TECHNICAL SPECIFICATION

ISO/TS 21268-4

First edition
2007-11-15

Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials —

Part 4:
Influence of pH on leaching with initial acid/base addition

Qualité du sol — Modes opératoires de lixiviation en vue d'essais chimiques et écotoxicologiques ultérieurs des sols et matériaux du sol —

Partie 4: Essai de dépendance au pH avec ajout initial d'acide/base

Reference number
ISO/TS 21268-4:2007(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

COPYRIGHT PROTECTED DOCUMENT

© ISO 2007

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 21268-4 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Soil and site assessment*.

ISO/TS 21268 consists of the following parts, under the general title *Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials*:

- *Part 1: Batch test using a liquid to solid ratio of 2 l/kg dry matter*
- *Part 2: Batch test using a liquid to solid ratio of 10 l/kg dry matter*
- *Part 3: Up-flow percolation test*
- *Part 4: Influence of pH on leaching with initial acid/base addition*

Introduction

In various countries, tests have been developed to characterize and assess the constituents that can be released from materials. The release of soluble constituents upon contact with water is regarded as a main mechanism of release, resulting in a potential risk to the environment during the use or disposal of materials. The intent of these tests is to identify the leaching properties of materials. The complexity of the leaching process makes simplifications necessary.

Not all of the relevant aspects of leaching behaviour can be addressed in one standard.

Tests to characterize the behaviour of materials can generally be divided into three categories (see References [1], [2] and [4]). The relationships between these tests are summarized below.

a) “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, role of dissolved organic carbon (DOC), ageing of material and physical parameters, are addressed in these defined tests.

b) “Compliance” tests are used to determine whether the material complies with a specific behaviour or with specific reference values. These tests focus on key variables and leaching behaviour previously identified by basic characterization tests.

c) “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 method belongs to category a) “Basic characterization” tests.

NOTE Up to now, the test procedures described in this part of ISO/TS 21268 have not been validated.

Soil quality — Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials —

Part 4:
Influence of pH on leaching with initial acid/base addition

1 Scope

This part of ISO/TS 21268 specifies a test method to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials.

It applies to the determination of the influence of pH on the leachability of inorganic and organic constituents from soil and soil material, and the ecotoxicological effects of eluates with respect to microorganisms, fauna and flora. The test is not suitable for constituents that are volatile under ambient conditions. The equilibrium condition, as defined in this part of ISO/TS 21268, is established by the addition of predetermined amounts of acid or base to reach desired final pH values.

The test procedure specified in this part of ISO/TS 21268 produces eluates that are subsequently characterized by physical, chemical and ecotoxicological standard methods.

For the purposes of ecotoxicological tests, the relevant pH range (see 9.2) will usually be pH 5 to 9.

NOTE 1 Volatile organic constituents include the low molecular weight components in mixtures such as mineral oil.

NOTE 2 It is not always possible to optimize test conditions simultaneously for inorganic and organic constituents and optimum test conditions may also vary between different groups of organic constituents. Test requirements for organic constituents are generally more stringent than those for inorganic constituents. The test conditions suitable for measuring the release of organic constituents will generally also be applicable to inorganic constituents.

NOTE 3 For ecotoxicological testing, eluates representing the release of both inorganic and organic contaminants are needed. In this document, ecotoxicological testing is meant to include genotoxicological testing.

This test cannot be used alone to determine the total leaching behaviour of a soil. More leaching tests are needed for that extended goal. This part of ISO/TS 21268 does not address issues related to health and safety. It only determines the leaching properties outlined in Clause 5.

2 Normative references

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

ISO 7027, *Water quality — Determination of turbidity*

ISO 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 10381-3, *Soil quality — Sampling — Part 3: Guidance on safety*

ISO 10381-4, *Soil quality — Sampling — Part 4: Guidance on the procedure for investigation of natural, near-natural and cultivated sites*

ISO 10381-5, *Soil quality — Sampling — Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*

ISO 10523, *Water quality — Determination of pH*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

3 Terms and definitions

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

3.1
leaching test
test during which a soil or soil material is put into contact with a leachant under strictly defined conditions and some constituents of the material are extracted

3.2
leachant
liquid used in a leaching test

3.3
eluate
solution obtained by a laboratory leaching test

3.4
single batch leaching test
leaching test in which a fixed amount of material is leached in one step with a fixed amount of leachant

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 litres per kilogram (l/kg) of dry matter.

3.6
dry matter content
w_{dm}
ratio, expressed in percent, between the mass of the dry residue, determined in accordance with ISO 11465, and the corresponding raw mass

3.7
water content
w_{H2O}
ratio, expressed in percent, between the mass of water contained in the material as received and the corresponding dry residue of the material.

NOTE The basis for the calculation of the moisture content is the mass of the dry residue in this part of ISO/TS 21268, as specified in ISO 11465 (for the determination of the water content of soil).

3.8
laboratory sample

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

[IUPAC:1997]

3.9
test sample

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

[IUPAC:1997]

3.10
test portion

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

[IUPAC:1997]

3.11
soil material

excavated soil, dredged materials, manufactured soils, treated soil and fill materials

[ISO 15176:2002]

4 Symbols and abbreviations

ANC	acid neutralization capacity
BNC	base neutralization capacity
C_B	is the concentration of the base (mol/l) (see 7.4)
DOC	dissolved organic carbon
L/S	liquid to solid ratio
m_D	is the mass of the dried sample (kg)
m_W	is the mass of non-dried sample (kg)
n_B	is the base consumption for the particular pH (mol /kg OH^+ dry matter)
t_0	time at the start of the leaching test
$V_\mathrm{A,}$ V_B	volume of acid /base used in leachant
V_d	volume of demineralized water used in leachant
V_L	volume of prepared leachant
w_dm	dry matter content of the soil
w_H2O	water content

5 Principle

Several separate test portions (up to eight) are leached at a fixed L/S ratio (L/S = 10 l/kg) with leachants containing different preselected amounts of acid or base containing a low concentration (0,001 mol/l) of calcium chloride in order to reach stationary pH values at the end of the extraction period (see 8.4). Each leachant is added in three steps in the beginning of the test. In the full test eight final pH-values are required, covering 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 an equilibrium condition can be assumed to be reached for most constituents in most soil materials to be characterized. The equilibrium condition, as defined in this part of ISO/TS 21268, is verified at the end of the extraction period.

