



SLOVENSKI STANDARD
oSIST prEN 14997:2013
01-oktober-2013

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

Ta slovenski standard je istoveten z: prEN 14997

ICS:

13.030.01 Odpadki na splošno Wastes in general

oSIST prEN 14997:2013 **en,fr,de**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

DRAFT
prEN 14997

September 2013

ICS 13.030.01

Will supersede CEN/TS 14997:2006

English Version

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

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iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN 14997:2015

<https://standards.iteh.ai/catalog/standards/sist/a155fc03-94d3-4426-bfde-45682b6ce9b9/sist-en-14997-2015>

Foreword

This document (prEN 14997:2013) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

This document will supersede 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.

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 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.

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 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 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) 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 European Standard is applicable for determining the influence of pH on the leachability of inorganic constituents from a waste material. Approaching equilibrium as defined in this document 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 document 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.

NOTE This generally requires the application of several test methods, behavioural modelling and model validation as specified in EN 12920.

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 14899, *Characterisation 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, *Characterisation of waste – Analysis of eluates*

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods*

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

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 14346:2007]

3.2 eluate

solution obtained by a laboratory leaching test

[SOURCE: EN 12457-1:2002]

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, 1997]

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 to be mixed 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, 1997]

3.9

test sample

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

[SOURCE: IUPAC, 1997]

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
<i>L/S</i>	liquid to solid-ratio
M_d	dried mass of the test portion

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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 demineralised water used in 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. At least 8 final pH values are required, covering at the minimum the range pH 4 - pH 12 (both included i.e. the lowest value ≤ 4 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 dry matter (-mol H^+ /kg dry matter).

NOTE 1 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 [2] or numerically derived from a curve representing each end-pH obtained as a function of the amounts of acid or base added.

NOTE 2 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, demineralised water, de-ionised water or water of equivalent purity ($5 < \text{pH} < 7,5$) with a conductivity $< 0,1$ mS/m according to grade 2 specified in EN ISO 3696

6.2 Nitric acid, $c(\text{HNO}_3) = 0,1$ mol/l to 5 mol/l

6.3 Sodium hydroxide, $c(\text{NaOH}) = 0,1$ mol/l to 5 mol/l

NOTE 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 must be present for the pH-electrodes and for input of acid and/or base and a small outlet for gas. The bottles have to be as close as possible to prevent CO₂ uptake.

NOTE At high pH values it is recommended to perform the test under nitrogen to prevent CO₂ 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 demineralised 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

NOTE 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 of the suspension is achieved by continuous addition of acid/base by measuring the pH of the solution in case of a deviation from the desired pH a continuous dosing of acid or base is required.

7.2.10 Vacuum filtration device or pressure filtration device

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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 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.

NOTE 1 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.

NOTE 2 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 7.2 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 ± 5 °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 total 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.

NOTE 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 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, correspondingly.

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.