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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

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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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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
$w_{\mathrm{H_2O}}$
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
$V_{A,}$ 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 $m_{\mathrm{D}} = 15$ 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.

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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 ($5 < \text{pH} < 7{,}5$), 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.