



SLOVENSKI STANDARD

SIST-TS CEN/TS 14997:2007

01-april-2007

Karakterizacija odpadkov - Izluževalni preskusi - Vpliv pH na izluževanje z uravnavanjem pH

Characterization of waste - Leaching behaviour tests - 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

<https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007>

Ta slovenski standard je istoveten z: **CEN/TS 14997:2006**

ICS:

13.030.01 Odpadki na splošno Wastes in general

SIST-TS CEN/TS 14997:2007 en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST-TS CEN/TS 14997:2007

<https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007>

ICS 13.030.01

English Version

Characterization of waste - Leaching behaviour tests - 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 Auslaugungsverhaltens - Einfluss des pH-Wertes auf die Auslaugung bei kontinuierlicher pH-Wert-Kontrolle

This Technical Specification (CEN/TS) was approved by CEN on 9 October 2006 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.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

[SIST-TS CEN/TS 14997:2007](https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007)

<https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007>



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Page

Foreword.....	3
Introduction	4
1 Scope	5
2 Normative references	5
3 Terms and definitions	5
4 Symbols and abbreviations	6
5 Principle.....	7
6 Reagents and laboratory devices	7
6.1 Reagents.....	7
6.2 Laboratory devices.....	7
7 Sampling.....	8
7.1 Laboratory sample.....	8
7.2 Test sample	8
7.3 Determination of dry residue of the sample	9
7.4 Test portion	9
8 Test procedure	9
8.1 Contact time	9
8.2 pH range	10
8.3 Leaching test.....	10
8.4 Natural pH.....	13
9 Eluate treatment, storage and analysis.....	13
10 Blank tests.....	13
11 Performance characteristics	14
12 Test report	14
Annex A (informative) Example of a data sheet	16
Annex B (informative) Operation and uses of the test: influence of pH on the leaching behaviour	17
Annex C (informative) Preliminary determination of the acid/base consumption.....	22
Annex D (informative) Examples of acid and base neutralisation capacities for waste, soil, sediment and construction materials.....	26
Bibliography.....	29

Foreword

This document (CEN/TS 14997:2006) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative Annex ZA, B, C or D, which is an integral part of this document.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST-TS CEN/TS 14997:2007](https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007)

<https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007>

Introduction

This document has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries. Tests to characterize the behaviour of waste materials can generally be divided into three categories. The relationship between these tests is summarized below:

- a) "Basic characterization" tests are used to obtain information on the short and long term leaching behaviour and characteristic properties of waste materials. Liquid/solid (*L/S*) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity, ageing of waste and physical parameters are addressed in these tests.
- b) "Compliance" tests are used to determine whether the waste complies with a specific behaviour or with specific reference values. The tests focus on key variables and leaching behaviour previously identified by basic characterization tests.
- 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 the compliance test(s). On-site verification tests are not necessarily leaching tests.

The test procedure specified in this document belongs to category (a): basic characterization tests.

In the test described in this standard an equilibrium condition is established as a result of continuous adjustment of pH. Size reduction is performed to accelerate reaching of equilibrium condition.

This test is different from the "Influence of pH on leaching with initial acid/base addition " (see CEN/TS 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 document specifies a test method for the determination of the influence of pH on the leachability of inorganic constituents from a waste material. Equilibrium condition as defined in the 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12506, *Characterization of waste – Analysis of eluates - Determination of pH, As, Ba, Cd, Cl, Co, Cr, Cr VI, Cu, Mo, Ni, NO₂⁻, Pb, total S, SO₄²⁻, V and Zn*

EN 13370, *Characterization of waste – Analysis of eluates – Determination of Ammonium-N, AOX, conductivity, Hg, phenol index, TOC, CN_{easily liberable}, F*

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

[SIST-TS CEN/TS 14997:2007](#)

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 ISO 5667-3, *Water quality – Sampling – Part 3: Guidance on the preservation and handling of water samples (ISO 5667-3:2003)*

EN ISO 3696:1995 *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

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

3.1

dry residue of the sample

W_{dr}

remaining mass fraction of a sample after a drying process at 105 °C

[EN 14346]

3.2

eluate

solution obtained by a laboratory leaching test

3.3
equilibrium condition

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

[IUPAC, 1997] [15]

3.5
liquid to solid-ratio

L/S
ratio between the amount of liquid (L) and of solid (S) in the test

NOTE *L/S* is expressed in l/kg dry matter.

