SLOVENSKI STANDARD
SIST-TS CEN/TS 14405:2004

01-oktober-2004

Karakterizacija odpadkov - Izluževalni preskusi - Preskus v koloni s tokom navzgor (pri določenih pogojih)

Characterization of waste - Leaching behaviour tests - Up-flow percolation test (under specified conditions)

Charakterisierung von Abfällen - Auslaugungsverhalten - Perkolationsprüfung im Aufwärtsstrom (unter festgelegten Bedingungen)

Caractérisation des déchets - Essais de comportement a la lixiviation - Essai de percolation a écoulement ascendant (dans des conditions spécifiées)

Ta slovenski standard je istoveten z: **CEN/TS 14405:2004**

ICS:

13.030.01 Odpadki na splošno Wastes in general

SIST-TS CEN/TS 14405:2004 **en**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 14405

June 2004

ICS 13.030.10

English version

Characterization of waste - Leaching behaviour tests - Up-flow percolation test (under specified conditions)

Caractérisation des déchets - Essais de comportement à la lixiviation - Essai de percolation à écoulement ascendant (dans des conditions spécifiées)

Charakterisierung von Abfällen - Auslaugungsverhalten - Perkolationsprüfung im Aufwärtsstrom (unter festgelegten Bedingungen)

This Technical Specification (CEN/TS) was approved by CEN on 24 March 2004 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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

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

Ref. No. CEN/TS 14405:2004: E

Contents

Foreword

This document (CEN/TS 14405:2004) has been prepared by Technical Committee CEN/TC 292 “Characterisation of waste”, the secretariat of which is held by NEN.

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

This Technical Specification was elaborated on the basis of:
NEN 7343:1995
NT ENVIR 002:1995

This Technical Specification specifies an up-flow percolation test to determine the leaching behaviour of granular waste materials under standardized percolation conditions. Another Technical Specification will be produced to specify a percolation test for simulating conditions of specific scenarios.

For a more complete characterization of the leaching behaviour of waste under specified conditions the application of other test methods is required (see ENV 12920). For informative references see the Bibliography.

The annexes A, B and C are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this 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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom

Introduction

In various European countries tests have been developed to characterize and assess the constituents that can be released 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 re-use or disposal of waste materials. These tests are intended 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.

Tests to characterize the behaviour of waste materials can be divided into three categories. The relationships between these tests are summarized below:

“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.

“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.

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

The test procedure described in this method belongs to category (1): basic characterization tests.

1 Scope

This Technical Specification is applicable to determine the leaching behaviour of inorganic constituents from granular waste (without or with size reduction (see **6.2**)). The waste body is subjected to percolation with water as a function of liquid to solid ratio under specified percolation conditions. The waste is leached under hydraulically dynamic conditions. The method is a once-through column leaching test and the test results establish the distinction between different release patterns, for instance wash-out and release under the influence of interaction with the matrix, when approaching local equilibrium between waste and leachant.

NOTE 1 The mentioned specified percolation conditions are arbitrary and are not simulating a specific scenario.

NOTE 2 Waste materials that show a saturated hydraulic conductivity between 10^{-7} m/s and 10^{-8} m/s can be subjected to this test, but it can be difficult to maintain the imposed flow rate. If a waste shows a saturated hydraulic conductivity below 10^{-8} m/s, the test should not be carried out. (See C.5 for a definition of ‘hydraulic conductivity’.)

NOTE 3 This procedure is generally not applicable to biologically degrading materials and materials reacting with the leachant, leading, for example, to excessive gas emission or excessive heat release.

NOTE 4 This procedure is applicable to materials showing solidification in the column, if the final hydraulic conductivity is within the specified range (see NOTE 1).

2 Normative references

This Technical Specification incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Technical Specification only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 12506, *Characterization of waste – Analysis of eluates – Determination of pH, As, Ba, Cd, Cl⁻, Co, Cr, Cr^{VI}, Cu, Mo, Ni, NO_2^-, Pb, total S, SO_4^{2-}, V and Zn.*

EN 13370, *Characterization of waste – Analysis of eluates – Determination of Ammonium, AOX, conductivity, Hg, phenol index, TOC, easily liberatable CN^-, F^-.*

prEN 14346:2002, *Characterization of waste – Calculation of dry matter by determination of dry residue or water content.*

prEN 14899, *Characterisation of Waste - Sampling of waste materials: Framework for the preparation and application of a Sampling.*

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

3 Terms and definitions

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

3.1
dry residue w_{dr}
remaining mass fraction in percent of a sample after a drying process at 105 °C

[prEN 14346:2002]

3.2
eluate
solution obtained by a laboratory leaching test

3.3
equilibrium condition
condition achieved when the pH deviation during a checking period is below a specified value (see **7.4**)

3.4
granular waste
solid waste that is neither monolithic, liquid, gas nor sludge

3.5
laboratory sample
sample or sub sample(s) sent to or received by the laboratory

[IUPAC, 1997]

3.6
leachant
liquid that is brought into contact with the test portion in the leaching procedure

3.7
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.