The results are expressed in milligrams per litre (mg/l) of constituents for each final pH value. For each final pH value, the quantity of acid that is added is also expressed in mol/kg H^+ dry matter and the quantity of base that is added is expressed as negative mol/kg H^+ dry matter.

NOTE 1 This test can also be performed using continuous pH control. The results are generally consistent (see Annex B).

NOTE 2 Other expressions of results are possible (including mg/kg of dry matter).

From the amount of acid and base used to reach a given end pH, the acid or base neutralization capacity (ANC, BNC) of the soil or soil material can also be determined.

NOTE 3 The pH range covered by the test can be restricted to a pH range relevant for the specific material and the considered problem (see 9.2).

NOTE 4 The leachant is made with 0,001 mol/l $CaCl_2$ to minimize the mobilization of DOC caused by a too-low ionic strength of the leachant. At the level of 0,001 mol/l $CaCl_2$ the complexation of metals with chloride is considered to be negligible.

The constituents in the eluate(s) are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates. The eluate may also be applied for subsequent ecotoxicity or genotoxicity testing.

After the test, the leaching conditions (in terms of pH, electrical conductivity, DOC and, optionally, turbidity and redox potential dictated by the material) are recorded.

NOTE 5 These parameters often control the leaching behaviour of soil materials and are therefore important for checking the leaching test.

6 Apparatus

6.1 Borosilicate glass, of high purity in accordance with ISO 5667-3, with a nominal volume of 1 l, glass bottles having caps of inert material, for example, PTFE (polytetrafluoroethylene). Rinsing is compulsory.

NOTE 1 If only inorganic parameters are analysed, alternative materials such as HDPE/PP bottles can be used, except for unpreserved samples for mercury analysis.

NOTE 2 If Boron analyses are necessary, any plastics bottles may be used, e.g. PTFE (polytetrafluoroethylene).

The nominal volume of 1 l is selected in combination with the mass, m_D, of 60 g, in order to minimize head-space. For m_D = 15 g and 30 g, bottle sizes of, respectively, 250 ml and 500 ml shall be used. In the case of materials with low densities, a deviation from this requirement can be necessary while still attempting to minimize headspace. This deviation should be reported.

Glass of high quality is considered adequate for both inorganic and organic contaminants, particularly since the pH range usually covered in soil testing does not reach the conditions (pH > 12 and pH < 4) where the glass itself is attacked. For ecotoxicity testing, eluates with both inorganic and organic contaminants are needed, which emphasizes the need to generate integrated eluates.

6.2 End-over-end tumbler (5 min^{-1} to 10 min^{-1}) **or roller table**, rotating at about 10 min^{-1}.

Other shaking devices can be used, provided that they can be shown to provide equivalent results. These agitation devices are specified for excessive abrasion leading to significant particle size reduction.

6.3 Filtration apparatus, either a vacuum filtration device (between 2,5 kPa to 4,0 kPa) or a high pressure filtration apparatus ($<$ 0,5 MPa). Cleaning is compulsory.

6.4 Pre-rinsed 0,45 µm membrane filters, for filtration (e.g. rinsed with 0,1 mol/l HNO_3 as described in 7.2, and water as described in 7.1).

The filters shall be glass fibre filters without organic glue or regenerated cellulose (S&S[1], RC 55[1] or comparable quality). The filter material shall be compatible with the extractant solution to be tested.

NOTE If only inorganic contaminants are to be analysed, alternative filter materials can be selected, e.g. cellulose acetate, PTFE.

6.5 Sieving equipment, with sieves of 2 mm nominal screen size.

NOTE Due to sieving, contamination of the sample can occur to an extent which affects the leaching of some constituents of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment or plasticizers from plastic sieves.

6.6 Centrifuge, operating at 20 000 g to 30 000 g using centrifuge tubes of FEP (fluorinated ethylene propylene) or tubes of an alternative material, which is inert with regard to both inorganic and organic compounds and suitable for high-speed centrifugation.

Alternatively, if a high-speed centrifuge is not available, a centrifuge operating at 2 000 g to 2 500 g using glass bottles may be used in combination with increased centrifugation time. Cooling shall be applied to maintain the desired temperature.

6.7 Glass bottles, with screw cap and PTFE (polytetrafluoroethylene) inlay for centrifugation.

6.8 Device for measuring electrical conductivity.

6.9 pH meter, in accordance with ISO 10523.

6.10 Redox potential meter (optional).

6.11 Balance, with an accuracy of at least 0,1 g.

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

6.13 Turbidity meter, as specified in ISO 7027.

6.14 Crushing equipment, a jaw crusher.

NOTE Due to particle size reduction, contamination of the sample can occur to an extent which affects the leaching of some constituents of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment.

---

[1] S&S and RC 55 are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO/TS 21268 and does not constitute an endorsement by ISO of these products.

7 Reagents

Reagents used shall be of analytical grade purity.

7.1 Leachant, demineralized water, deionized water or water of equivalent purity ($5 < pH < 7{,}5$), with a conductivity of $< 0{,}5$ mS/m in accordance with grade 3, specified in ISO 3696, made up to 0,001 mol/l $CaCl_2$.

If organic contaminants are considered, a blank test should be included to prove that no background contamination exists in the leachate.

7.2 Rinsing solutions: nitric acid 0,1 mol/l (analytical grade) and/or organic solvent (acetone).

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

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

8 Sample pretreatment

8.1 Sample size

Obtain a representative laboratory sample of at least 1 kg (dry matter) of the material. Use a sample splitter (6.12) or apply coning and quartering to split the sample.

Sampling should be performed in accordance with the guide to the preparation of a sampling plan for soil materials, as specified in ISO 10381-1 to ISO 10381-5, in order to obtain representative laboratory samples.

If the proportions in terms of L/S are maintained, larger volumes of eluate can be produced if needed for ecotoxicity testing. Deviations to compensate for volume requirement shall be reported in the test report.

The size of the laboratory sample is dependent on the particle size distribution of the soil to be analysed (refer to sampling standard). The prescribed sample size will generally be adequate. In specific cases, a smaller sample size can be accepted (for instance, if for specific reasons, less material is available), provided that the test can be carried out as specified in 8.2. The deviation shall be recorded in the test report.

8.2 Particle size reduction

The tests shall be carried out, preferably, on material as received. However, the test portion to be prepared shall have at least 95 % (mass fraction) of grain size $\leqslant 4$ mm. If required, the laboratory sample shall be sieved (6.5). Oversized natural material (e.g. stones, pebbles, twigs) in the sample shall be separated and discarded. The mass and nature of the oversized material shall be recorded. If oversized material is not of natural origin and exceeds 5 % (mass fraction), the entire oversized fraction shall be crushed with suitable crushing equipment. On no account shall the material be finely ground. Irrespective of any necessary size reduction, the separate fractions with the exception of the non-crushable material shall be mixed to constitute the test sample. Only in the case that the laboratory sample cannot be crushed or sieved because of its moisture content, the laboratory sample may be dried. The drying temperature shall not exceed 25 °C.

NOTE If national regulations specify other particle sizes, these can be applied.

Sieving and drying at 25 °C can still lead to loss of some volatile components (inorganic and organic). In this case, particle size reduction and drying is best avoided, as it can alter the leaching characteristics.

8.3 Determination of the dry matter content and of water content

The whole test sample, complying with the size criterion in 8.2, shall not be further dried. The moisture content of the test sample shall be determined on a subsample at (105 ± 5) °C. It shall be taken into account when adjusting the L/S ratio. The dry mass of the sample shall be determined at (105 ± 5) °C, in accordance with ISO 11465, and the dry matter content calculated in accordance with Equation (1):

$$w_{\mathrm{dm}} = 100 \cdot m_{\mathrm{D}} / m_{\mathrm{W}} \tag{1}$$

where

w_{dm} is the dry matter content, in percent (%);

m_{D} is the mass of the dried sample, in kilograms (kg);

m_{W} is the mass of non-dried sample, in kilograms (kg).

The water content (w_{H2O} in percent) is calculated in accordance with Equation (2):

$$w_{\mathrm{H_2O}} = 100 \cdot \left(m_{\mathrm{W}} - m_{\mathrm{D}}\right) / m_{\mathrm{D}} \tag{2}$$

If volatile or unstable compounds are present in the soil sample, this gravimetric method cannot be used for accurate determination of the water content (see ISO 11465).

If the soil sample is air-dried prior to testing, the dry matter content $w_{\mathrm{dm,AD}}$ of the air-dried sample shall be determined as well.

8.4 Preparation of test portion

Prepare at least eight test portions by the use of a sample splitter or by coning and quartering. Based on sample heterogeneity and eluate volume requirement for analysis, the test portion size shall be either m_{D} = 15 g, 30 g or 60 g (with an accuracy of ± 1 g).

Calculate the non-dried mass of the test portion, m_{W}, to be used for the test, in accordance with Equation (3):

$$m_{\mathrm{W}} = \frac{m_{\mathrm{D}}}{w_{\mathrm{dm}}} \cdot 100 \tag{3}$$

where

m_{D} is the dried mass of the test portion (g);

m_{W} is the non-dried mass of the test portion (g);

w_{dm} is the dry matter content of the soil (%).

9 Procedure

9.1 Contact time

The leaching procedure consists of three defined stages:

- Period A (acid/base addition) from t_0 to (t_0 + 4 h) for acid/base additions in three steps;
- Period B (equilibration period) from (t_0 + 4 h) to (t_0 + 44 h) equilibration period;
- Period C (verification period) from (t_0 + 44 h) 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.

9.2 pH-range

The full test shall cover the range pH 4 to pH 12 (both included, i.e. the lowest value $\geqslant 4$ and the highest value $\leqslant 12$) with eight 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.

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.

The pH range covered by the test may be restricted to a pH range relevant to the specific material and the considered problem. The pH range to be covered may depend on the specific properties of the soil 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, for example, for the purpose of ecotoxicological tests; the relevant pH range will usually be pH 5 to 9.

9.3 Leaching test

9.3.1 General

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

9.3.2 Preparation of leachant

Identify the acid or base consumption for reaching the relevant pH values as A (mol/kg H^+ dry matter) or B (mol/kg OH^- dry matter) and the total volume of leachant.

NOTE The acid or base consumption for the considered pH values can 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 ± 0,2 (l/kg) for the actual size of test portion m_w (see 8.4), including the volume of acid or base, in accordance with Equation (4):

$$V = 10 \cdot m_\mathrm{D} \tag{4}$$

where

V is the total volume of the added extractant solution, in millilitres (ml);

m_D is the dry mass of the test portion, in grams (g) (see 8.4);

10 is the factor, in litres per kilogram (l/kg).

NOTE In relation to the L/S-ratio, V in this equation is equivalent to “L” and m_D is equivalent to “S”.

Calculate the amount of leachant to be added to the actual size of test portion and compensate for the water content in the test portion in accordance with Equation (5):

$$V_\mathrm{L} = V - (\frac{100}{w_\mathrm{dm}} - 1) \cdot m_\mathrm{D} \tag{5}$$

where

V is the total volume of the added extractant solution, in millilitres (ml);

m_D is the dry mass of the test portion, in grams (g) (see 8.4);

w_{dm} is the dry matter of the soil, in percent (%) (see 8.3);

V_{L} is the volume of prepared leachant, in millilitres (ml).

Prepare the leachant from demineralized water (see 7.1) and acid or base (see 7.2 or 7.4) according to the acid/base consumption for the relevant pH.

Prepare the acid-adjusted leachant in accordance with Equation (6) and Equation (7):

$$V_{\mathrm{L}} = V_{\mathrm{d}} + V_{\mathrm{A}} \tag{6}$$

$$V_{\mathrm{A}} = \frac{n_{\mathrm{A}} \cdot m_{\mathrm{D}}}{C_{\mathrm{A}}} \tag{7}$$

where

V_{L} is the volume of prepared leachant, in millilitres (ml);

V_{d} is the volume of demineralized water used, in millilitres (ml);

V_{A} is the volume of acid needed, in millilitres (ml);

n_{A} is the acid consumption for the particular pH, in mol/kg H^+ dry matter;

m_{D} is the dry mass of the test portion, in grams (g) (see 8.4);

C_{A} is the concentration of the acid, in moles per litre (mol/l) (see 7.2).

Prepare the base-adjusted leachant in accordance with Equations (8) and (9):

$$V_{\mathrm{L}} = V_{\mathrm{d}} + V_{\mathrm{B}} \tag{8}$$

$$V_{\mathrm{B}} = \frac{n_{\mathrm{B}} \cdot m_{\mathrm{D}}}{C_{\mathrm{B}}} \tag{9}$$

where

V_{L} is the volume of prepared leachant, in millilitres (ml);

V_{d} is the volume of demineralized water used, in millilitres (ml);

V_{B} is the volume of base needed, in millilitres (ml);

n_{B} is the base consumption for the particular pH, in mol /kg OH^- dry matter;

m_{D} is the dry mass of the test portion, in grams (g) (see 8.4);

C_{B} is the concentration of the base, in moles per litre (mol/l) (see 7.4).

Split the volume V_{L} of leachant into three equal parts, $V_{\mathrm{L}}/3$.

9.3.3 Leaching procedure

Carry out the test at a temperature of (20 ± 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 bottle sizes of, respectively, 250 ml, 500 ml and 1 000 ml.

Clean the bottle before use by filling it with 1 mol/l nitric acid (see 7.3), leaving it for at least 24 h and then rinsing it with demineralized water (see 7.1).

Place one of the test portions in the rinsed bottle.

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

a) first fraction at t_0;

b) second fraction at $t_0 + 30$ min;

c) third fraction at $t_0 + 2$ h.

Close the bottle and agitate the suspension (see 7.2) between each leachant addition. Measure and record the pH before the addition of the second and third fractions, and, if deviations are observed from the expected pH at that time, prepare additional bottles with modified acid/base additions.

Development of carbon dioxide should be taken into account when using acidic leachants. This may lead to pressure build-up. When this is expected or observed, the pressure can be relieved by opening the bottle a few times during the test. Main gas production will, however, take place in the first period of acid addition.

Continue to agitate after the last leachant addition until $t = t_0 + 48$ h.

NOTE 1 For the high pH values ($\text{pH} > 9$), CO_2 uptake can affect the leaching process. This can 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 the pH, stop the agitation and allow the mix to settle for 5 min. Measure the pH by inserting the clean electrode into the supernatant.

NOTE 2 The pH value at $t_0 + 4$ h is used for checking that sufficient pH adjustment has been obtained by the acid or base additions, respectively.

Since the pH is measured directly in the suspension, rinse the pH electrode thoroughly and dry softly 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 assigned to the analysis of the eluate.

NOTE 3 The pH is measured directly in the bottle at $t_0 + 48$ h, since filtration can 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 approaching an equilibrium condition. A special note shall be made in the report if this requirement is not met.

If too many experimental points deviate (more than three amongst eight), the conclusion is that this test is not applicable to this material.

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

Allow the suspended solids to settle for (15 ± 5) min.

Transfer the supernatant to centrifuge tubes (6.7).

There are two options for solid-liquid separation.

1) Centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high-speed centrifuge (6.6).

2) Centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower speed centrifuge (6.6).

Cooling shall be applied to maintain the temperature at (20 ± 5) °C (see 6.6).

NOTE 4 Based on Stoke's law, the results of both centrifugation methods are expected to be comparable.

Gentle breaking shall be applied in order to avoid resuspension. Deceleration time shall not exceed 20 min.

After centrifugation, the eluate shall be transferred immediately to an appropriate container for measurement of pH, redox potential (see also the paragraph above starting with "When equilibrium conditions".) and stored for subsequent chemical analysis and/or ecotoxicological testing.

If only inorganic constituents are measured, the eluate can be filtered using the appropriate membrane filters (6.4) and a vacuum or pressure filtration device (6.3). When filtration as specified is not possible in less than 1 h with a liquid flow rate of at least 30 ml/cm^2/h, a liquid-solid separation procedure, specific for the considered case, shall be applied. Report the details in the test report. This specific procedure shall not include the use of additives.

For inorganic constituents, it is often preferable to precentrifuge the eluate at 2 000 g to 3 000 g for 20 min using glass bottles with screw cap and polytetrafluoroethylene inlay (or, if possible, using the leaching bottle directly) prior to filtration.

NOTE 5 Such a specific liquid-solid separation procedure can include settling, prefiltration on coarser filter, centrifugation, filtration on large size membrane filter, filtration at high pressure, filtration at increasing high pressure following a first period without pressure, etc.

Determine the volume of eluate V_E.

Measure immediately the electrical conductivity (in millisiemens per metre, mS/m), temperature, DOC and pH of the eluate. Measurement of turbidity and redox potential E_h in millivolts (mV) is highly recommended.

