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

Part 4:

**Influence of pH on leaching with initial
acid/base addition**

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*Qualité du sol — Modes opératoires de lixiviation en vue d'essais
chimiques et écotoxicologiques ultérieurs des sols et matériaux
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Partie 4: Essai de dépendance au pH avec ajout initial d'acide/de 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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Impact assessment*.

This first edition of ISO 21268-4 cancels and replaces ISO/TS 21268-4:2007, which has been technically revised. The main changes compared to the previous edition are as follows:

- the maximum grain size has been changed to <2 mm as usual for soil;
- references in [Clause 2](#) and the Bibliography have been updated;
- [Clause 12](#) "Performance characteristics" has been technically revised;
- a new informative [Annex D](#) "Repeatability and reproducibility data" has been added;
- a new informative [Annex E](#) "Calculation of centrifugation duration depending on centrifugation speed and rotor dimensions" has been added.

A list of all parts in the ISO 21268 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In various countries, tests have been developed to characterize and assess the substances which can be released from materials. The release of soluble substances upon contact with water is regarded as a main mechanism of release, which results 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^[1].

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 addressed in ISO 18772 and EN 12920. The relationships between these tests are summarized below.

“Basic characterization” tests are used to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials. Liquid/solid ratios (L/S), 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.

“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.

“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.

This document was originally elaborated on the basis of CEN/TS 14429:2005. Especially, modifications considering requirements on subsequent ecotoxicological testing and analysis of organic substances have been included. Validation results have been adopted from US-EPA^[5].

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

Part 4:

Influence of pH on leaching with initial acid/base addition

1 Scope

This document specifies a test to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials.

The document has been developed to measure the pH-dependent release of inorganic and organic substances from soil and soil-like material as well as to produce eluates for subsequent ecotoxicological testing. For ecotoxicological testing, see ISO 15799 and ISO 17616. The equilibrium condition, as defined in this document, is established by the addition of predetermined amounts of acid or base to reach desired final pH values.

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

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

NOTE 3 Within the category of organic substances, a significant difference in behaviour exists between the more polar, relatively water-soluble compounds and apolar, hydrophobic organic substances (HOCs). In the latter case, mechanisms of release (e.g. particle-bound or dissolved organic carbon-bound) can be more crucial as well as sorption losses of soluble HOCs on different materials with which they come in contact (e.g. bottles, filters). The test and the results should be used for leaching of organic substances only with thorough consideration of the specific properties of the substances in question and the associated potential problems.

NOTE 4 For ecotoxicological testing, eluates representing the release of both inorganic and organic substances are needed. In this document, ecotoxicological testing is meant to include genotoxicological testing.

The test method produces eluates, which can subsequently be characterized by physical, chemical and ecotoxicological methods in accordance with existing standard methods. The test is not suitable for substances that are volatile under ambient conditions.

For the purposes of ecotoxicological tests, the relevant pH range (see 8.2) will usually be pH 5 to pH 9.

This test is mainly aimed at being used for routine and control purposes, and it cannot be used alone to describe all leaching properties of a soil. Additional leaching tests are needed for that extended goal. This document does not address issues related to health and safety. It only determines the leaching properties outlined in [Clause 5](#).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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: Preservation and handling of water samples*

ISO 7027-1, *Water quality — Determination of turbidity— Part 1: Quantitative methods*

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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1 leaching test

test during which a material is put into contact with a *leachant* (3.2) under strictly defined conditions and some substances of the material are extracted

3.2 leachant

liquid used in a *leaching test* (3.1)

Note 1 to entry: For the purpose of this document, the leachant is specified in 6.1

3.3 eluate

solution recovered from a *leaching test* (3.1)

Note 1 to entry: Eluate is also referred to as leachate.

3.4 liquid to solid ratio

L/S

ratio between the total volume of liquid (L in litres), which in this extraction is in contact with the soil sample, and the dry mass of the sample (S in kg of dry matter)

Note 1 to entry: L/S is expressed in litres per kilogram (l/kg).

