

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

ICS 47.020.30

National foreword

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Notification of the start of the review period, with a request for the submission of comments from users of this Draft for Development, will be made in the appropriate issue of *Update Standards*. According to the replies received, the responsible BSI Committee will judge whether the Draft for Development can be converted into a British Standard or what other action should be taken.

Observations which it is felt should receive attention before the official call for comments will be welcomed. These should be sent to the Secretary of BSI Technical Committee B/508/3, Characterisation of waste, at British Standards House, 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

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Summary of pages

This document comprises a front cover, an inside front cover, the CEN/TS title page, pages 2 to 21 and a back cover.

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Amendments issued since publication

Amd. No.	Date	Comments

This Draft for Development was published under the authority of the Standards Policy and Strategy Committee on 31 July 2006

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ISBN 0 580 48237 5

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

This Technical Specification (CEN/TS) was approved by CEN on 2 February 2006 for provisional application.

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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.

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

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.

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:

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

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 (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:

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

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 $t_0 + 4$ 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:

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

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);

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

V_L is the volume of leachant to be added expressed in milliliters (ml).

Prepare the leachant from de-mineralized water (see 6.1.1) and acid or base (see 6.1.2 or 6.1.4) according to the acid/base consumption for the relevant pH.

Prepare the acid adjusted leachant as follows:

$$V_L = V_{demin} + V_A \quad (5)$$

$$V_A = \frac{A \times M_d}{C_A} \quad (6)$$

where

V_L is the volume of prepared leachant expressed in milliliters (ml);

V_{demin} is the volume of demineralised water used expressed in milliliters (ml);

V_A is the volume of acid needed expressed in milliliters (ml);

A is the acid consumption for the pH in mol H^+ /kg DM;

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

C_A is the concentration of the acid expressed in mol per liter (m/l) (see 6.1.2).

Prepare the base adjusted leachant as follows:

$$V_L = V_{demin} + V_B \quad (7)$$

$$V_B = \frac{B \times M_d}{C_B} \quad (8)$$

where

V_L is the volume of prepared leachant expressed in milliliters (ml);

V_{demin} is the volume of demineralised water used expressed in milliliters (ml);

V_B is the volume of base needed expressed in milliliters (ml);

B is the base consumption for the pH, mol H^+ /kg DM;

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

C_B is the concentration of the base expressed in mole per liter (mol/l) (see 6.1.4).

Split the volume V_L of leachant into 3 equal fractions $V_L/3$.

8.3.3 Procedure

Carry out the test at a temperature of $20\text{ °C} \pm 5\text{ °C}$.

Select the appropriate bottle size according to the test portion size. For $M_d = 15\text{ g}$, 30 g and 60 g , this means respectively bottle sizes of 250 ml , 500 ml and $1\ 000\text{ ml}$.

Clean the bottle before use by filling it with 1 mol/l nitric acid (see **6.1.3**), leaving it for at least 24 h and then flushing it out with de-mineralized water (see **6.1.1**).

Place one of the test portions in the rinsed bottle.

Add the leachant volume $V_L/3$ at three different times:

- 1st fraction at t_0 ;
- 2nd fraction at $t_0+30\text{ min}$;
- 3rd fraction at $t_0+2\text{ h}$.

Close the bottle and agitate the suspension (see **6.2.1**) between each leachant addition. Measure and record pH before addition of fractions 2 and 3, and prepare additional bottles with modified acid/base addition, if deviations are observed from the expected pH at that time.

Continue to agitate after the last leachant addition until $t = t_{0+48\text{ h}}$.

NOTE 1 For some waste materials gas formation may occur (e.g. CO_2 , H_2). It is recommended to pay attention when opening the bottles to release the pressure (preferably in a fume hood).

NOTE 2 For the high pH values ($\text{pH} > 9$) CO_2 uptake may affect the leaching process. This may be reduced by minimizing the contact time with the air during handling.

Measure and record the pH at $t_{0+4\text{ h}}$, $t_{0+44\text{ h}}$, $t_{0+48\text{ h}}$. For the measurement of pH stop the agitation and allow the mix to settle for 5 min . Measure pH by inserting the electrode into the leachant in the bottle.

NOTE 3 The pH value at $t_{0+4\text{ h}}$ is used for checking that sufficient pH correction has been obtained by the acid or base additions.

Since the pH is measured directly in the suspension, rinse the pH electrode thoroughly and dry before and between uses in order not to contaminate the suspension.

NOTE 4 pH is measured directly in the bottle at $t_{0+48\text{ h}}$ since filtration can change pH in the eluate.

Report the pH deviation between $t_{0+4\text{ h}}$ and $t_{0+44\text{ h}}$.

The deviation between pH at $t_{0+44\text{ h}}$ and pH at $t_{0+48\text{ h}}$ shall not exceed $0,3\text{ pH units}$, which is the limit for achieving equilibrium condition (see **3.3**). A special note shall be made in the report if this requirement is not met.

If too many experimental points deviate (more than 3 amongst 8), the equilibration time is insufficient and a longer contact time will be needed. The contact time is extended by 24 h and the pH deviation $t_{0+48\text{ h}}$ and pH at $t_{0+72\text{ h}}$ is verified.

Allow the suspended solids to settle for $15\text{ min} \pm 5\text{ min}$.

8.4 Natural pH

Repeat 8.3 for a test portion without acid or base addition, when determining the volume of de-mineralized water to be added. Measure of pH after 4 h , 44 h and 48 h as described in 8.3.

9 Performance characteristics

The performance of the test regarding repeatability and reproducibility is dependent on the tested waste material and also on the testing conditions. When the standard was adopted by CEN, the test specified in this Technical Specification was not validated and no data were available on robustness, repeatability and reproducibility.

10 Test report

In order to conform to this Technical Specification include the following information in the test report:

- a) reference to the present Technical Specification;
- b) date of receipt of the laboratory sample;
- c) sampling report according to 7.1;
- d) date of the test (beginning and end);
- e) a complete identification of the laboratory sample (including dry residue);
- f) pre-treatment (e.g. method of size-reduction, drying, sub-division) and storage conditions;
- g) temperature range during the performance of the test;
- h) selected pH interval and number of test portions tested (N) within this interval;
- i) dry mass of the test portion (g);
- j) acid volumes (ml) and concentrations (mol/l), base volumes (ml) and concentrations (mol/l) and the corresponding amounts of H_3O^+ or OH^- (mol/kg DM);
- k) leachant volume (ml) and the resulting L/S (l/kg);
- l) pH values at $t_{0+4 \text{ h}}$, $t_{0+44 \text{ h}}$ and $t_{0+48 \text{ h}}$, and difference in pH between $t_{0+4 \text{ h}}$ and $t_{0+44 \text{ h}}$ and between $t_{0+44 \text{ h}}$ and $t_{0+48 \text{ h}}$;
- m) any deviation from the test method and the reason of this deviation together with all circumstances that have influenced the results;
- n) record the data for the test results on a data sheet.

NOTE An example of the data sheet is given in Annex A.

Annex A (informative)

Example of a data sheet

Table A.1 — An example of a data sheet for the recording of test results

	Unit	Bottle no.							
		1	2	3	4	5	6	7	8
Dry mass of the test portion	g								
Acid volume	ml								
Acid concentration	mol/l								
Base volume	ml								
Base concentration	mol/l								
H ₃ O ⁺ or OH ⁻	mol/kg DM								
Leachant volume	ml								
Resulting L/S	l/kg								
pH at t_{0+4} h									
pH at t_{0+44} h									
pH at t_{0+48} h									
Δ pH (t_{0+4} h)-(t_{0+44} h)									
Δ pH (t_{0+44} h)-(t_{0+48} h)									
etc.									

Annex B (informative)

Preliminary determination of the acid/base consumption

B.1 General

In order to estimate the required amount and the concentration of acid or base needed to perform the test properly two methods are possible:

- a) a titration procedure to estimate the ANC and the BNC;
- b) an arbitrary division of the maximum acid/base consumption for the extreme pH values.

B.2 Titration procedure to estimate the magnitude of ANC and BNC

B.2.1 Reagents and laboratory devices

- a) bottles made of polypropylene (PP), PTFE or polyethylene (PE);
- b) stirring or agitation device. This is a magnetic stirring device, using a polytetrafluorethylene (PTFE) coated magnetic stirring rod, or a mechanical stirring device, made of glass or PTFE;
- c) nitric acid (pro analysis), 1 mol/l;
- d) sodium hydroxide, NaOH, 1 mol/l;
- e) distilled water, de-mineralized water or water of equivalent purity ($5 < \text{pH} < 7$) with a conductivity $< 0,5 \text{ mS/m}$;
- f) titrator (optional).

B.2.2 Test portion

Test portions are prepared in accordance with the procedure in Clause 7. Based on sample heterogeneity, it is recommended that the test portion size is either $M_d = 15 \text{ g}$, 30 g or 60 g (dry weight) (with a tolerance of $\pm 10 \%$).

B.2.3 Procedure

B.2.3.1 Preparation

Place the test portions in rinsed bottles one for acid titration and the other for alkaline titration. The test aims at a final L/S ratio of 10 after acid or base addition. If the L/S exceeds 11 because of the high acid or base consumption of the material at the specific pH value a stronger acid or base should be used for pH adjustment.

Add an amount V of de-mineralized water in the bottles establishing a liquid to solid ratio (L/S) about 9. Calculate the volume V as follows assuming the density of water to be 1 g/ml :

$$V = 9 \times M_d - (M_w - M_d) \quad (\text{B.1})$$

where

V is the amount of demineralized water added in the bottles expressed in millilitres (ml)

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

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

Record the amount V of water added.

NOTE If information is available on the material concerning a particularly strong ANC or BNC another initial L/S can be used to allow the final L/S to remain ≤ 11 .

B.2.3.2 Natural pH

Put the two filled bottles on the agitation device. Agitate or stir for 1 h. Determine the pH of the eluate directly in the bottles after settling for 10 min.

B.2.3.3 Acid titration

Add a portion of acid (B.2.1.c), manually or by use of the titrator, into one of the bottles from B.2.3.1 and determine the pH directly in the bottle after 30 min agitation or stirring. Record the amount and concentration of acid added, and the pH obtained.

NOTE 1 The amount of acid needed to get a decrease in pH can vary between materials and the initial portions need therefore be small in order to see the magnitude of the first response by the material.

NOTE 2 In case of high acid demand manual addition of strong acid in the beginning of the determination is practical and a shorter response time than 30 min may be used.

Continue to add portions of acid and to measure pH after 30 min stirring or agitation after each portion added. Repeat until the entire pH interval from the natural pH (B.2.3.2) to pH 4 or below is obtained and the distance between the obtained pH values is smaller than 1,5 pH values. Record the amounts and concentrations of acid added, and the pH values obtained.

B.2.3.4 Base addition

Add a portion of base (B.2.1.d) into the other of the bottles from B.2.3.2, and determine the pH directly in the bottle after 30 min agitation or stirring. Record the added amount and concentration of base, and the obtained pH.

NOTE 1 The amount of base needed to get an increase in pH vary between materials and the initial portions need therefore be small in order to see the magnitude of the first response by the material.

NOTE 2 In case of high base demand manual addition of strong base in the beginning of the determination is practical and a shorter response time than 30 min may be used.

Continue to add portions of base and to measure pH after 30 min stirring or agitation after each portion added. Repeat until the entire pH interval from the initial pH (B.2.3.1) to pH 12 or above is obtained and the distance between the obtained pH values is smaller than 1,5 pH value.

NOTE 3 In order not to under estimate the ANC or the BNC it is recommended to wait 24 h for pH 4 in case of very alkaline materials or pH 12 for materials with a high buffer capacity.

B.2.4 Expression of results

Make a curve of pH versus amounts of acid and base expressed in mol H^+ /kg and mol OH^- /kg.

B.3 Arbitrary division of the maximum acid/base consumption for the extreme pH values

B.3.1 General

For waste materials with a very strong acid-base capacity, manual titration may lead to excessive experimental duration when the maximum acid and base consumption in order to reach respectively pH 4 and pH 12 is unknown. In this procedure the natural pH and acid and base consumption at pH 4 and pH 12 respectively is estimated.

B.3.2 Reagents and laboratory devices

- a) bottles made of polypropylene (PP), PTFE or polyethylene (PE);
- b) stirring or agitation device. This is a magnetic stirring device, using a polytetrafluorethylene (PTFE) coated magnetic stirring rod, or a mechanical stirring device, made of glass or PTFE;
- c) titrator;
- d) nitric acid (pro analysis), 1 mol/l;
- e) sodium hydroxide, NaOH, 1 mol/l;
- f) distilled water, de-mineralised water or water of equivalent purity ($5 < \text{pH} < 7$) with a conductivity $< 0,5 \text{ mS/m}$.

B.3.3 Test portion

Test portions are prepared in accordance with the procedure in Clause 7. Based on sample heterogeneity, it is recommended that the test portion size is either $M_d = 15 \text{ g}$, 30 g or 60 g (dry weight) (with a tolerance of $\pm 10 \%$).

B.3.4 Procedure**B.3.4.1 Preparation**

Place two of the test portions in rinsed bottles one for acid titration and the other for alkaline titration. The test aims at a final L/S ratio of 10 after acid or base addition. If the L/S exceeds 11 because of the high acid or base consumption of the material at the specific pH value a stronger acid or base should be used for pH adjustment.

Add an amount V of de-mineralized water in the bottles establishing a liquid to solid ratio (L/S) about 9. Calculate the volume V as follows assuming the density of water to be 1 g/ml :

$$V = 9 \times M_d - (M_w - M_d) \quad (\text{B.2})$$

where

V is the amount of demineralized water added in the bottles expressed in millilitres (ml);

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

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

Record the amount V of water added.

NOTE If information is available on the material concerning a particularly strong ANC or BNC another initial LS can be used to allow the final LS to remain ≤ 11 .

B.3.4.2 Natural pH

Put the two filled bottles on the agitation device. Agitate or stir for 1 h. Determine the pH of the eluate directly in the bottles after settling for 10 min.

B.3.4.3 Acid titration

Titrate one of the bottles in B.3.4.1 with acid (B.3.2.d) in the titration equipment set at pH 4. Confirm the acid consumption over a titration period of 24 h. Record the added amount and concentration of acid, and the obtained pH.

NOTE 1 If the time taken to complete this last point needs to be reduced, samples may be size reduced to below $0,5 \text{ mm}$ (as no analysis is foreseen).

NOTE 2 If this equipment is not available, manual titration may be carried out with the objective of achieving pH 4 as soon as possible including an overnight waiting period to validate the last measurement points.

NOTE 3 If the time taken to complete this last point needs to be reduced, samples may be size reduced to below 0,5 mm (as no analysis is foreseen).

B.3.4.4 Base addition

Titrate the other of the bottles in B.3.4.1 with base (B.3.2.e) in the titration equipment set at pH 12. Confirm the base consumption over a titration period of 24 h. Record the added amount and concentration of base, and the obtained pH.

NOTE 1 If this equipment is not available, manual titration may be carried out with the objective of achieving pH 12 as soon as possible including an overnight waiting period to validate the last measurement points.

NOTE 2 If the time taken to complete this last point needs to be reduced, samples may be size reduced to below 0,5 mm (as no analysis is foreseen).

B.3.5 Expression of results

The acid consumption to reach pH 4 and the base consumption to reach pH 12 is recorded together with the natural pH.

Divide the amount of acid by double the number of pH values intended to be tested within the acid pH range (pH range as a result of acid addition).

Divide the amount of base by double the number of pH values intended to be tested within the alkaline pH range (pH range as a result of base addition).

NOTE 1 If e.g. between the natural pH and pH 4,5 pH values are sought and if the maximum acid consumption is 5 mol H⁺/kg, it is recommended to prepare 10 different acid solutions from 50 mmolH⁺/l to 500 mmolH⁺/l at evenly-spaced intervals.

NOTE 2 This method allows limitation of the misevaluation of the ANC and BNC and the selection of the 7 solutions to be analyzed after reaching the stationary pH. This allows also choosing solutions leading to the same pH, i.e. along a potential pH plateau corresponding to the buffer capacity of the material (e.g. carbonates) of high interest in terms of behavior.

NOTE 3 Due to buffering of the matrix equally spaced portions generally will not lead to proper final pH values. Doubling the number of bottles is no guarantee for obtaining properly spaced final pH values. Through interpolation, it will be possible to estimate the proper amounts needed from the curve of final pH against acid/base consumption.

Annex C (informative)

Examples of acid and base neutralisation capacities for waste, soil, sediment and construction materials and use of the data

C.1 Examples of acid and base neutralisation capacities

From previous work [1,2,3,4] data on acid - and base neutralisation capacity data have been obtained for a wide range of materials. This implies that this previous knowledge can be used in 8.3.2 of the pH dependence test. The information provided may not be entirely accurate for a sample under consideration. However, the variation within one material class is generally not very large.

In Figure C.1 acid neutralisation and base neutralisation capacities for waste, soil and construction materials are given, which can be used as a starting point to carry out a pH dependence test. This information allows the selection of the relevant acid or base strength as well as the approximate amount of acid or base needed for a given sample weight to reach a certain desired end pH. This information can come in the place of the pre-titration as described in Annex B.

The selection of the acid strength is based on the buffer capacity of the material. In case of low buffer capacity in materials such as soil, metallurgical slag, sintered brick and sintered aggregates, the lowest acid/base strength specified in the standard should be used.

For the pH dependence test using continuous pH control, which is aimed at reaching a fixed and pre-determined pH, the acid strength is the most crucial parameter. For the pH dependence test with fixed initial acid/base addition, the proper amounts of acid/ base to be added needs to be determined. Generally more bottles are prepared in this latter test mode, from which the proper end pH values are selected for further analysis.

To calculate the amount of acid or base needed from the ANC/BNC expressed in mol/kg the following formula applies:

$$A_{\text{pHX}} = 1000 \times \text{ANC}_{\text{pHX}} \times \frac{M}{N_{\text{pHX}}} \quad (\text{C.1})$$

where

A_{pHX} is the amount of acid needed expressed in ml to reach pH = X for sample of weight M ;

ANC_{pHX} is the acid neutralization capacity expressed in mol per kilogram from Figure C.1 at pH = X;

N_{pHX} is the acid normality selected based on the buffer capacity around pH=X expressed in mol per litre (mol/l);

M is the sample weight expressed in kilograms (kg).

C.2 Use of acid and base neutralisation capacity data

These ANC/BNC characterisation test data can be used to assess the amount of acid (as H^+) or base (as OH^-) needed to reach a given final pH in a waste. This property can be used also to determine the final pH of mixed waste in a landfill by summing the buffer capacities (provided that there is no chemical interaction between the different wastes). The data can also be used to assess the effect of external influences, such as carbonation and oxidation (acid production resulting from sulphide oxidation) on the pH of a waste (as a first approximation, only considering the H^+ providing, without taking into account the specific effect of carbonation or sulphate in solution on the solubility of the different elements.). It can be used also to assess the development of a pH front with potential consequences for changes in leaching behaviour or durability of cement-based materials (carbonation may lead to loss of structural integrity of cement stabilised waste). The external influences need to be calculated in

the same units to allow comparison, so carbonation would have to be assessed through volume of gas, concentration of CO₂, conversion to corresponding moles of H⁺ neutralising capacity.

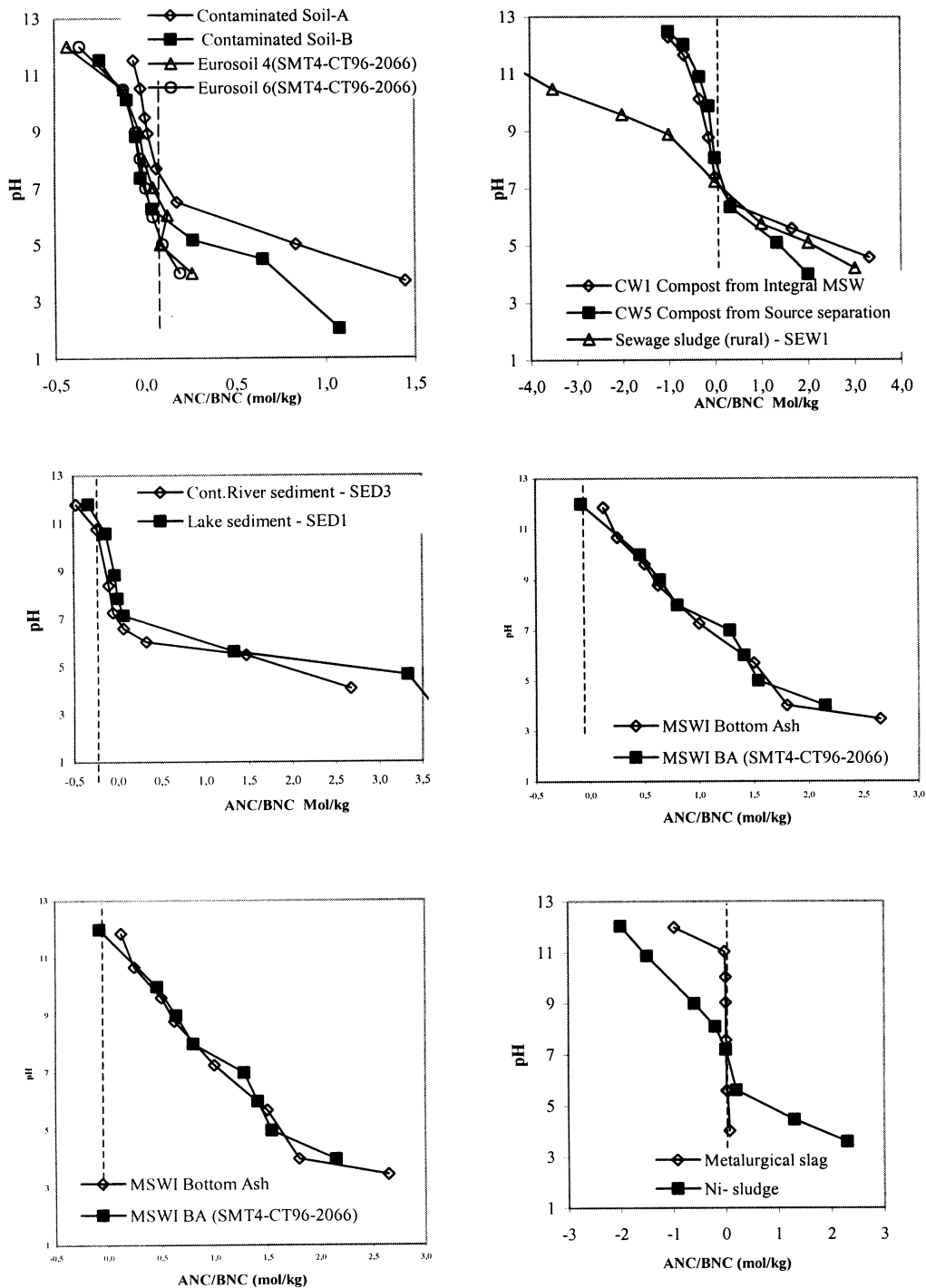


Figure C.1 — Acid/base neutralization curves for a wide range of materials

NOTE The base addition is given as negative values – [6, 8, 9].

Annex D (informative)

Examples of steps in the full evaluation of ANC and BNC for waste materials

The flow sheet of Waste characterization (Figure D.1) contains several steps. This CEN/TS relates to step 5 in the scheme 'Extraction'. Furthermore test portion preparation and measurement report are two of the steps relating to the method standardized in this Technical Specification.

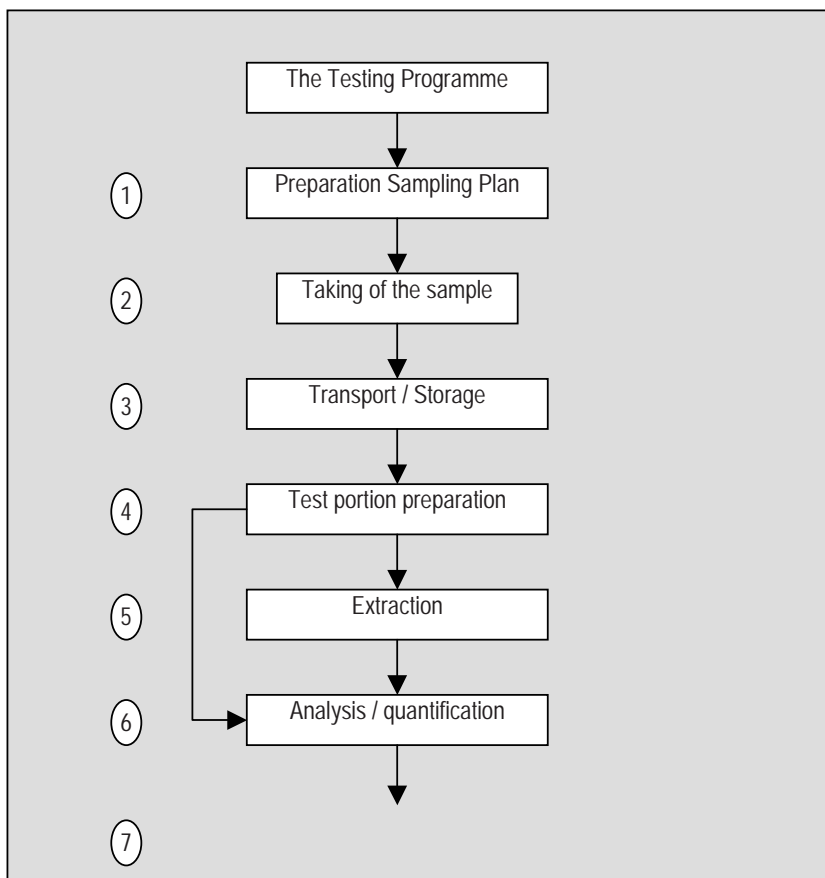


Figure D.1 - Flowsheet of Waste

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