TECHNICAL SPECIFICATION

ISO/TS 21268-3

First edition
2007-07-15

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

Part 3: Up-flow percolation test

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

Partie 3: Essai de percolation à écoulement ascendant

Reference number
ISO/TS 21268-3:2007(E)

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Published in Switzerland

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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 normative 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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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 21268-3 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 characterise and assess the constituents which can be released from materials. The release of soluble constituents 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.

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

Tests to characterise the behaviour of materials can generally be divided into three categories (EN 12920; EN/TS 14405) and are addressed in ISO 18772 [13]. The relationships between these tests are summarised below:

a) "Basic characterisation" 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 tests.

b) "Compliance" tests are used to determine whether the material complies with a specific behaviour or with specific reference values. The tests focus on key variables and leaching behaviour previously identified by basic characterisation 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 b): basic characterisation tests.

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

This Technical Specification was elaborated on the basis of CEN/TS 14405.

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

Part 3: Up-flow percolation test

1 Scope

This part of ISO/TS 21268 specifies a test, which is aimed at determining the leaching behaviour of inorganic and organic constituents from a soil and soil material. The method is a once-through percolation test with water (0,001 mol/l $CaCl_2$) under standardized conditions of flow rate. The material is leached under dynamic hydraulic conditions. The eluates obtained can be used to determine the ecological properties of the soil with respect to micro-organisms, flora and fauna. The test results enable the distinction between different release patterns, for instance wash-out and release under the influence of interaction with the matrix, when approaching local equilibrium between material and leachant.

This test method produces eluates, which can subsequently be characterised by physical, chemical and ecotoxicological methods in accordance with existing standard methods. The results of eluate analysis are presented as a function of the liquid/solid ratio. The test is not suitable for species that are volatile under ambient conditions.

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 optimise 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 also meant to include genotoxicological testing.

The application of this test method alone is not sufficient for the determination of the leaching behaviour of a material under specified conditions different to those from the test procedure, since this generally requires the application of several test methods, behavioural modelling and model validation. This part of ISO/TS 21268 does not address issues related to health and safety. It only determines the leaching properties as outlined in Clause 4.

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 7027:1999, *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 10381-6, *Soil quality — Sampling — Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in the laboratory*

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 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

NOTE For the purposes of this part of ISO/TS 21268, the leachant is water as specified in 5.1.

3.3
eluate
solution recovered from a leaching test

3.4
liquid to solid ratio
L/S
the 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 L/S is expressed in l/kg.

3.5
laboratory sample
sample or subsample(s) sent to or received by the laboratory

3.6
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

3.7
test portion
quantity of material of appropriate size for measurement of the concentration or other properties of interest, taken from the test sample

NOTE 1 The test portion can be taken from the laboratory sample directly if no pre-treatment of sample is required, but usually it is taken from the test sample.

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

3.8
granular material
solid material, not being monolithic

NOTE It is not a gas, a liquid or a sludge.

3.9
dry matter content
w_{dm}
ratio expressed in percent between the mass of the dry residue and the corresponding raw mass

NOTE It is determined in accordance with ISO 11465.

3.10
soil materials
excavated soil, dredged materials, manufactured soils, treated soils and fill materials

[ISO 15176:2002, definition 3.1.4]

4 Principle

This part of ISO/TS 21268 describes a method to determine the release of constituents from soil and soil material, packed in a column into a leachant percolating through it. A continuous vertical up-flow is used, which allows a column test under water-saturated conditions. The test conditions, including the flow rate of the leachant, are chosen such that it can be concluded from the results, which components are rapidly being washed out and which components are released under the influence of interaction with the matrix.

The test portion of the material with a specified particle size is packed in a column in a standardised manner. Pre-equilibration is applied to reach local equilibrium at the start. The column size is related to the amount of eluate needed for subsequent analysis and testing. The leachant is demineralised water with 0,001 M $CaCl_2$. The leachant is percolated in up-flow through the column at a specified flow rate up to a fixed L/S ratio. The eluate is collected in several separate fractions.

After the test, the leaching conditions, in terms of pH, electrical conductivity or DOC and, optionally, turbidity or redox potential dictated by the material, shall be recorded.

NOTE These parameters often control the leaching behaviour of soil materials and are therefore important for evaluation of the test results. Dissolved organic carbon (DOC), in particular, is crucial in soil and soil materials for many inorganic and organic constituents.

The properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates and/or the eluate may be subjected to subsequent ecotoxicological testing.

The results of the test are expressed as a function of liquid/solid ratio, in terms of both concentration (mg of the constituents released per litre eluate) and release [mg of the constituents released cumulatively per kg of material (dry mass)] of the constituents.