3.5 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

3.6 water content

w_{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 1 to entry: The basis for the calculation of the water content is the mass of the dry residue in this document, as specified in ISO 11465 (for the determination of the water content of soil).

3.7**laboratory sample**

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

3.8**test sample**

sample, prepared from the *laboratory sample* (3.7), from which *test portions* (3.9) are removed for testing or analysis

3.9**test portion**

quantity of material of appropriate size for measurement of the concentration or other properties of interest taken from the *test sample* (3.8)

Note 1 to entry: The test portion can be taken from the *laboratory sample* (3.7) directly if no pre-treatment of the sample is required, but usually it is taken from the test sample.

Note 2 to entry: A unit or increment of proper homogeneity, size and fineness, needing no further preparation, can be a test portion.

3.10**soil-like material**

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

4 Principle**iTeh STANDARD PREVIEW**

The test portions, which originally or after suitable pre-treatment have a particle size less than or equal to 2 mm, are brought into contact with water containing a low concentration (0,001 mol/l) of calcium chloride or demineralised water (5.1) under defined conditions. Several separate test portions (up to eight) are leached at a fixed L/S ratio ($L/S = 10\text{ ml/kg}$) with leachants containing different preselected amounts of acid or base 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 amount 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 substances in most soil-like materials to be characterized. The equilibrium condition, as defined in this document, is verified at the end of the extraction period.

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

NOTE 1 This test may 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 neutralization capacity (ANC) or base neutralization capacity (BNC) of the soil or soil-like 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 8.2).

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 substances 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-like materials and are therefore important for checking the leaching test.

5 Reagents

Reagents used shall be of analytical grade purity.

5.1 Demineralized water or 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 to **0,001 mol/l CaCl_2** .

5.2 Calcium chloride ($\text{CaCl}_2 \cdot 2 \text{H}_2\text{O}$), analytical grade.

5.3 Sodium azide (NaN_3), analytical grade.

5.4 Nitric acid (pro-analysis) (HNO_3), 0,1 mol/l to 5 mol/l, and 0,1 mol/l rinsing solution.

5.5 Sodium hydroxide (NaOH) or **potassium hydroxide** (KOH), 0,1 mol/l to 5 mol/l.

NOTE The use of KOH instead of NaOH may enhance the leaching of certain cations such as NH_4^+ and Cs^+ by cation exchange.

5.6 Organic solvent (acetone, analytical grade) for rinsing and cleaning

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, and it should be assured that previously used bottles have no background level of analyte.

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

NOTE 2 To prevent organic compounds from degradation by light use a dark room, dark colored glassware or place a layer of aluminium-foil around the leaching equipment.

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

The volume of 1 l is selected in combination with the mass, m_D , of 60 g, in order to minimize headspace in the bottle. 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 density, deviation from this requirement can be necessary while still ensuring minimize headspace. This deviation should be reported.

NOTE 3 Glass of high quality is considered adequate for both metals and organic substances, particularly since the pH range usually covered in soil testing does not reach the conditions ($\text{pH} > 12$ and $\text{pH} < 3$) where the glass itself may be partially dissolved. For ecotoxicity testing, eluates with both inorganic and organic substances are needed, which emphasizes the need to generate integrated eluates.

NOTE 4 Heat treatment of used glassware at 550 °C can be used to remove traces of analytes. However, this treatment has been shown to increase adsorption of organic substances from the air.

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 may be used, provided that they can be shown to provide equivalent results. These agitation devices are specified because excessive abrasion leading to significant particle size reduction should be avoided.

6.3 Filtration apparatus, either a vacuum filtration device (between 2,5 kPa and 4,0 kPa) or a high-pressure filtration apparatus ($< 0,5 \text{ MPa}$). Rinsing is compulsory. When semi-volatile substances are to be analysed, vacuum filtration shall not be used.

