

**Characterization of waste – Leaching behaviour
tests – Acid and base neutralization capacity test**

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TECHNICAL SPECIFICATION
SPÉCIFICATION TECHNIQUE
TECHNISCHE SPEZIFIKATION

CEN/TS 15364

April 2006

ICS 47.020.30

English Version

**Characterization of waste - Leaching behaviour tests - Acid and
base neutralization capacity test**

Caractérisation des déchets - Essais de comportement à la
lixiviation - Essai de capacité de neutralisation des acides
et des bases

Charakterisierung von Abfällen - Untersuchung des
Auslaugungsverhaltens - Prüfung der Säure- und Base-
Neutralisierungskapazität

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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	6
6 Reagents and laboratory devices.....	7
7 Sampling	7
8 Test procedure.....	9
9 Performance characteristics.....	12
10 Test report.....	12
Annex A (informative) Example of a data sheet	13
Annex B (informative) Preliminary determination of the acid/base consumption	14
Annex C (informative) Examples of acid and base neutralisation capacities for waste, soil, sediment and construction materials and use of the data	18
Annex D (informative) Examples of steps in the full evaluation of ANC and BNC for waste materials	20
Bibliography.....	21

Foreword

This Technical Specification (CEN/TS 15364:2006) has been prepared by Technical Committee CEN/TC 292 "Characterisation of waste", the secretariat of which is held by NEN.

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.

CEN/TS 15364:2006 (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 behavior 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 one 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.

NOTE When sufficient information is available for a material type or class, a single measurement to a relevant final pH may suffice. In that case, the procedure would qualify as a category 2 method.

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 method to assess the influence of pH on leaching with initial acid/base addition, in that the chemical analysis of inorganic constituents in eluates produced is not carried out.

The neutralization capacity can also be derived from a test addressing the influence of pH on leaching by continuous pH control as described in prEN 14997. The test addressing the influence of pH on leaching by continuous pH control can be particularly suitable in the case of measurement of pH influence on leaching at a pH where a small pH change leads to strong change in release.

1 Scope

This Technical Specification is applicable to determine the acid and/or base neutralization capacity of a material. The property is expressed in mol/kg. This Technical Specification 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. The property allows assessing the final pH of mixed waste as well as consequences of external influences (carbonation, oxidation) on the pH of a waste.

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

2 Normative references

The following referenced documents are indispensable for the application of this Technical Specification. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

prEN 14346:2004, *Characterization of waste – Calculation of dry matter by determination of dry residue and water content*

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

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 14346:2004]

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 h of the test is below 0,3 pH unit

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

CEN/TS 15364:2006 (E)**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
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 de-mineralized water used in leachant
V_L	volume of added leachant
w_{dr}	dry residue of the waste

5 Principle

Separate test portions are prepared 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). From the amounts of acid or base needed to cover the pH range the acid or base neutralization capacity (ANC, BNC) of the test material is determined. The equilibrium condition as defined in 8.1 is verified at the end of the extraction period.

For each final pH value 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 H⁺/kg DM (-mol H⁺/kg DM). The net mol/kg (+ for acid - for base) is presented as a function of pH.

NOTE 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

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

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.

6.2.6

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

6.2.7

Sieving equipment with sieve of 1 mm nominal screen sizes

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 EN 14899 or a standard derived from EN 14899 in order to obtain a representative laboratory sample.

CEN/TS 15364:2006 (E)

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 6.2.7), 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 °C ± 5 °C according to prEN 14346. The dry residue is calculated as follows:

$$w_{dr} = 100 \times \frac{m_d}{m_r} \tag{1}$$

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 (6.2.6) or by coning and quartering. 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 to be used for the test as follows:

$$M_w = \frac{M_d}{w_{dr}} \times 100 \tag{2}$$

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

NOTE 1 To ensure that the appropriate pH values can be obtained in one run additional bottles can be prepared.

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. The pH range to be covered can depend on the specific properties of the 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 Acid-, base neutralization capacity test

8.3.1 General

The following procedure applies for each of the chosen pH values to be tested.

8.3.2 Preparation of leachant

Estimate the acid or base consumption needed to reach the relevant pH values as A (mol H^+ /kg DM) or B (mol H^+ /kg DM).

NOTE 1 An estimate of the acid or base consumption for the considered pH values may be derived from available literature information, from the preliminary procedures in Annex B or from information in Annex C.

Calculate the volume V of liquid to establish $L/S = 10 \text{ l/kg} \pm 0,2 \text{ l/kg}$ for the actual size of test portion M_w (see 7.4) including the volume of acid or base.

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

where

V is the total volume of liquid in the test in 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 to be added to the actual size of test portion and compensate for the moisture content in the test portion, as follows: