

Check the materials and equipment specified in 7.2.3, 7.2.4, 7.2.7, 7.2.8 and 7.2.10 before use for proper operation and absence of interfering elements that may affect the result of the test.

Calibrate the equipment specified in 7.2.1, 7.2.6, 7.2.11 and 7.2.12.

7.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

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

7.2.2 Bottles or vessels equipped with a lid.

Use bottles or vessels (250 ml for the test portions of 15 g of dry mass, 500 ml for test portions of 30 g dry mass and 1 l for test portions of 60 g dry mass) made of polypropylene (PP), polyethylene (PE) or PTFE. In the bottles or the lid of the vessels an opening shall be present for the pH electrodes and for input of acid and/or base and a small outlet for gas. The bottles shall be as close as possible to prevent CO_2 uptake.

At high pH values it is recommended to perform the test under nitrogen to prevent CO_2 intake.

7.2.3 Crushing equipment, e.g. a jaw crusher.

NOTE Crushing is prescribed to avoid unnecessary grinding to very fine particle sizes, such as takes place in a rotary swing mill, ball mill or similar device.

7.2.4 Stirring device

The parts in contact with sample and eluate shall be made of materials not affecting the outcome of the test like glass, PTFE.

7.2.5 Membrane filters, with a pore size of 0,45 µm.

Membrane filters for the filtration device, fabricated from inert material, which is compatible with the waste. Filter shall be pre-rinsed with demineralized water or similarly clean in order to remove DOC.

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

7.2.7 Sample splitters, for sub-sampling of laboratory samples of a minimum test portion size equivalent of 15 g of dry mass.

7.2.8 Sieving equipment, with sieve of 1 mm nominal screen sizes.

Due to crushing and sieving, contamination of the sample may occur to an extent, which may affect the leached amounts of some constituents of concern, e.g. Co and W from tungsten carbide crushing equipment or Cr, Ni, Mo and V from stainless steel equipment.

7.2.9 Device for continuous pH control, with a precision of 0,2 pH units through acid/base addition.

NOTE The pH control is achieved by continuously measuring the pH of the suspension and in case of a deviation from the desired pH a continuously dosing of acid or base is required to maintain the desired pH.

7.2.10 Vacuum filtration device or pressure filtration device.

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

7.2.12 Redox potential meter (optional).

8 Sample preparation

8.1 Laboratory sample

The laboratory sample shall consist of a mass equivalent of at least 1 kg of dry mass. In case less material is available, a justification shall be provided in the test report.

Perform sampling in accordance with EN 14899 or a standard derived from EN 14899 in order to obtain a representative laboratory sample.

8.2 Preparation of the test sample

The tests shall be made on material with a grain size of 95 % less than 1 mm. In order to ensure that the test sample consists in 95 % mass of particles less than 1 mm in diameter, it shall be sieved, using the sieving equipment, to separate the oversized particles. If oversized material exceeds 5 % (mass) the entire oversized fraction shall be crushed. Any non-crushable material (e.g. metallic parts such as nuts, bolts, scrap) shall be separated from the oversized fraction and the weight and nature of the non-crushable material shall be recorded. Crushed and uncrushed material shall be mixed to constitute the test sample.

Moist material that is not possible to sieve needs to be dried prior to sieving and/or crushing. The drying temperature shall not exceed 40 °C.

Perform size reduction, drying, if needed, and sub-sampling according to specifications provided in EN 15002.

The crushed material can change upon storage due to ageing of fresh surfaces. It is therefore recommended to test the material as soon as possible after crushing.

It is recommended that materials with a high natural pH should be crushed under nitrogen in order to avoid contact with air leading to carbonation.

8.3 Determination of dry residue

The whole test sample, complying with the size criteria in 8.2 shall not be further dried. The dry residue (w_{dr}) of the test sample shall be determined on a separate test portion.

The dry residue of the sample shall be determined at 105 °C ± 3 °C according to EN 14346. The dry residue expressed as a percentage of the mass fraction is calculated as follows:

$$w_{dr} = \frac{m_d \times 100}{m_r} \quad (1)$$

where

w_{dr} is the dry residue of the sample, expressed as percentage (%);

m_d is the mass after drying , in grams (g);

m_r is the mass before drying, in grams (g).