3.8
sample
quantity that is representative of a certain larger quantity

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

3.10
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

[IUPAC, 1997]

4 Principle

This Technical Specification describes a method to determine the release of constituents from waste, with or without size reduction, packed in a column with a leachant percolating through it. A continuous vertical up-flow is used, so that the column is water saturated. The test conditions, including the flow rate of the leachant, enable a conclusion to be drawn from the results as to which components are rapidly being washed out and which components are released under the influence of interaction with the matrix.

The test portion of the waste to be tested is packed in a column in a standardized manner. The leachant is percolated in up-flow through the column at a specified flow rate up to a fixed L/S ratio. The eluate is collected in several separate fractions that are characterized physically and chemically according to existing standard methods. In the test, equilibrium conditions at the outlet of the column are verified after an equilibration period by measuring a pH deviation.

The results of the test are expressed as a function of L/S ratio, in terms of both mg of the constituents released per litre of eluate, and mg of the constituents released cumulatively per kg of waste material (dry matter).

5 Equipment and reagents

5.1 General

The materials and equipment specified in **5.3** to **5.16** shall be checked before use for proper operation and absence of interfering elements that may affect the result of the test (see **7.7**).

The equipment specified in **5.4**, **5.5, 5.11** and **5.14** shall also be calibrated.

5.2
Column made of glass or plastics (e.g. PMMA, PTFE) with an internal diameter of 5 cm or 10 cm and a filling height of about 30 cm ± 5 cm, fitted with filters (see **5.9**) in bottom and top section. In the top and bottom sections of the column a filter plate or a thin layer of fine-grained non-reactive material (e.g. fine quartz sand) is applied to ensure proper water flow over the width of the column and as a support for the pre-filter.

NOTE An illustration of the column and its accompanying equipment is given in annex A.

5.3
Adjustable volumetric pump (peristaltic or equivalent). For small columns, running at a linear velocity of 15 cm/day, the capacity shall be adjustable between 10 ml/h and 20 ml/h and for wide columns, running at the same linear velocity, the capacity shall be adjustable between 40 ml/h and 60 ml/h.

5.4
Analytical balance with an accuracy of at least 0,1 g.

5.5
Conductivity meter with an accuracy of at least 0,1 mS/m.

5.6
Crushing equipment: a jaw crusher or a cutting device.

5.7
Demineralized water with a conductivity of a maximum of 0,1 mS/m.

5.8
Glass or plastics (e.g. HDPE / PP / PTFE / PET) 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).

5.9
Membrane filters for in-line or off-line filtration of the eluates, with a pore size of 0,45 µm.

5.10
Nitric acid (pro analyse), 1 mol/l.

5.11
pH meter with an accuracy of at least 0,05 pH units.

5.12
Plastic hose material.

5.13
Pre-filters for the column with a pore size of 1,5 µm to 8 µm.

NOTE If the eluate is to be used for bio-assays or other specific situations, in which fine particles are important, pre-filters with a larger pore size should be used.

5.14
Redox potential meter (optional).

5.15
Sample splitter for sub-sampling of laboratory samples (optional).

5.16
Sieving equipment with sieves of 4 mm and 10 mm nominal screen size.

6 Sample pre-treatment

6.1 General

Sampling shall be performed in accordance with WI 00292001 or a standard derived from WI 00292001, in order to obtain a representative sample. Sample pre-treatment shall consist of sample preparation, taking a test portion and determining the dry residue of the test sample.

NOTE If it is the intention to determine the leaching behaviour of a waste material, including ageing effects, the test portion should be representative for the material in the practical situation, also as far as ageing conditions are concerned.

6.2 Sample preparation

A laboratory sample shall be obtained of at least 2,5 kg of the material if a small column (diameter 5 cm) is to be used and of at least 10 kg, if a wide column (diameter 10 cm) is to be used. The laboratory sample shall be stored in closed packages and kept under conditions, which prevent any alterations of the waste material.

NOTE 1 Storage at low temperatures (4 °C) may be required, in order to minimise unwanted alterations of the waste material.

