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Leaching behaviour tests – Influence of pH on leaching with initial acid/base addition – Horizontal standard

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Foreword

This document (prEN xxxxxx) has been prepared by project HORIZONTAL, the management of standards is held by BT/TF 151.

This document is currently submitted for consultation to CEN and ISO bodies.

This European Standard has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries. This European Standard was prepared by project HORIZONTAL with support of BT/TF151.

Explanation about development of horizontal standards

This European Standard received approval from the CEN Technical Board on(date).

This European Standard was elaborated on the basis of:

PrEN 14429

This European Standard specifies a pH dependence leaching test to determine the leaching behaviour of granular materials under imposed pH conditions.

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

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Introduction

This European standard has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries. Tests to characterize the behaviour of 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 materials. Liquid/solid (L/S) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity, ageing of material and physical parameters are addressed in these tests.
2. "Compliance" tests are used to determine whether the material 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 material 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 standard belongs to category (1): basic characterization tests.

In the test, which is described in this European standard 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 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 WI 292033) 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 A 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 European standard is applicable to determine the influence of pH on the leachability of inorganic constituents from a 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 standard 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 material 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

This European standard 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 only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applied.

ENV 12506: 2000, *Characterization of waste - Chemical analysis of eluates - Determination of pH, As, Cd, Cr^{vi}, Cu, Ni, Pb, Zn, Cl, NO₂⁻, SO₄²⁻*

ENV 12920:1996, *Characterization of waste - Methodology for the determination of the leaching behaviour of waste under specific conditions*

ENV 13370:2000, *Characterization of waste – Analysis of eluates – Determination of Ammonium-N, AOX, conductivity, Hg, phenol index, TOC, CN_{easily liberated}, F⁻*

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

WI 292001, *Characterization of waste – Sampling of liquid and granular materials including paste-like materials and sludges- Part 1: A framework for sampling plan preparation*

ISO-EN 5667-3:1995, *Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples.*

3 Terms and definitions

For the purposes of this European Standard, 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

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

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	the 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 prepared leachant
w_{dr}	dry residue of the material

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 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 H^+ /kg DM (-mol H^+ /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 material 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 – 5 mol/l

6.1.3

Nitric acid (pro analysis), 1 mol/l

6.1.4

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

6.2 Laboratory devices

6.2.1

Agitation device: end-over-end tumbler (5 - 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

Membrane filters for the filtration device, fabricated from inert material, which is compatible with the material and has a pore size of 0,45 µm. Filter must 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 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

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 WI 292001 or a standard derived from WI 292001 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 material (%);

m_d is the mass after drying (g);

m_r is the mass before drying (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 (g);

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

w_{dr} is the dry residue of the material (%);

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

Measure pH in the liquid after each of these periods.

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

8.2 pH-range

The test shall cover the range pH 4-12 (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 of which only the ones with the desired final pH values are retained for analysis.

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 may 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 Leaching test

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

8.3.1 Preparation of leachant

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

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

Calculate the volume V of liquid to establish $L/S = 10 \pm 0.2$ (l/kg) for the actual size of test portion M_d (see 7.4) including the volume of acid or base.

$$V = 10 \cdot M_d$$

where

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

M_d is the dry mass of the test portion (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:

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

where

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

M_d is the dry mass of the test portion (g) (see **7.4**);

w_{dr} is the dry residue of the material [%] (see **7.3**);

V_L is the volume of prepared leachant (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:

$$V_L = V_{de\ min} + V_A$$

$$V_A = \frac{A \cdot M_d}{C_A} \quad (3)$$

where

V_L is the volume of prepared leachant (ml).

$V_{de\ min}$ is the volume of demineralised water used (ml);

V_A is the volume of acid needed (ml);

A is the acid consumption for the pH (mol H⁺/kg DM)

M_d is the dry mass of the test portion (g) (see **7.4**);

C_A is the concentration of the acid (mol/l) (see **6.1.2**);

Prepare the base adjusted leachant as follows:

$$V_L = V_{de\ min} + V_B$$

$$V_B = \frac{B \cdot M_d}{C_B} \quad (3)$$

where

- V_L is the volume of prepared leachant (ml).
- V_{demin} is the volume of demineralised water used (ml);
- V_B is the volume of base needed (ml);
- B is the base consumption for the pH (mol OH⁻/kg DM)
- M_d is the dry mass of the test portion (g) (see 7.4);
- C_B is the concentration of the base (mol/l) (see 6.1.4);

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

8.3.2 Leaching procedure

Carry out the test at a temperature of 20 °C ± 5 °C.

Select the appropriate bottle size according to the test portion size. For $M_d = 15$ g, 30 g and 60 g, this means respectively bottle sizes of 250 ml, 500 ml and 1000 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 min;
- 3rd fraction at t_0+2 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 $t = t_0+48$ h.

NOTE 1 For some materials gas formation may occur (e.g. CO₂, H₂). Pay attention when opening the bottles to release the pressure (preferably in a fume hood).

NOTE 2 For the high pH values (pH>9) CO₂ 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 h, t_0+44 h, t_0+48 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 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.

The pH value measured before filtration at t_0+48 h will be the one associated to the analysis of the eluate.

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NOTE 4 pH is measured directly in the bottle at t_0+48 h since filtration may change pH in the eluate.

Report the pH deviation between t_0+4 h and t_0+44 h.

The deviation between pH at t_0+44 h and pH at t_0+48 h shall not exceed 0,3 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 conclusion is that this test is not applicable to this material.

NOTE 6 When equilibrium condition is strictly compulsory for specific use but not fulfilled in the test, it is possible to continue the test to maximum 7 days for all the 8 experimental points (selected pH values) in order to avoid association of results at 48 hours with those at longer leaching time. In this case, pH deviation could be limited to 0,3 pH units for the last 24h (maximum between 6th and 7th days). The experimental point(s), which do not conform to these new conditions, will not be exploited and this fact shall be mentioned in the report. This specific procedure is not part of this standard.

Allow the suspended solids to settle for 15 min \pm 5 min.

Rinse the part of filtration device (see 6.2.9), which is in contact with the eluate with nitric acid (see 6.1.3), and flush with ultra pure water before any use.

Filter the suspension through a 0,45 μ m membrane filter (see 6.2.5) using the filtration device (see 6.2.9).

Proceed immediately with the eluate treatment as specified in clause 9.

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 in 8.3.

NOTE A very low conductivity in the eluate at natural pH may increase generation of colloids, which may clog filters and increase analyzed concentrations. This can be avoided by addition of NaNO₃ in the leachant at a concentration comparable to the concentration of acid or base in the closest pH values tested. The use of NaNO₃ (amount and concentration) shall be reported.

9 Eluate treatment, storage and analysis

Divide the eluate into an appropriate number of sub-samples for different chemical analysis. Preserve the eluate sub-samples depending on the elements to be analysed and store them, in accordance with the requirements in EN-ISO 5667-3.

Determine the concentrations of constituents of interest using the methods of eluate analysis (e.g. prENV 12506 and prENV 13370).

10 Blank test

The contribution of the leachant, devices and membrane filters to the result shall be determined by running a blank test periodically in order to check how the whole procedure is performed.

Obtain three different blank tests by carrying out the procedure according to 8.3 and 9 without the solid material with the addition of:

1. both the maximum amounts of acid and base to the leachant of similar volume as used in the tests ;
2. acid to obtain pH 4;
3. base to obtain pH 12.

The eluates of the blank tests shall fulfil the minimum requirement that the concentration of each considered element shall be less than 10 % of the concentration in the eluate. If the concentration of an element is below the detection limit for the analytical method applied, the blank test requirements will be fulfilled if the blank test is below the same limit of detection. If this requirement is not fulfilled it is necessary to reduce contamination.

The most recent blank test results shall be available. .

Do not subtract the results of the blank test determination from the test results.

NOTE 1 The test is aiming at equilibrium condition in which the contribution from the equipment is included. Subtraction of the blank test results will therefore lead to an incorrect equilibrium concentration.

11 Performance characteristics

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

12 Test report

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

- Reference to the present standard;
- Date of receipt of the laboratory sample and test report according to 7.1
- Date of the test (beginning and end);
- A complete identification of the laboratory sample (including dry residue);
- Pre-treatment (e.g. method of size-reduction, drying, sub-division) and storage conditions;
- Temperature range during the performance of the test;
- Selected pH interval and number of test portions tested (N) within this interval;
- Dry mass of the test portion (g);
- 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);
- Leachant volume (ml) and the resulting L/S (l/kg)
- 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 $t_0+48\text{h}$;
- The method of preservation for the different elements in accordance with 9.
- The analytical report in accordance with 9.;
- The date of the latest blank test. Blank results shall be available upon request.
- Any deviation from the test method and the reason of this deviation together with all circumstances that have influenced the results;

Record the data for the test results on a data sheet,

Note: an example of of the data sheet is given in Annex A

Annex A (informative)

Operation and uses of the test: influence of pH on the leaching behaviour

A.1 Clarification of the two modes of operation of the test : influence of pH on the leaching behaviour

Two standards are aiming at determining the influence of pH on the release of the inorganic constituents from a material.

In the test described in this document (Leaching behaviour test – influence of pH on leaching with initial acid/base addition), an 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 material. Size reduction is performed to accelerate reaching of equilibrium condition.

This test is different from the test « Leaching behaviour test – influence of pH on leaching with continuous pH control », in which 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.

Figure A.1 shows a typical pH variation during the test for an alkaline material at a final pH of 5 for each of the two modes. They differ in the manner how the final pH is reached. This is one of the differences between the two tests. Another one is, for example the total amount of acid/base added during the test.

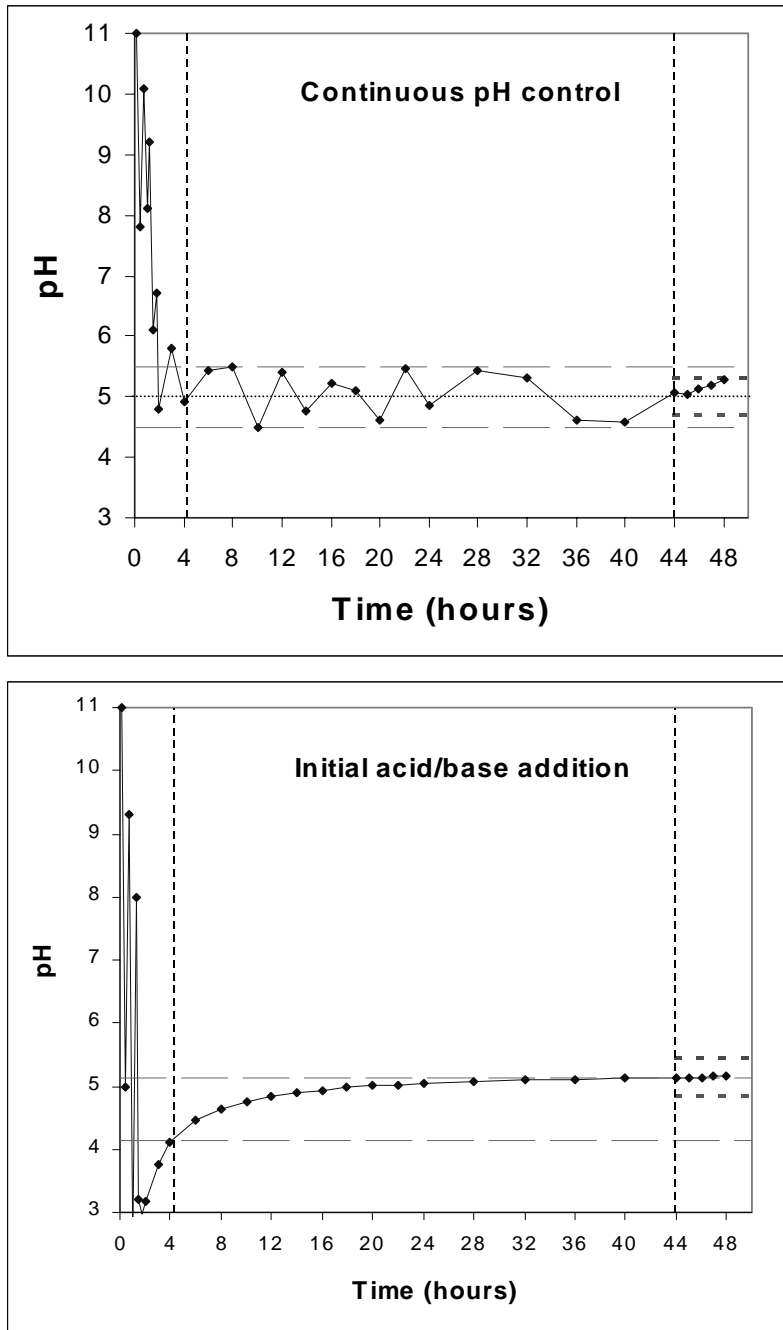


Figure A.1 — Typical pH variations during the two modes of the tests for determining the influence of pH on the leaching behaviour of an alkaline material at a final pH around 5

A.2 Expression of results

Three graphical presentations of the results can be obtained. They provide a visual representation of the test results as a trend (see A.3):

1. pH at t0+48 hours (see 8.3.2) versus the amounts of acid/base added (ANC and BNC curve) expressed in mol H^+/OH^- added per kg of dry matter;
2. concentration of each analysed constituent in eluates in log scale in mg/l versus pH at t0+48 hours;
3. concentration of each analysed constituent in eluates in linear scale in mg/l versus the amounts of acid or base added (mmol of H^+/OH^- /kg dry matter).

NOTE In the third graph pH at t0+48 hours can also be added as a second ordinate.

In case when the leached amounts (U_x) (mg/kg dry material) are needed, they can be directly calculated by multiplying the concentrations in mg/l by the L/S value (normally L/S =10 ml/g dry material).

A.3 Scope and limits of the application field of the test

This test provides information on the influence of pH on leaching under the experimental conditions specified in this standard. It does not directly take the effects of other parameters such as influence of other acid and base than the nitric acid/sodium hydroxide used in the test, dissolved organic carbon, complexation, redox conditions into account (see A.4.4).

This test method is a parameter specific test as specified in the ENV 12920 "Characterization of waste - Methodology for the determination of the leaching behaviour of a material under specified conditions". The application of this test method alone is not sufficient for the determination of the detailed leaching behaviour of a material under specific conditions.

NOTE This generally requires the application of several test methods, behavioural modelling and model validation as specified in the ENV 12920.

Therefore, provided that the nitric acid/sodium hydroxide used in the test as well as the other experimental conditions are relevant for the considered scenario, this test is useful to:

- qualify and quantify the material resistance to acid-basic attack through the concentration measured, as a function of pH and the acid/base amount needed to reach a given final pH;
- identify the chemical behaviour trends and the availability levels of components at different pH values under the experimental conditions specified in this test. These values can be used as input to modelling of chemical behaviour using geochemical speciation models (e.g. MINTEQA2). In many cases, it also provides insight in the relevance of particular solubility controls and release mechanisms (e.g. formulate hypothesis on the dissolution mechanisms);
- provide data to feed dynamic behavioural models, for instance under the following relationship: solubilisation = f (pH or meq H⁺/g) in the physico-chemical context linked with the presence of the other compounds in the material. This is not always possible with available literature data.

The test results can usually not be linked directly to concentrations observed under field conditions.

A.4 Examples of uses of the test: influence of pH on the leaching behaviour

Below a number of possible uses of this test are provided.

A.4.1 Identification of the sensitivity of leaching to pH over the environmentally relevant pH range

The test provides insight in the sensitivity of leaching of components from a specific material to pH (see **Figure A.2**). This factor has been found to be a major release controlling parameter in virtually all materials. Obviously, the relevant pH range for a given application may be limited. However, for characterisation purposes the full pH range from at least 4 to 12 is important as different uses of the information relate to different pH domains. In Figure A.2 the leaching behaviour under the influence of pH is illustrated for Cd from heavily sewage sludge amended soil [6]. An indication of the repeatability of the method can be obtained from the duplicate test data. The test was performed with initial acid/base addition. This Figure A.2 indicates also pH ranges typical of some material – conditions of scenarios combinations.

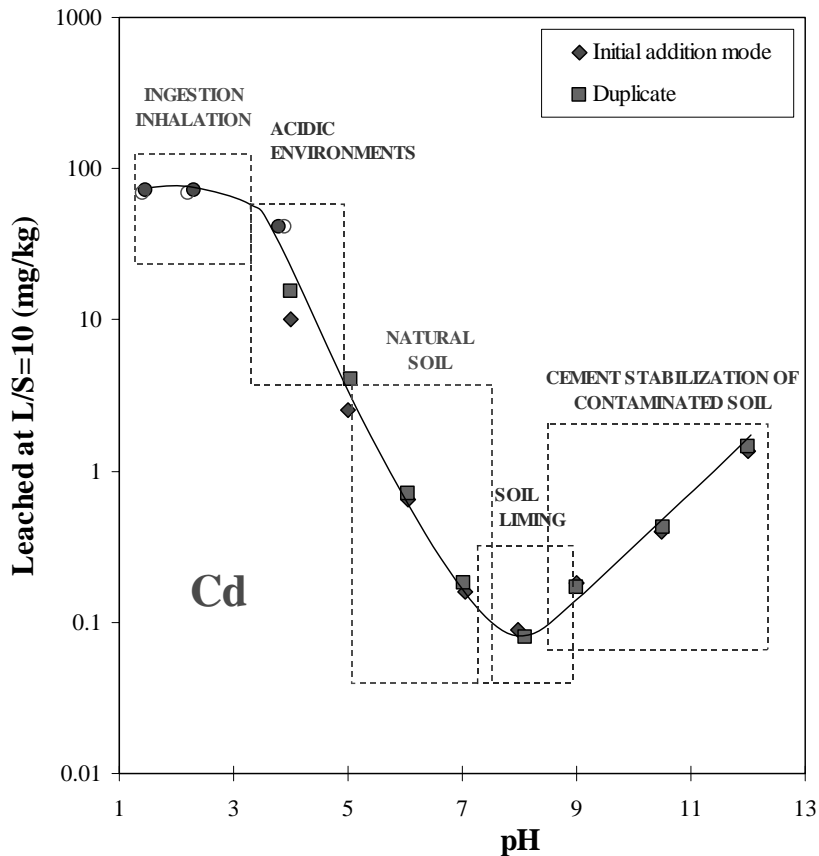


Figure A.2 — Illustration of the influence of pH on the leaching behaviour of a heavily sewage sludge amended soil as obtained in a pH range 4-12 (test performed with initial acid/base addition) its use in relation to different scenarios for the same material

A.4.2 Basis for chemical speciation and modelling

A crucial step for long term behaviour prediction is to identify to which extent the exposure conditions in terms of pH are dominated by the material to be evaluated or by the surrounding imposes its pH on the material. External impacts on the pH-conditions are e.g. acid rain, atmospheric and biodegradation derived CO₂, sulfide oxidation, and leachates from other materials. In these cases, to a varying degree other complexing agents than H⁺/OH⁻ may also have an impact on the leaching process for certain elements. With the test it is possible to identify the impact of changes in pH when the effect of external complexing agents are limited. Other influences can be identified with the test. However, other information is needed to quantify such effects.

The time frame of change can often be estimated using the acid neutralisation capacity or base neutralisation capacity and a rate for the acid/ base producing source.

Analytical data from the test on the influence of pH on the leaching behaviour may be used as input data for geochemical speciation codes (such as MINTQA2, GEOCHEM WORKBENCH, ECOSAT, CHESSE, SPEC, ORCHESTRA,...) to identify the solubility control by specific mineral phases (see **Figure A.3**). However, the results should be handled with some caution since the model databases contain solubility constants for well-studied minerals and may not include solubility constants for amorphous phases, less crystallised solids, solid solutions etc. Other processes such as sorption processes and interaction with dissolved organic matter may be a part in the pH-dependent behaviour can also to some extent with additional data be evaluated from the geochemical modelling (see **Figure A.4**).

The results of the test on the influence of pH may be an important source of input data for dynamic behavioural models, (e.g. solubilisation = f (pH or meq H⁺/g) in the physico-chemical context). In comparison to literature data the test not only gives information on the pH behaviour of single substances in the material but also gives information on the interactions between substances as a function of pH.

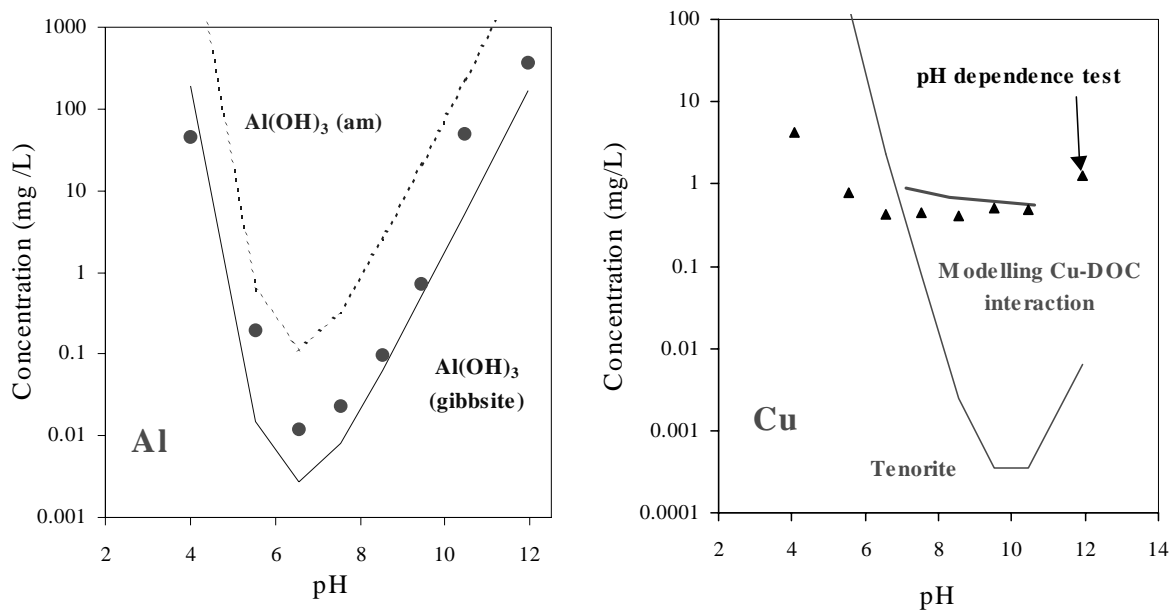


Figure A.3 — Example of modelling of the chemical speciation of Al and Cu in eluates from Municipal Solid Waste Incinerator bottom ash. The test data on the influence of pH have been used as input for the MINTEQA2 showing for Al a good match with gibbsite and for Cu a poor match with tenorite. In the latter case, the incorporation of interaction with organic ligands resulted in a much better fit of the measured data points [10]

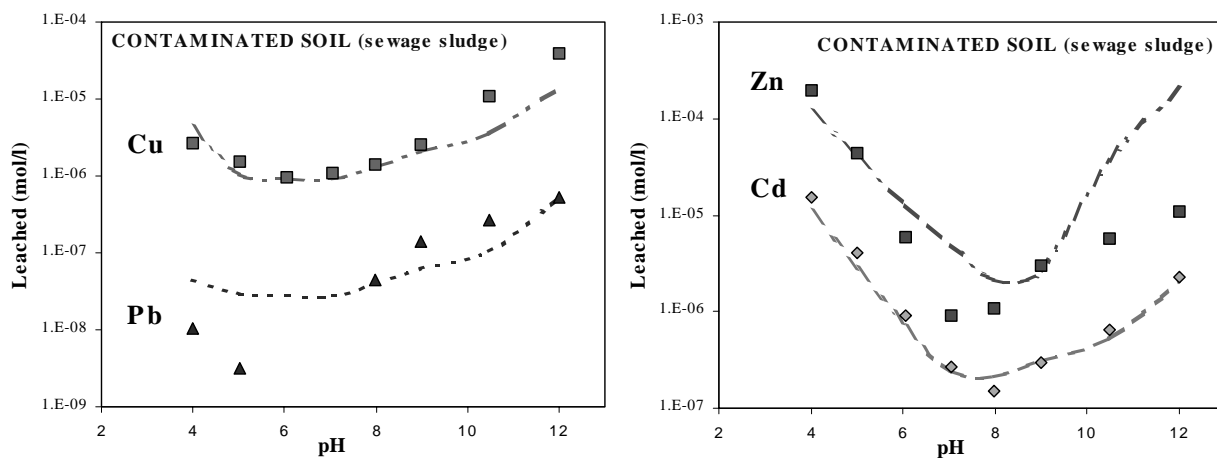


Figure A.4 — Example of modelling of the chemical speciation of Cd, Cu, Pb and Zn in eluates from contaminated soil. The test data on the influence of pH have been used as input for the NICCA-Donnan model in ECOSAT showing for Copper and Cadmium a good match between prediction and measurement. The main factor controlling metal mobility in this case is the interaction of metals with particulate and dissolved organic matter (DOC) [6].

A.4.3 Identification of leaching controlling factors

By accumulating test data and knowledge about the influence of pH on leaching of a certain kind of material, one could identify general trends for some constituents (e.g. Pb, Cd). The comparison between such trends for most samples from a certain material type and available data on individual samples of that material may show a deviation from the observed general trends indicative of specific characteristics of that sample, based on experimental testing conditions (nitric acid/sodium hydroxide addition used in the test). In case of different incinerator residues, the Cd leachability is largely controlled by Cl complexation and by different maximum Cd leachability as reflected by the plateau's in leaching at low pH (see **Figure A.5**).

In the example shown in Figure A.2 one might normally expect for Cd a leaching behaviour, in which the leachability is low at high pH (pH > 9; see for instance Cd leaching behaviour from MSWI bottom ash in

Figure A.5). However, the presence of DOC in the heavily sewage sludge amended soil results in strong Cd-DOC complexation. This leads to increased leachability of Cd with increasing pH at pH > 9. DOC leachability increases with increasing pH, which is directly visible in the subsequent extracts by an increased coloration of the solution from yellow or light brown to darker brown (humic substances).

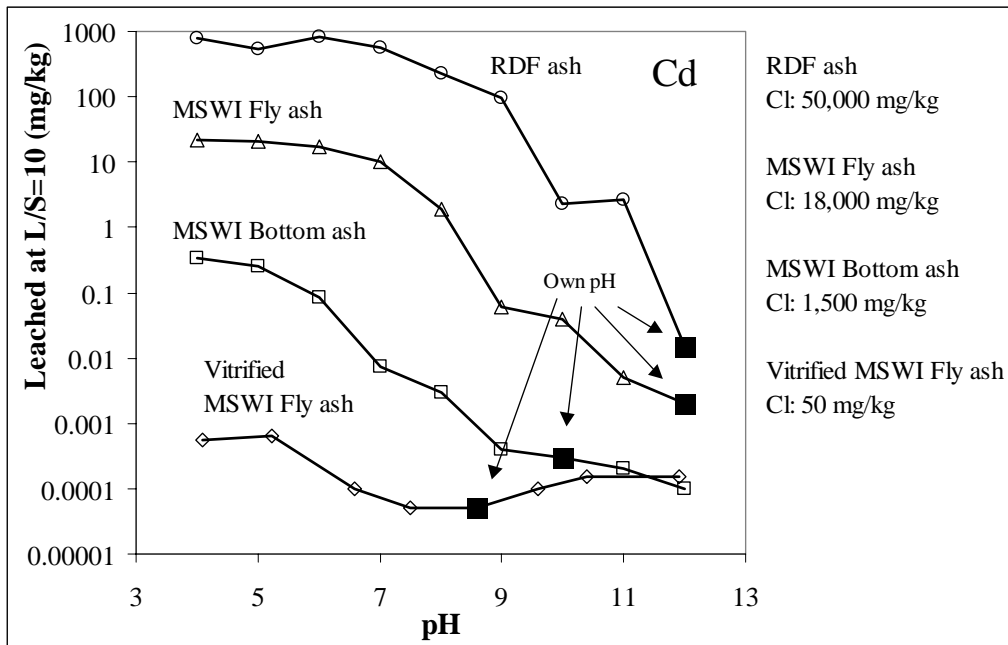


Figure A.5 — Leachability of Cd from incinerator residues illustrating the role of Cl in complexing Cd and thus changing the Cd leaching behaviour as a function of pH. (RDF = Refuse Derived Fuel; MSWI = Municipal Solid Waste Incineration)

A.4.4 Identification of the sensitivity to pH variations affecting the repeatability of leaching tests

In many leaching tests, the material itself dictates the pH conditions in the extract and consequently the leaching (e.g EN 12457). In the pH domain dictated by the materials itself, leaching may be very sensitive to minor changes in pH, which may lead to factors or sometimes order of magnitude differences in the leached quantity of specific components. These are aspects inherent to the leaching behaviour of materials and the leaching process related to changes of physico-chemical equilibria. It is crucial to be aware of such sensitivities for a given material. The pH domain, in which such sensitivities occur, may vary by element and by material. In Figure A.2 it is clear that the largest change of Cd leachability resulting from a relatively small change in pH occurs where the leachability – pH curve is at its steepest (Cd: sensitive pH range 4 – 7).

A.4.5 Basis for the comparison of different (international) leaching tests

Many leaching tests have been developed world-wide. Their comparability is complicated due to different test conditions and to various objectives (e.g. up-flow percolation test - determine the leaching behaviour of inorganic constituents from granular material - and pH influence test: determine the influence pH on the leachability of inorganic constituents from a material).

Such comparisons have to be done preferably in the framework of the ENV 12920 standard.

However, when a material's leaching behaviour is controlled by solubility, concentrations as a function of pH should theoretically become more and more comparable. For instance, several studies [4,6,8], in which different leaching tests have been compared, show that the pH influence test forms a good basis for mutual comparison of leaching tests. As in most leaching tests developed for basic characterisation and compliance testing pH is one of the most relevant leachability-controlling factor (see **Figure A.2**). In Figure A.6 a comparison of Cd leachability from heavily sewage sludge amended soil is given where several different leaching test results are compared with the pH dependence leaching test data for the same material.

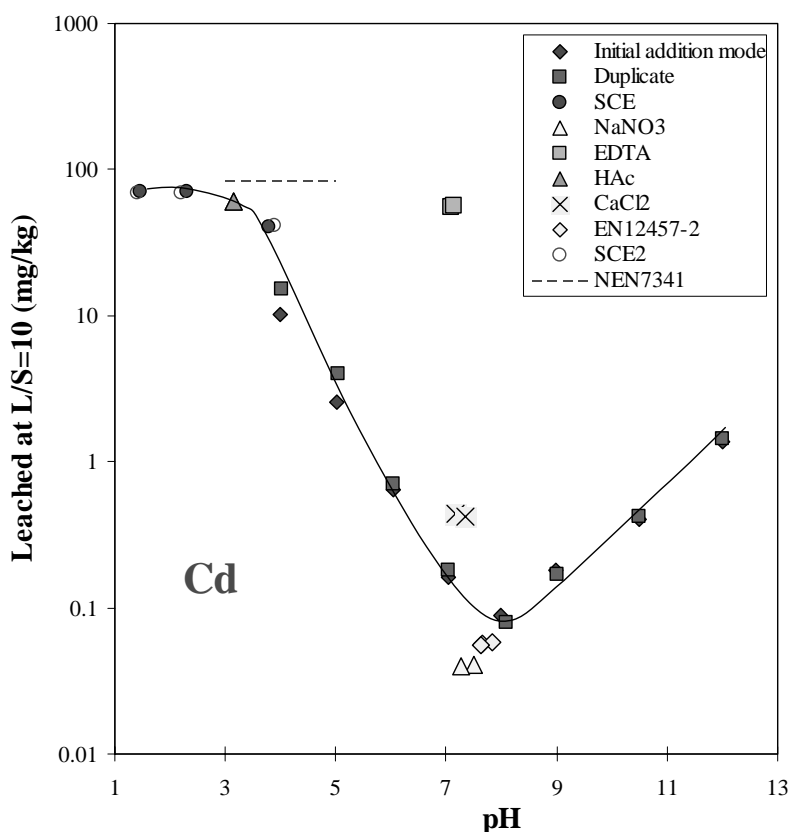


Figure A.6 — Illustration of the influence of pH on the leaching behaviour of a heavily sewage sludge amended soil as obtained in a pH range 4-12 (test performed with initial acid/base addition). Others leaching procedures results are added to illustrate the importance of pH in the process of release

LEGEND pH influence test with initial acid/base addition with a duplicate test; HAc - Acetic acid extraction test; EDTA - EDTA extraction test; CaCl₂ and NaNO₃ - mild water extraction methods; SCE - sequential chemical extraction data (fractions presented in cumulative fashion); NEN 7341 – Availability test data.

Extractions with different mild water leachants, acidic extracts and a sequential chemical extraction are compared. The figure shows that, apart from EDTA, different tests with different experimental conditions feature leaching in a very similar magnitude when compared at the appropriate test pH (Extraction with EDTA does not fit the pH characteristic of Cd for this material due to the Cd complexation behaviour of EDTA, which is almost independent of pH).

A.4.6 Mutual comparison of separate productions or charges form the same material class

As a corollary of the uses describes in the paragraphs above the pH influence test results can be useful to identify differences in leaching behaviour within one material class. Obviously, in such cases analysis of the full range is no longer needed. The pH condition to be verified can be selected according to the most relevant condition for the material in a scenario under consideration. In terms of leaching behaviour as a function of pH, materials from regular production processes (primary or secondary material) will tend to show the same leaching character, although between charges variations in release level at a given pH may occur. The factor controlling such variation may be traced back to operational parameters and can often be used for control measures in production.

A.4.7 Mutual comparison of different materials

Using pH dependence test information as the basis (final pH obtained by nitric acid/sodium hydroxid addition), the leaching character of different materials can be compared. From such a comparison, far more can be deduced with respect to factors potentially controlling the leaching behaviour of materials than results from single extraction tests or time dependent leaching tests ever can. Factors identified as relevant controlling factors for one material can be identified as being relevant to other materials as well. A perceived inorganic material may show typical behaviour of

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materials containing organic matter (not to be confused with organic micropollutants), which would trigger analysis of DOC and lead to conclusions on the possible role of organic complexants.

By obtaining information on a wide range of materials with widely varying properties, effects of specific factors can be recognized. Consequently, in the case of unknown materials, the pH dependence test may provide conclusions on possible reasons for deviating or unexpected behaviour on the basis of the database results. However such comparison is relevant only when the nitric acid/sodium hydroxide used in the test as well as the other experimental conditions are relevant for the considered scenario

Annex B (informative)

Preliminary determination of the acid/base consumption

In order to determine the amount and concentration of acid/base two methods are possible:

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

B.1 Titration procedure to estimate the ANC and the BNC

B.1.1 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), 0.1 – 14.4 mol/l;
- d) Sodium hydroxide, NaOH, 0.1 – 5 mol/l;
- e) Distilled water, de-mineralized water or water of equivalent purity ($5 < \text{pH} < 7$) with a conductivity < 0.5 mS/m;
- a) Titrator (optional).

B.1.2 Test portion

Test portions are prepared in accordance with the procedure in section 7. Based on sample heterogeneity, test portion size shall be either $M_d = 15$ g, 30 g or 60 g (dry weight) (with a tolerance of ± 10 %).

B.1.3 Procedure

B.1.3.1 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:

$$V = 9 \cdot M_d - (M_w - M_d) \quad (\text{ml}) \quad (\text{A.1})$$

where

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

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

Record the amount V of water added.

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

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B.1.3.2 Natural pH

Put the two filled bottles on the agitation device. Agitate/stir for 1 hour. Determine the pH of the eluate directly in the bottles after settling for 10 minutes.

B.1.3.3 Acid titration

Add a portion of acid (B.1.1.c), manually or by use of the titrator, into one of the bottles from B.1.3.1 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 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 minutes may be used.

Continue to add portions of acid and to measure pH after 30 min stirring/agitation after each portion added. Repeat until the entire pH interval from the natural pH (B.1.3.2) 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.1.3.4 Base addition

Add a portion of base (B.1.1.d) into the other of the bottles from B.1.3.2, 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 minutes may be used.

Continue to add portions of base and to measure pH after 30 min stirring/agitation after each portion added. Repeat until the entire pH interval from the initial pH (B.1.2.1) 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.1.4 Expression of results

Make a curve of pH versus amounts of acid and base expressed in molH⁺/kg and molOH⁻/kg.

B.2 Arbitrary division of the maximum acid/base consumption for the extreme pH values

B.2.1 General

For 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.2.2 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), 0.1 – 14.4 mol/l;
- e) Sodium hydroxide, NaOH, 0.1 – 5 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.2.3 Test portion

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

B.2.4 Procedure

B.2.4.1 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 :

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

where

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

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

Record the amount V of water added.

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

B.2.4.2 Natural pH

Put the two filled bottles on the agitation device. Agitate/stir for 1 hour. Determine the pH of the eluate directly in the bottles after settling for 10 minutes.

B.2.4.3 Acid titration

Titrate one of the bottles in B.2.4.1 with acid (B.2.2.d) in the titration equipment set at pH 4. Confirm the acid consumption over a titration period of 24 hours. 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 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).

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B.2.4.4 Base addition

Titrate the other of the bottles in B.2.4.1 with base (B.2.2.e) in the titration equipment set at pH 12. Confirm the base consumption over a titration period of 24 hours. 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.2.5 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 molH+/kg, prepare 10 different acid solutions from 50 mmolH+/l to 500 mmolH+/l at evenly-spaced intervals.

NOTE 2 This method allows to limit 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 to choose 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 term 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

From previous work [2,3,4,6,7,8,9] 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 8.3.1 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 material, soil and construction materials are given, which can be used as starting point to carry out a pH dependence test. In Table C.1 the underlying data are given (expressed in mol/kg). 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 of the standard.

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:

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

where

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

ANC_{pHX} is the acid neutralization capacity in Mol/kg from table 1 at pH = X;

N_{pHX} is the acid normality selected based on the buffer capacity around pH=X in Mol/l;

M is the sample weight in kg.

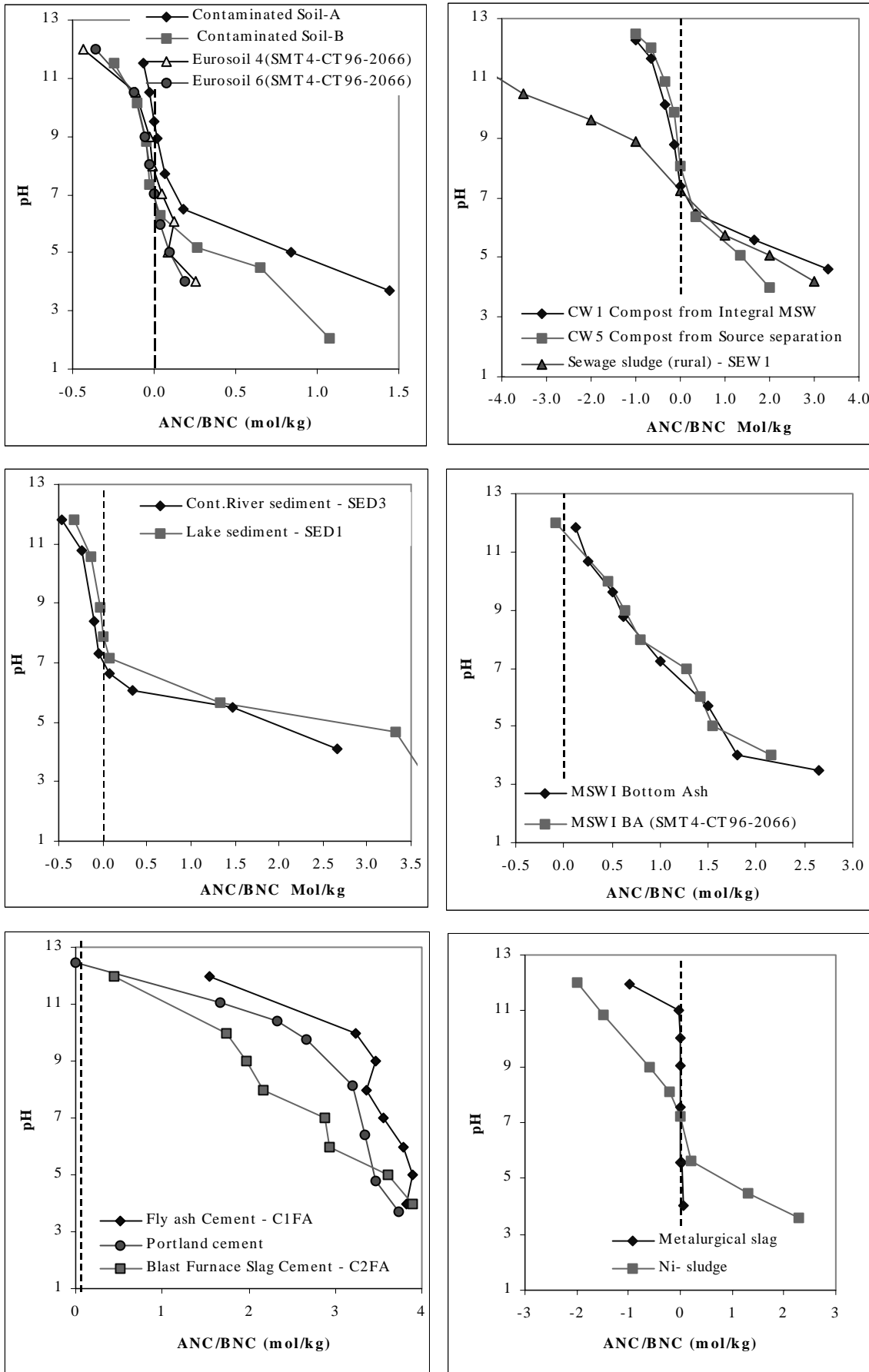


Figure C.1 — Acid/base neutralization curves for a wide range of materials

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

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