8.4 Preparation of the test portion

Prepare at least 8 test portions by the use of a sample splitter or by coning and quartering in accordance with EN 15002. Based on sample heterogeneity and eluate volume requirement for analysis, test portion size shall be either M_d = 15 g, 30 g or 60 g (with a tolerance of ± 10 %).

Calculate the undried mass of the test portion M_w in grams to be used for the test in accordance with Formula (2):

$$M_w = \frac{M_d}{w_{dr}} \times 100 \quad (2)$$

where

M_w is the undried mass of the test portion, in grams (g);

M_d is the dry mass of the test portion, in grams (g);

w_{dr} is the dry residue of the sample, expressed as percentage (%).

9 Procedure

9.1 Contact time

The leaching procedure consists of two defined stages:

— Period A (equilibration period) from t_0 up to t_0 + 44 h for equilibration at continuously controlled pH;

— Period B (verification period) from t_0 + 44 h up to t_0 + 48 h for verification of equilibrium condition at continuously controlled pH.

Record the amount of acid or base added after each of these periods.

Measure the pH in the liquid after each of these periods.

The total contact period (A+B) is 48 h.

9.2 pH range

The test shall cover the range pH 2 to pH 12 (both included i.e. the lowest value ≤ 2 and the highest value ≥ 12) with at least 8 pH values tested including the natural pH (without acid or base addition). The maximum difference between two consecutive pH values shall not exceed 1,5 pH units.

The pH range covered by the test may be restricted to a pH range relevant for the specific material and the considered problem. The pH range to be covered may also depend on the specific properties of the waste material, the available information on this material and the questions to be answered by performing the test. The number of pH levels considered can be reduced or increased in a specific pH domain as needed. Release measured at low pH (pH = 2) can be used to estimate the potential availability for leaching, which is a relevant property for geochemical modelling.

9.3 Leaching test

9.3.1 General

The following procedure applies for each of the chosen pH values to be tested. Testing at natural pH is described in 9.4.

9.3.2 Preparation of leachant

Estimate the acid or base consumption for reaching the relevant pH values. Symbol A (mol H^+/kg dry matter) is used for the pre-estimated acid consumption and symbol B (mol OH^-/kg dry matter) is used for the pre-estimated base consumption. Use this to determine the required acid and base strength.

The acid or base consumption for the considered pH values may be derived from available information, from the preliminary procedures in Annex C or from information in Annex D.

The volume (V) of liquid consists of the water content of the test sample and the amount of leachant (V_L). Calculate the volume (V) of liquid to establish *L*/*S* = (10 ± 0,2) l/kg for the actual size of test portion M_w (see 8.4) including the volume of acid or base in accordance with Formula (3).

$$V = 10 \times M_d \tag{3}$$

where

V is the total volume of liquid in the test, in ml;

M_d is the dry mass of the test portion, in grams (g) (see 8.4).

NOTE 1 In relation to *L*/*S* -ratio, V in this formula is equivalent with the ”L” and M_d is equivalent to the “S”.

Calculate the amount of leachant (V_L) to be added to the actual size of test portion and compensate for the moisture content in the test portion, in accordance with Formula (4):

$$V_L = V - (\frac{100}{w_{dr}} - 1) \times M_d \tag{4}$$

where

V_L is the volume of added leachant, in ml;

V is the total volume of liquid in the test, in ml;

M_d is the dry mass of the test portion, in grams (g) (see 8.4);

w_{dr} is the dry residue of the waste, expressed as percentage of total weight (%) (see 8.3).

The volume of acid or base added shall not exceed a volume corresponding to M_d 1 ($V_{L/S\,1}$). This volume is calculated in accordance with Formula (5):

$$V_{L/S1} = 1 \times M_d \tag{5}$$

where

$V_{L/S\,1}$ is the volume of liquid corresponding to *L*/*S* 1 in the test, in ml;

M_d is the dry mass of the test portion, in grams (g) (see 8.4).

Calculate the minimum concentration of acid (C_A) or base (C_B) to be used as follows, with Formula (6) or Formula (7).

$$C_A = \frac{A \times M_d}{V_A} \tag{6}$$

or