Analysis of DOC in the eluate is needed, as this property is relevant both for release of inorganic substances, as well as for organic substances. Proceed immediately with the eluate treatment, as specified in Clause 10.

Annex A contains an example of a specific liquid-solid separation procedure for soil sample.

9.4 Natural pH

Repeat 9.3 for a test portion without acid or base addition, when determining the volume of demineralized water to be added. Measure the pH after 4 h, 44 h and 48 h, as in 9.3.

A very low conductivity in the eluate at natural pH can increase generation of colloids, which can clog filters and increase analysed concentrations. This is minimized by addition of $CaCl_2$ in the leachant at a concentration of 0,001 mol/l.

10 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 ISO 5667-3.

Determine the concentrations of constituents of interest using the methods of eluate analysis (e.g. ISO 11047).

When using eluates for bioassays, they shall be processed as soon as possible, but storage shall not exceed 7 d at 4 °C.

11 Analytical determination

Since the analysis step is not included in the scope of this part of ISO/TS 21268, the analytical method specified in the International Standards dedicated to such analysis shall be incorporated, including the date of analysis, the storage conditions until the analysis is performed, the quantification limit of the analytical method being used, the address of the laboratory and the name(s) of operator(s).

12 Blank test

The contribution of the leachant, devices and membrane filters to the results 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 procedures specified in 9.3 and Clause 10 without the solid material with the addition of:

a) both the maximum amounts of acid and base to the leachant of similar volume as used in the tests;

b) acid to obtain pH 4;

c) 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.

13 Calculation

The analysis of the eluate(s) produced by the leaching test provides the concentrations of the constituents in the eluate(s), expressed in milligrams per litre (mg/l). The final results are expressed as the amount of constituent leached relative to the total mass of the sample, in milligrams per kilogram (mg/kg) of dry matter.

Calculate the quantity of a constituent leached from the material, based on the dry mass of the original material, from Equation (10):

$$w(\mathrm{X}) = \rho_1(\mathrm{X}) \cdot \left[\left(V_\mathrm{L} / m_\mathrm{D} \right) + \left(w_{\mathrm{H_2O}} / 100 \right) \right] \tag{10}$$

where

$w(\mathrm{X})$ is the release of a constituent X at a L/S = 10, in milligrams per kilogram (mg/kg of dry matter);

ρ_1 (X) is the analyte concentration, in milligrams per litre (mg/l);

V_L is the volume of leachant used, in litres (l);

w_{H2O} is the water content, as calculated in Equation (2);

m_D is the mass of the dried test portion, in kilograms (kg).

14 Test report

The test report shall refer to this part of ISO/TS 21268 and include the following details:

a) any information necessary for the complete identification of the sample;

b) the test results;

c) any details that are optional or deviations from the specifications of this part of ISO/TS 21268, and any effects which may have affected the results.

15 Performance characteristics

When this part of ISO/TS 21268 was adopted by ISO, the test specified in this document was not validated and no data were available on robustness, repeatability and reproducibility.

Annex A
(informative)

Example of a specific liquid-solid separation procedure for soil sample

A.1 Introduction

The original scope of leaching tests covers in particular solid substances containing larger amounts of dissolved salts. The general feasibility of these methods has limitations, when the solubility of substances is determined in soil sample, in particular when, for example, oxidized, adsorbed or organically-bound heavy metals are rather insoluble in those materials. Despite their low solubility, heavy metals are important from an environmental point of view. The lower the “pure” solubility of heavy metals in a contaminated soil sample, the bigger is the relative influence of colloidal particle portions in eluate on the end result.

Especially in the case of fine-textured soil samples being rich in humus but poor in electrolytes, the filter cake produced during filtration exhibits very fine pores and less colloids pass through the membrane filter. Thus the production of filter cake largely affects the “solubility” of heavy metals, which is identified by this method. To obtain comparable results, it is necessary to stipulate the factors determining the height of the filter cake. In addition to sample-specific properties, the thickness of the filter cake is determined predominantly by the filter diameter and the volume of the eluate to be filtered.

A.2 Apparatus

A.2.1 Pressure filtration unit for membrane filter (diameter 142 mm).

A.2.2 Membrane filter of pore size 0,45 µm.

If another filter size is used, the volume to be filtered is modified according to the filter surface; the essential precondition is that the relationship between the volume to be filtered and the filter surface is complied with [relationship: about 1 l volume to 158 cm^2 filter surface (diameter 142 mm)].

A.2.3 Media-guiding material (in contact with extracts), in polytetrafluoroethylene.

A.3 Procedure

For sedimentation of the larger particles, allow the suspension to stand for 15 min after shaking.

Decant almost completely the supernatant liquid into a centrifuge tube or bottle device.

Apply centrifugation (30 min at 2 000 g).

Almost complete decanting of supernatant liquid into the membrane pressure filter apparatus.

Apply, after 5 min of filtration without pressure, a pressure of 100 kPa (1 bar) to accelerate filtration. If after 15 min less than two thirds of the eluate have passed through the filter, increase the pressure to 200 kPa (2 bar). If necessary, increase the pressure to a maximum of 350 kPa (3,5 bar) after 30 min. Continue the filtration until all the supernatant of centrifugation has passed through the filter. If the filtration is still incomplete after 2 h, stop the filtration, collect the incomplete filtrate and prepare it for analysis.

By using this procedure, a significant reduction in the possible errors resulting from the proportions of colloidal, dissolved heavy metals, in filtrate can be ensured. Consequently, it is not allowed to decant the first part of the filtrate and to put it again on the filter – a method that is quite common in several laboratories.

Annex B
(informative)

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

B.1 Comparison of the mode of operation of the test with the pH continuous control mode — Influence of pH on the leaching behaviour

Both modes are aimed at determining the influence of pH on the release of the inorganic and organic constituents from a soil sample (check in the whole document).

In the test described in this document, an equilibrium condition is established at different pH values, as a result of the reaction between preselected amounts of acid or base and test portions of the soil. Size reduction is performed to accelerate reaching an equilibrium condition.

In addition to the pH influence on leaching, the test addressing the influence of pH on leaching by continuous pH control (see CEN/TS 14997) is suitable for solubility control at a precisely specified pH. The test addressing the influence of pH on leaching by continuous pH control can be particularly suitable when materials are tested which have a very low buffer capacity, or in the case of measurement of pH influence on leaching at a pH where a small pH change leads to strong change in release.

B.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 B.3):

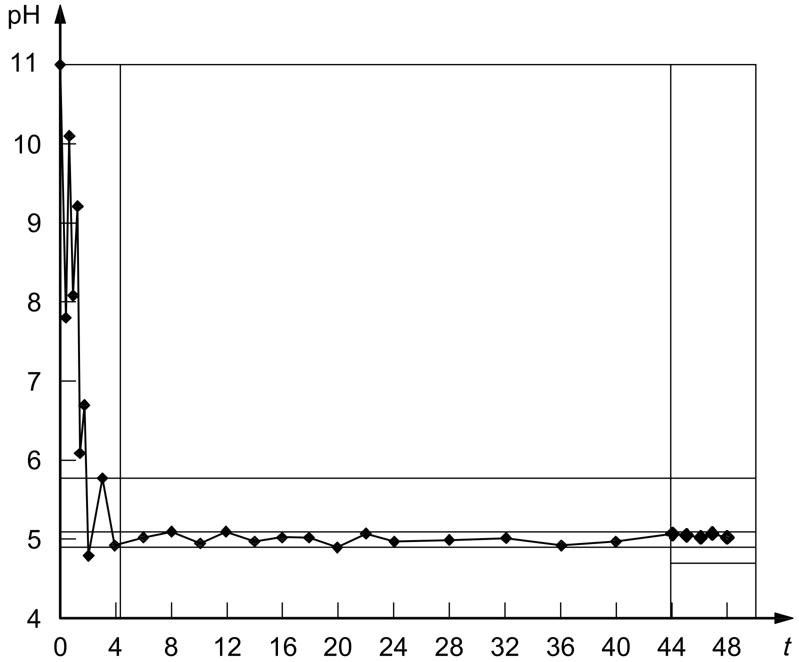
a) pH at $t_0 + 48$ h (see 9.3.3) versus the amounts of acid/base added (ANC and BNC curve), expressed in mol/kg H^+/OH^- of dry matter;

b) concentration of each analysed constituent in eluates in log scale, in milligrams per litre (mg/l), versus pH at $t_0 + 48$ h;

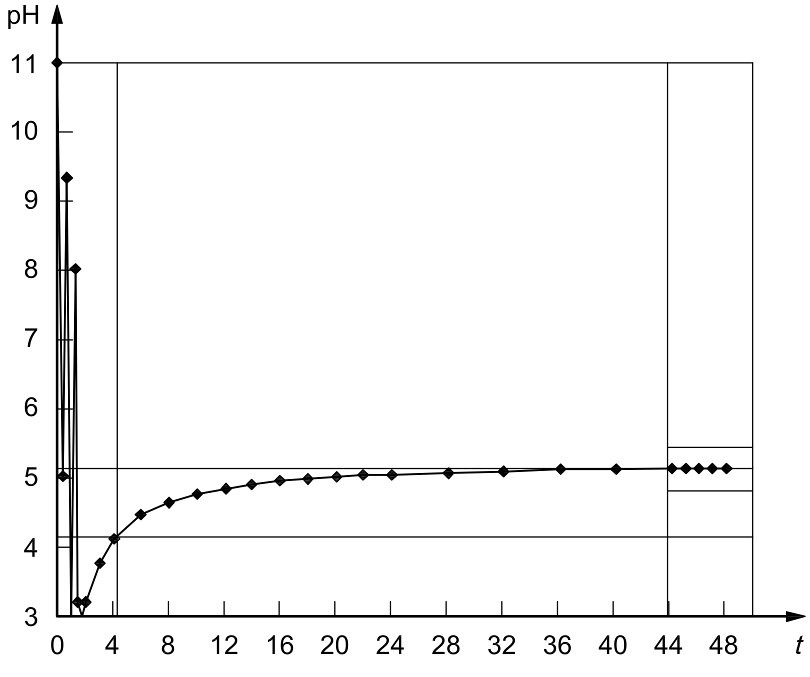
c) concentration of each analysed constituent in eluates in linear scale, in milligrams per litre (mg/l), versus the amounts of acid or base added (mmol of H^+/OH^- per kg of dry matter).

NOTE In the third visual representation, the pH at $t_0 + 48$ h can also be added as a second ordinate.

In the case when the leached amounts (U_x) (mg/kg of dry soil) are needed, they can be directly calculated by multiplying the concentrations in milligrams per litre (mg/l) by the L/S value (usually L/S = 10 l/kg of dry material).



a) pH STAT-MODE



b) ANC MODE

Figure B.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 of about 5

B.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 part of ISO/TS 21268. It does not directly take the effects of other parameters, such as the influence of acids and bases other than the nitric acid/sodium hydroxide used in the test, dissolved organic carbon, complexation, redox conditions, into account.

This test method 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 soil under specific conditions.