To investigate whether the material has to be crushed and to investigate which type of column is to be used, the laboratory sample shall be sieved (see **5.16**).

The use of the small or wide column shall depend on the particle size of the material, according to Table 1.

Table 1 - Use of column type, related to particle size of waste

Fraction < 4 mm	Fraction ≥ 10 mm	Column to be used (with required size-reduction)
≥ 95 % (*m*/*m*)		Small column (without size reduction), or Wide column (without size reduction)
80 % (*m*/*m*) to 95 % (*m*/*m*)	≤ 5 % (*m*/*m*)	Small column (with size reduction of the fraction ≥ 4 mm), or Wide column (without size reduction)
≤ 80 % (*m*/*m*)	≤ 5 % (*m*/*m*)	Wide column (without size reduction)
	> 5 % (*m*/*m*)	Wide column (with size reduction of the fraction ≥ 10 mm

Prepare a test sample. Use a sample splitter (see **5.15**) or apply coning and quartering to split the laboratory sample.

The test shall be carried out preferably on a sample in the condition (particle size, moisture content) as it was delivered to the laboratory.

If oversized material is to be size reduced (in accordance with Table 1), the entire oversized fraction shall be crushed with a crushing device (see **5.6**). On no account shall the material be finely ground. Non-crushable material (e.g. metallic parts such as nuts, bolts, scrap) in the sample shall be separated and the mass and the nature of the material shall be recorded.

If the sample cannot be sieved or split or crushed because of its moisture content, it is allowed to dry the sample, but no further than is required for the sample preparation. The drying temperature shall not exceed 40 °C.

NOTE 2 Depending on the maximum particle size, the splitting can require size reduction of the coarser particles to comply with the rules of sampling.

NOTE 3 Due to crushing and sieving, contamination of the sample can occur to an extent, which is affecting the release 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.

NOTE 4 Fibrous materials and plastics can often be size-reduced only after cryogenic treatment.

NOTE 5 Drying may lead to oxidation and/or carbonation. If the material is fresh and has to be tested as a non oxidized / non carbonated material, the drying should be conducted in an inert atmosphere.

6.3 Test portion

At least two test portions shall be taken from the test sample. To fill the column completely, the quantity of one of the test portions shall be such that its volume after compaction is 0,6 l (in case a column with a diameter of 5 cm is used), or 2,4 l (in case a column with a diameter of 10 cm is used). A sample splitter (see **5.15**) shall be used or coning and quartering shall be applied to split the test sample and prepare the test portion(s).

6.4 Determination of dry residue

The dry residue of the test sample shall be known and taken into account when calculating the L/S ratio.

Determine the dry residue (w_{dr}) at 105 °C ± 5 °C according to prEN 14346, on a separate test portion. Calculate the dry residue in accordance with equation 1.

$$w_{dr} = m_d \times 100 / m_r \quad [\%] \qquad (1)$$

where

w_{dr} dry residue of the waste expressed as percentage [%];

m_r mass of the undried test portion expressed in grams [g];

m_d dry mass of the test portion expressed in grams [g].

7 Procedure

7.1 Temperature

Carry out the up-flow percolation test at a temperature of 20 °C ± 5 °C.

NOTE A constant temperature of 20 °C in the test can be achieved by either controlling the temperature of the lab, or controlling the temperature of the leachant and insulating the column and accompanying equipment.

7.2 Preparation

Rinse the column, including top and bottom sections and filters (see **5.2**) and bottles (see **5.8**) with nitric acid (see **5.10**) and water (see **5.11**) consecutively. Weigh the dry column, including top and bottom sections, filters and filter plates or layers of fine-grained material, to an accuracy of 1 g.

7.3 Packing of the column

Fit the bottom section, equipped with a filter plate or a layer of fine-grained chemically inert material (e.g. fine quartz sand) of approximately 1 cm and a pre-filter (see **5.13**) to the column. Fill the column with the test portion, up to a bed height of 30 cm ± 5 cm, in at least five consecutive layers, as follows:

- Introduce each layer into the column in three sub-layers and level each sub-layer separately.
- Pack each layer using as a rammer a weight of 125 g in case of a column with a diameter of 5 cm, and of 500 g in case of a column with a diameter of 10 cm. Drop the weight three times on each layer falling down 20 cm along a rod used as a guide. Fix this rod to the centre of a disk, which is placed on the layer to be packed. Cover the whole surface of the column with the disk (as is shown in Figure 1).
- For the last layer, check the remaining height and adjust the necessary mass in order to get 30 cm ± 5 cm.