6.4 0,45 μm membrane filters, prerinsed or similarly cleaned (e.g. rinsed with $0,1 \text{ mol/l HNO}_3$ (5.4) and water (5.1) (only for analysis of inorganic substances).

The filters shall be chosen so as not to adsorb (or release) substances of interest.

NOTE This can be tested in preliminary experiments.

6.5 Glass fibre filters, with a degree of separation of $0,7 \mu\text{m}$.

The filters shall be chosen so as not to adsorb (or release) substances of interest.

NOTE This can be tested in preliminary experiments.

6.6 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 substances of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment or plasticisers from plastic sieves.

6.7 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.

NOTE Potential sorption of hydrophobic organic substances to the centrifuge tubes can be tested in preliminary experiments.

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.

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

6.9 Device for measuring electrical conductivity.

6.10 pH meter, in accordance with ISO 10523.

6.11 Redox potential meter (optional).

6.12 Balance, with an accuracy of at least $0,1 \text{ g}$.

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

6.14 Turbidity meter, as specified in ISO 7027-1.

6.15 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 substances of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment.

7 Sample pretreatment

7.1 Preparation of laboratory sample and specification of particle size

A representative laboratory sample of at least 2 kg (dry matter) is obtained (e.g. as described in ISO 18400 series and ISO 23909) and shall be stored in closed packages and at low temperatures (4 °C), in order to avoid unwanted changes in the material (see e.g. ISO 18400-105).

The test shall be carried out on soil or soil-like material sieved to < 2 mm (e.g. as described in ISO 11464). Oversized material of natural origin in the sample shall be separated and discarded. The type and amount of all discarded material shall be reported. If oversized material of anthropogenic origin is present and assumed to contain substances of interest, this part can be subject to alternative sample preparation or testing.

If the laboratory sample cannot be homogenised or sieved because of its water content, it is allowed in this case only to dry the laboratory sample (e.g. as described in ISO 11464). The drying temperature shall not exceed 30 °C.

NOTE 1 Sieving and drying at more than 30 °C, as well as crushing, can lead to a loss of semi-volatile substances (inorganic and organic) and can alter the leaching characteristics).

NOTE 2 Due to sieving, contamination of the sample can occur to an extent that affects the leaching of some substances of concern, e.g. chromium, nickel and molybdenum from stainless steel equipment or plasticisers from plastic sieves.

7.2 Preparation of test sample

Use a sample splitter (6.13) or apply coning and quartering to split the laboratory sample and obtain a test sample. The size of test sample required depends on the volume of eluate needed for the specific purpose and the subsequent chemical analysis and/or ecotoxicological tests to be carried out on the eluate.

NOTE 1 If needed for chemical analysis or ecotoxicological testing, larger volumes of eluate can be obtained by combining eluates from replicate tests after centrifugation (or filtration). Alternatively, larger volumes of eluate may also be produced in a single test, provided that the ratios in terms of L/S and minimum headspace are maintained.

NOTE 2 The required amount of the test sample is dependent on the particle size distribution of the soil to be analysed (see ISO 23909). The specified sample amount will generally be adequate. In specific cases, a smaller sample amount can be accepted, for instance, if for specific reasons less material is available, provided that the test can be carried out as specified in 7.2 to 7.4.

7.3 Determination of the dry matter content and of water content

The whole test sample, complying with the size criterion in 7.2, shall not be further dried. The moisture content of the test sample shall be determined on a separate test portion at (105 ± 5) °C. If the soil sample is air-dried prior to testing, the dry matter content w_{dm} of the air-dried sample shall be determined as well. This shall be taken into account when adjusting the L/S. The dry mass of the sample shall be determined at (105 ± 5) °C, in accordance with ISO 11465, and the dry matter content calculated with [Formula \(1\)](#):

$$w_{dm} = 100 \times m_D / m_W \quad (1)$$

where

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

m_D is the mass of the dried sample, expressed in kilograms (kg);

m_W is the mass of undried sample, expressed in kilograms (kg).