

**Karaktärisering av avfall – Bestämning av
lakegenskaper – pH-inverkan på lakning vid
initial tillsättning av syra/bas**

**Characterization of waste – Leaching behaviour
tests – Influence of pH on leaching with initial
acid/base addition**

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TECHNICAL SPECIFICATION
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English Version

**Characterization of waste - Leaching behaviour tests - Influence
of pH on leaching with initial acid/base addition**

Caractérisation des déchets - Essais de comportement à la
lixiviation - Influence du pH sur la lixiviation avec ajout initial
d'acide/base

Charakterisierung von Abfällen - Untersuchung des
Auslaugungsverhaltens - Einfluss des pH-Wertes unter
vorheriger Säure/Base Zugabe

This Technical Specification (CEN/TS) was approved by CEN on 11 February 2005 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.

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Foreword

This CEN Technical Specification (CEN/TS 14429:2005) has been prepared by Technical Committee CEN/TC 292 "Characterisation of waste", the secretariat of which is held by NEN.

The annexes A, B, C and D are informative.

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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

CEN/TS 14429:2005 (E)**Introduction**

This Technical Specification 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 are summarized below:

1. "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.
2. "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.
3. "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 described in this Technical Specification belongs to category (1): basic characterization tests.

In the test, which is described in this Technical Specification equilibrium condition is established at different pH values as a result of the reaction between pre-selected amounts of acid or base and test portions of the waste material. Size reduction is performed to accelerate reaching of equilibrium condition.

This test is different from the "pH dependence test with continuous pH control" (pH static test see prEN 14997) in which the pH is controlled at pre-selected values over the entire testing period by continuous measurement and automatic addition of acid or base. 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 Technical Specification is applicable to determine the influence of pH on the leachability of inorganic constituents from a waste material. Equilibrium condition as defined in the standard is established by addition of pre-determined amounts of acid or base to reach desired end pH values. This test method produces eluates, which are subsequently characterized physically and chemically.

This Technical Specification is a parameter specific test as specified in ENV 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 ENV 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 12506:2003, *Characterization of waste – Analysis of eluates – Determination of pH, As, Ba, Cd, Cl-, Co, Cr, CrVI, Cu, Mo, Ni, NO₂-, Pb, total S, SO₄²⁻ and Zn.*

ENV 12920, *Characterization of waste – Methodology for the determination of the leaching behaviour of waste under specified conditions.*

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 and water content.*

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

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 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 at the last 4 hours of the test is below 0,3 pH unit

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3.4

laboratory sample

sample or subsample(s) sent to or received by the laboratory

[IUPAC, 1997]

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]

3.9

test sample

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

[IUPAC, 1997]

4 Symbols and abbreviations

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_f 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 waste

5 Principle

Separate test portions are leached at a fixed L/S ratio with leachants containing pre-selected amounts of acid or base in order to reach stationary pH values at the end of the extraction period. Each leachant is added in three steps in the beginning of the test. At least 8 final pH-values are required, covering at the minimum the range pH 4 to 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 extraction period.

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 DM and the quantity of base that is added is expressed as negative mol OH^- /kg DM (-mol OH^- /kg DM).

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 acid or base neutralization capacity (ANC, BNC) of the waste is also determined.

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).

6 Reagents and laboratory devices

6.1 Reagents

6.1.1

Distilled water, demineralized water or water of equivalent purity with a conductivity $< 0,1$ mS/m

6.1.2

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

6.1.3

Nitric acid (pro analysis), 1 mol/l

6.1.4

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

6.2 Laboratory devices

6.2.1

Agitation device: end-over-end tumbler (5 r/min to 10 r/min) or roller table rotating at about 10 r/min

6.2.2

Analytical balance (accurate to 0,1 g)

6.2.3

Bottles (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) or polyethylene (PE).

6.2.4

Crushing equipment, e.g. a jaw crusher

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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.

6.2.5

Membrane filters for the filtration device, fabricated from inert material, which is compatible with the waste and has a pore size of 0,45 µm. Filter shall be pre-rinsed with demineralised water or similarly clean in order to remove DOC.

6.2.6

pH meter with a measurement accuracy of at least $\pm 0,05$ 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 1mm nominal screen sizes

NOTE Due to crushing and sieving, contamination of the sample may occur to an extent, which can 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

Vacuum filtration device or pressure filtration device

7 Sampling**7.1 Laboratory sample**

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

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

7.2 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 (see **6.2.8**), 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 There is no obligation to sieve if it is obvious that the material will fully pass the sieve on 1 mm.

NOTE 2 The crushed material can change upon storage due to ageing of fresh surfaces. It is therefore preferable to test the material as soon as possible after crushing.

NOTE 3 It is recommended that materials with a high natural pH be crushed under nitrogen in order to avoid contact with air leading to carbonation.

7.3 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 shall be determined at $105\text{ °C} \pm 5\text{ °C}$ according to prEN 14346. The dry residue is calculated as follows:

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

where

w_{dr} is the dry residue of the waste expressed as a 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. 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 to be used for the test as follows:

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

where

M_d is the dried mass of the test portion expressed in grams (g);

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

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

8 Test procedure

8.1 Contact time

The leaching procedure consists of three defined stages:

- Period A (acid/base addition) from t_0 up to $t_0 + 4\text{ h}$ for acid/base addition in three steps;
- Period B (equilibration period) from $t_{0+4\text{ h}}$ up to $t_0 + 44\text{ h}$ equilibration period;
- Period C (verification period) from $t_{0+44\text{ h}}$ up to $t_0 + 48\text{ h}$ for verification of equilibrium condition.

Measure pH in the liquid after each of these periods.

The total contact period (A+B+C) 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.