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

a) qualify and quantify the material resistance to acid-basic attack through the concentration measured, as a function of the pH and the acid/base amount needed to reach a given final pH;

b) 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, GEOCHEM WORKBENCH, PHREEQC, ECOSAT, ORCHESTRA, CHESS, SPEC, etc.). In many cases, it also provides insight in the relevance of particular solubility controls and release mechanisms (e.g. formulate a hypothesis on the dissolution mechanisms);

c) provide a basis of reference for different leaching tests, as it has been shown that pH is one of the major controlling factors distinguishing tests from one another;

d) compare leaching behaviour, with respect to pH, of the same parameter from different soils or different material classes to be able to demonstrate similarities in solubility controlling conditions irrespective of material matrix;

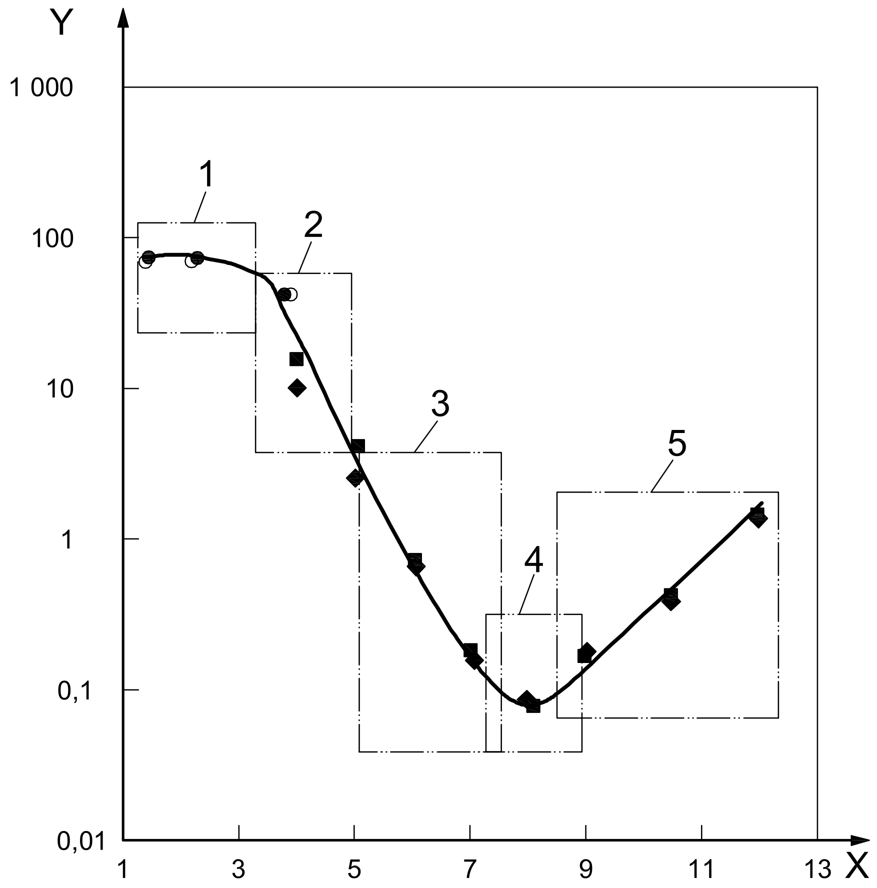
e) provide data to feed dynamic behavioural models, for instance, under the following relationship: solubilization $= f$(pH or meq H^+/g) in the physico-chemical context linked with the presence of the other compounds in the material. This is not always possible with available literature data.

On the contrary, this test is not meant for

- quantifying a maximum removable fraction, as the concentrations obtained correspond to a steady state situation close to chemical equilibrium. For example, the values obtained for the lowest pH and at high pH can only be considered as approaching the maximum removable fraction of, respectively, metals and oxyanions;
- simulating actual situations in specific scenarios, because, in addition, at least information on low L/S will be needed.

B.4 Example: 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 B.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 characterization 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 B.2, the leaching behaviour under the influence of pH is illustrated for cadmium (Cd) from heavily sewage sludge amended soil [1]. An indication of the repeatability of the method can be obtained from the duplicate test data. The test was performed with an initial acid/base addition. Figure B.2 also indicates pH ranges typical of some “soil” – “conditions of scenarios” combinations.



Key

X pH

Y Cd leached at L/S = 10 mg/kg

◆ initial addition mode

■ duplicate

1 ingestion/inhalation

2 acidic environment

3 natural soil

4 soil liming

5 cement stabilization of contaminated soil

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

Annex C
(informative)

Preliminary determination of the acid/base consumption

C.1 General

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

a) a titration procedure to estimate the ANC and the BNC (see C.2);

b) an arbitrary division of the maximum acid/base consumption for the extreme pH values (see C.3).

C.2 Titration procedure to estimate the ANC and the BNC

C.2.1 Reagents

C.2.1.1 **Nitric acid** (per analysis), 0,1 mol/l to 14,4 mol/l.

C.2.1.2 **Sodium hydroxide**, NaOH, 0,1 mol/l to 5 mol/l.

C.2.1.3 Distilled water, demineralized water or water of equivalent purity ($5 < \text{pH} < 7$), with a conductivity of $< 0,5$ mS/m.

C.2.2 Apparatus

C.2.2.1 **Bottles made of polypropylene (PP), PTFE or polyethylene (PE)**.

C.2.2.2 **Stirring or agitation device**, i.e. a magnetic stirring device, using a polytetrafluoroethylene (PTFE) coated magnetic stirring rod, or a mechanical stirring device, made of glass or PTFE.

C.2.2.3 **Titrator** (optional).

C.2.3 Test portion

Test portions are prepared in accordance with the procedure in 8.4. Based on sample heterogeneity, it is recommended that the test portion size is either m_D = 15 g, 30 g or 60 g (dry mass), with a tolerance of ± 1 g.

C.2.4 Procedure

C.2.4.1 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 demineralized 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:

$$V = 9\,m_{\mathrm{D}} - (m_{\mathrm{W}} - m_{\mathrm{D}}) \qquad \text{(C.1)}$$

where

m_{W} is the non-dried 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 $\leqslant$ 11.

C.2.4.2 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.