The procedure described in this part of ISO/TS 21268 is based on the more stringent test requirements for determining the release of organic constituents and/or for subsequent ecotoxicological testing. If only the release of inorganic constituents is to be measured, less stringent requirements may be adapted for some steps of the procedure.

5 Reagents and materials

5.1 Demineralised water or **deionised water** or **water of equivalent purity** ($5 < pH < 7{,}5$) with a conductivity < 0,5 mS/m in accordance with grade 3 specified in ISO 3696, made to 0,001 M $CaCl_2$.

5.2 Rinsing solutions: nitric acid (pro analyse) 0,1 mol/l and an organic solvent (acetone, pro analyse).

6 Apparatus

The materials and equipment specified in 6.2 to 6.13 shall be checked before use for proper operation and absence of interfering substances, which can affect the result of the test.

The equipment specified under 6.5, 6.6, 6.13 and 6.14 shall also be calibrated.

6.1 Column made of glass or plastics with an internal diameter of 5 cm or 10 cm and a filling height of about (30 ± 5) cm, fitted with filters (6.3) in the bottom and top sections made of appropriate materials ensuring minimum interference with the contaminants of interest [e.g. polychlorotrifluoroethylene (PCTFE)]. In the top and bottom of the column, a filter plate or a thin layer of fine-grained non-reactive material (e.g. fine quartz sand) is applied to ensure proper water flow over the width of the column and as a support for the pre-filter.

NOTE 1 A drawing of the column and accompanying equipment is given in Figure A.1.

NOTE 2 Glass of high quality is usually considered adequate for both metal and organic contaminants, particularly since the pH range usually covered in soil testing does not reach the conditions (pH > 10 and pH < 3) where glass itself is attacked. For ecotoxicity testing, eluates with both metal and organic contaminants are needed, which emphasises the need to generate integrated eluates.

NOTE 3 When only organic constituents are analysed, stainless steel column and fittings can be applied.

6.2 Filters for in-line or off-line filtration of the eluates; they shall not adsorb the compounds of interest. This shall be tested in preliminary experiments.

NOTE For organic compounds, glass-fibre filters without organic glue are suitable. If only inorganic contaminants are analysed, alternative filter materials can be selected (e.g. cellulose acetate, PTFE).

6.3 Pre-filters for the column with a pore size of 1,5 µm to 20 µm. The filters shall be glass-fibre filters without organic glue.

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

6.4 Peristaltic pump with an adjustable capacity of between 0 ml/h and 60 ml/h.

NOTE Additional pumps can be used.

6.5 Analytical balance with an accuracy of at least 0,1 g.

6.6 pH meter with an accuracy of at least ± 0,05 pH units.

6.7 Electrical conductivity meter with an accuracy of at least 0,1 mS/m.

6.8 Tubing material (made of ethylene-tetrafluoroethylene, ETFE) adapted to the analysis to be performed (see ISO 5667-3).

NOTE When only organic constituents are analysed, stainless steel tubing can be used. When only inorganic constituents are analysed, polytetrafluoroethylene (PTFE) or similar tubing materials can be used.

6.9 High-quality **glass bottles** with an appropriate volume, and with a screw cap with a PTFE inlay, for eluate collection and preservation of eluate samples (in accordance with ISO 5667-3).

NOTE If only inorganic contaminants are analysed, alternative bottle materials can be selected (e.g. HDPE, PTFE).

6.10 Crushing equipment: a jaw crusher or a cutting device.

6.11 Sieving equipment with sieves of 4 mm nominal screen size.

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

6.13 Redox potential meter (optional).

6.14 Turbidity meter as specified in ISO 7027.

6.15 Centrifuge operating at 20 000*g* to 30 000*g* using centrifuge tubes of fluorinated ethylene propylene (FEP) 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.

7 Sample pre-treatment

7.1 Sample preparation

Obtain a laboratory sample of at least 10 kg, in case a wide column (10 cm) is used. Use a sample splitter (6.12) or apply coning and quartering to split the sample.

The laboratory sample shall be stored in closed packages and at low temperatures (4 °C), in order to prevent unwanted changes in the material (see ISO 18512).

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 10381-6, in order to obtain representative laboratory samples.

NOTE 1 Depending on the maximum particle size, the splitting can require reduction of the coarser particles to comply with the rules of sampling.

NOTE 2 The size of the laboratory sample is dependent on the particle size distribution of the soil to be analysed (ISO 10381-1 to ISO 10381-6). The prescribed sample size will generally be adequate.

NOTE 3 If a 10 kg laboratory sample is not available, the test may be carried out with less material.

NOTE 4 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).

NOTE 5 Alternatively, larger volumes of eluate may also be produced in a single test using a larger column, provided that the proportions in terms of L/S are maintained.

Any deviation(s) to accommodate sample size or volume requirements shall be recorded in the test report.