3.6
leachant

liquid to be mixed with the test portion in the leaching procedure

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

[IUPAC, 1997]

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST-TS CEN/TS 14997:2007
https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007](https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007)

3.9
test sample

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

[IUPAC, 1997]

4 Symbols and abbreviations

ANC	acid neutralization capacity
BNC	base neutralization capacity
DOC	dissolved organic carbon
<i>L/S</i>	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 demineralised water used in leachant
w_{dr}	dry residue of the sample

5 Principle

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 condition can be assumed to be reached for most constituents in most waste materials to be characterized. The equilibrium condition 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 equilibrium condition 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 8.2).

<https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-c599f0589215/sist-ts-cen-ts-14997-2007>

6 Reagents and laboratory devices

6.1 Reagents

6.1.1 Distilled water, demineralised water or water of equivalent purity with a conductivity < 0,1 mS/m [ISO 3696].

6.1.2 Nitric acid (pro analysis), 0,1 mol/l to 5 mol/l.

6.1.3 Sodium hydroxide, NaOH, 0,1 mol/l to 5 mol/l.

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

6.2 Laboratory devices

6.2.1 Analytical balance (accurate to 0,1 g).

6.2.2 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 CO_2 uptake.

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

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

NOTE Crushing is prescribed in 7 to avoid unnecessary grinding to very fine particle sizes, such as takes place in a rotary swing mill, ball mill or similar device.

6.2.4 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.

6.2.5 Membrane filters for the filtration device.

Use filters or a filter device, fabricated from inert material, which is compatible with the waste and has a pore size of 0,45 µm. Filter must be pre-washed with demineralized water or similarly clean in order to remove DOC.

6.2.6 pH meter.

Use a pH meter with a measurement accuracy of at least ± 0,05 pH units.

NOTE At high pH values the use of specific electrodes is recommended.

6.2.7 Sample splitters for a minimum test portion size equivalent of 15 g of dry mass.

6.2.8 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.

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

<https://standards.iteh.ai/catalog/standards/sist/85af63e5-ed22-4125-83ad-59a9e9c1c10f>

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.

6.2.10 Vacuum filtration device or pressure filtration device.

7 Sampling

7.1 Laboratory sample

Perform sampling in accordance with EN 14899 in order to obtain a representative laboratory sample.

The laboratory sample shall consist of a mass equivalent of at least 1 kg of dry mass.

7.2 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.

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.

7.3 Determination of dry residue of the sample

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 $105\text{ °C} \pm 5\text{ °C}$ according to EN 14346. The dry residue expressed as a percentage of the mass fraction is calculated as follows:

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

where

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

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

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

7.4 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 $M_d = 15\text{ g}$, 30 g or 60 g (with a tolerance of $\pm 10\%$).

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

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

where

M_w is the total mass of the test portion expressed in grams (g);

M_d is the dry mass of the test portion expressed in grams (g).

8 Test procedure

8.1 Contact time

The leaching procedure consists of two defined stages:

- Period A (equilibration period) from t_0 up to $t_0 + 44\text{ h}$ for equilibration at continuously controlled pH;
- Period B (verification period) from $t_{0+44\text{ h}}$ up to $t_0 + 48\text{ h}$ for verification of equilibrium condition at continuously controlled pH.

The amount of acid or base added is recorded after each of these periods.

The pH in the liquid is recorded after each of these periods.

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

8.2 pH range

The test shall cover the range pH 4 to pH 12 (both included i.e. the lowest value ≤ 4 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.

8.3 Leaching test

8.3.1 General

The following procedure applies for each of the chosen pH values to be tested. Testing at natural pH is described in 8.4.

8.3.2 Preparation of leachant

Identify the acid or base consumption for reaching the relevant pH values as A (mol H⁺/kg dry matter) or B (mol OH⁻/kg dry matter).

NOTE 1 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 $L/S = (10 \pm 0.2)$ l/kg for the actual size of test portion M_w (see 7.4) including the volume of acid or base in accordance with Equation 3.

$$V = 10 \times M_d \quad (3)$$

where

V is the total volume of liquid in the test expressed in millilitres (ml);

M_d is the dry mass of the test portion expressed in grams (g) (see 7.4).

NOTE 2 In relation to L/S -ratio, V in this equation 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 Equation 4:

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

where

V_L is the volume of added leachant expressed in millilitres (ml).

V is the total volume of liquid in the test expressed in millilitres (ml);

w_{dr} is the dry residue of the waste expressed as percentage of total weight (%) (see 7.3);