C.2.4.3 Acid titration

Add a portion of acid (C.2.1.1), manually or by use of the titrator, into one of the bottles from C.2.4.1 and determine the pH directly in the bottle after 30 min. Record the amount and concentration of acid added, and the pH obtained.

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

In the case of a high acid demand, the 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 adding portions of acid, and measure the pH after 0,5 h, stirring or agitating after each portion is added. Repeat until the entire pH interval from the natural pH (C.2.4.2) to pH 4 or below is obtained and the distance between the obtained pH values is smaller than the 1,5 pH values. Record the amounts and concentrations of acid added, and the pH values obtained.

C.2.4.4 Base addition

Add a portion of base (C.2.1.2) into the other of the bottles from C.2.4.1, and determine the pH directly in the bottle after 30 min. Record the added amount and concentration of base, and the obtained pH.

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

In the case of a high base demand, the 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 adding portions of base and measure the pH after 30 min, stirring or agitating after each portion is added. Repeat until the entire pH interval from the initial pH (C.2.4.2) to pH 12 or above is obtained and the distance between the obtained pH values is smaller than the 1,5 pH value.

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

C.2.5 Expression of results

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

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

C.3.1 General

For soils with a very strong acid-base capacity, manual titration may lead to an 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.

C.3.2 Reagents

C.3.2.1 **Nitric acid** (pro-analysis), 0,1 mol/l to 14,4 mol/l.

C.3.2.2 **Sodium hydroxide**, NaOH, 0,1 mol/l to 5 mol/l.

C.3.2.3 Distilled water, demineralized water or water of equivalent purity ($5 < \text{pH} < 7$), with a conductivity of $< 0{,}5$ mS/m.

C.3.3 Apparatus

C.3.3.1 **Bottles made of polypropylene (PP), PTFE or polyethylene (PE)**.

C.3.3.2 **Stirring or agitation device.** This is a magnetic stirring device, using a polytetrafluoroethylene (PTFE) coated magnetic stirring rod, or a mechanical stirring device, made of glass or PTFE.

C.3.3.3 **Titrator**.

C.3.4 Test portion

Test portions are prepared in accordance with the procedure in 8.4. Based on sample heterogeneity, it is recommended that the test portion size is either m_D = 15 g, 30 g or 60 g (dry mass) (with a tolerance of ± 1 g).

C.3.5 Procedure

C.3.5.1 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 demineralized water in the bottles establishing a liquid to solid ratio (L/S) of about 9. Calculate the volume, V, as follows assuming the density of water to be 1 g/ml:

$$V = 9m_D - \left(m_W - m_D\right) \qquad \text{(C.2)}$$

where

m_W is the non-dried 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 $\leqslant 11$.

C.3.5.2 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.

C.3.5.3 Acid titration

Titrate one of the bottles in C.3.3.1 with nitric acid (C.3.2.1) 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.

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

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.

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

C.3.5.4 Base addition

Titrate the other of the bottles in C.3.3.1 with base (C.3.2.2) 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.

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.

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

C.3.6 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, for example, between the natural pH and pH 4,5, pH values are sought, and if the maximum acid consumption is 5 mol/kg H+, prepare 10 different acid solutions from 50 mmol/l H^+ to 500 mmol/l H^+ at evenly-spaced intervals.

NOTE 2 This method allows limitation of the misevaluation of the ANC and BNC and the selection of the seven solutions to be analysed 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 behaviour.

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.

Bibliography

[1] EN 12920, *Characterization of waste — Methodology for the determination of the leaching behaviour of waste under specified conditions*

[2] CEN/TS 14429, *Characterization of waste — Leaching behaviour tests — Influence of pH on leaching with initial acid/base addition*

[3] CEN/TS 14997, *Characterization of waste — Leaching behaviour tests — Influence of pH on leaching with continuous pH-control*

[4] ISO 18772, *Soil quality — Guidance on leaching procedures for subsequent chemical and ecotoxicological testing of soils and soil materials*

[5] ISO 10381-6, *Soil quality — Sampling — Part 6: Guidance on the collection, handling and storage of soil under aerobic conditions for the assessment of microbiological processes, biomass and diversity in the laboratory*

[6] ISO 11047, *Soil quality — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc — Flame and electrothermal atomic absorption spectrometric methods*

[7] ISO 15176:2002, *Soil quality — Characterization of excavated soil and other soil materials intended for re-use*

[8] ISO 15799, *Soil quality — Guidance on the ecotoxicological characterization of soils and soil materials*

[9] Harmonisation of leaching/extraction tests, 1997. *Studies in Environmental Science*, **70** (eds. Van der Sloot, H.A., Heasman, L., Quevauviller, Ph., Elsevier Science, Amsterdam), 292 pp.

[10] Van der Sloot, H.A., Hjelmar, O., Bjerre Hansen, J., Woitke, P., Lepom, P., Leschber, R., Bartet, B. and Debrucker, N. Validation of CEN/TC 292 Leaching Tests and Eluate Analysis Methods PrEN 12457 parts 1-4, ENV 13370 and ENV 1250. In: *Co-operation with CEN/TC 308, 2001,* ECN-C-01-117

[11] Hund-Rinke, K., Kördel, W., Hennecke, D., Eisenträger, A. and Heiden, S. Bioassays for the Ecotoxicological and Genotoxicological Assessment of Contaminated Soils (Results of a Round Robin Test). Part I. Assessment of a Possible Groundwater Contamination: Ecotoxicological and Genotoxicological Tests with Aqueous Soil Extracts, *JSS - J. Soil & Sediments,* **2**(1), 2002, pp. 43-50

[12] Van der Sloot, H.A. Harmonisation of leaching/extraction procedures for sludge, compost, soil and sediment analyses. Chapter 7. In: *Single and Sequential Extraction Procedures for Soil and Sediment Fractionation Studies* (ed. Quevauviller, Ph., Royal Society of Chemistry), 2002, pp. 142-174

ICS 13.080.05

Price based on 23 pages