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)

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

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** ($5 < \text{pH} < 7{,}5$) 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 ($\text{pH} > 10$ and $\text{pH} < 3$) 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.

7.2 Particle size reduction

The test shall be carried out preferably on material as received. However, the test portion to be prepared shall have a grain size of less than or equal to 4 mm at least 95 % (mass fraction). Oversized material in the sample shall be separated by sieving over a 4 mm sieve. The mass and the nature of the oversized material shall be recorded. If oversized material is not of natural origin or 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. If the laboratory sample cannot be crushed or sieved because of its water content, it is allowed, only in this case, to dry the laboratory sample. The drying temperature shall not exceed 25 °C. For the processing of soil samples, refer to ISO 10381-6.

NOTE 1 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 form stainless steel equipment or plasticisers from plastic sieves.

NOTE 2 Sieving and drying at 25 °C can lead to a loss of semi-volatile components (inorganic and organic). In this case, particle size reduction and drying is best avoided.

7.3 Test portion

Take, from the test sample, a test portion with a volume of approximately 0,6 l, if the column has a diameter of 5 cm, and of 2,4 l, if the column has a diameter of 10 cm. Use a sample splitter (6.12) or apply coning and quartering to split the test sample.

7.4 Determination of dry matter content

The dry matter content of the sample shall be known and taken into account when calculating the L/S ratio.

Determine the dry matter content (w_{dm}) on a separate test portion. The dry mass shall be determined at (105 ± 5) °C in accordance with ISO 11465. The dry matter content is calculated in Equation (1):

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

where

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

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

m_{W} is the mass of the undried sample (kg).

If, for reasons expressed in 7.2 or 7.3, the material was (partly) dried before particle size reduction and/or sample splitting, the overall mass loss shall be taken into account.

8 Procedure

8.1 Temperature

The percolation test shall be carried out at a temperature of (20 ± 5) °C.

NOTE A constant temperature of 20 °C in the test can be achieved by either controlling the temperature of the laboratory, or controlling the temperature of the leachant and insulating the column and accompanying equipment.

For material that is very sensitive to biological degradation, performance of the test at reduced temperature (e.g. 4 °C) and preventing direct exposure to light will limit biological activity significantly. If the test is modified in this way, this deviation shall be reported in the test report.

8.2 Preparation

Rinse the column with top and bottom sections (6.1), filters (6.2) and bottles (6.9) with nitric acid and/or an organic solvent (5.2) and water (5.1) respectively. Weigh the dry column, including the top and bottom sections, filters and filter plates or layers of fine-grained material, to an accuracy of 1 g.

NOTE Alternatively, a heat treatment can be applied to clean temperature-resistant equipment of sorbed organic contaminants.

8.3 Packing of the column

Fit the bottom section, equipped with a filter plate or a layer of fine-grained chemically inert material (e.g. fine quartz sand) of approximately 1 cm and a pre-filter (6.3) to the column. Fill the column with the test portion, up to a bed height of (30 ± 5) cm in at least five consecutive layers.

- Introduce each layer into the column in three sub-layers and level each sub-layer separately.
- Pack each layer, using as a rammer a weight of 125 g in the case of a column with a diameter of 5 cm, and of 500 g in the case of a column with a diameter of 10 cm. Drop the weight three times onto each layer, letting it fall down 20 cm along a rod used as a guide. Fix this rod to the centre of a disk, which is placed on the layer to be packed. Cover the whole surface of the column with the disk (as is shown in Figure A.1).
- For the last layer, check the remaining height and adjust the necessary mass in order to get (30 ± 5) cm.

NOTE 1 In order to determine the proper mass for each layer, a preliminary test may be carried out. In that case, put a 7 cm to 8 cm layer in the column, pack it and calculate the mass necessary to obtain a layer of approximately 6 cm.

NOTE 2 If the column is not high enough to work according to the above-mentioned packing procedure, a heightening device may be required.

NOTE 3 It is difficult to pack some specific materials in the column in an appropriate way. Annex B gives guidelines on how to handle that specific case.

Fit the top section of the column, equipped with a filter plate and a pre-filter (6.3), to prevent entrainment of fine particles with the eluate. The top section and pre-filter shall be fitted such that the liquid flow cannot bypass the filter, and such that no open space (dead volume) is left above the material.

Care should be taken in positioning the pre-filter in place; the filter may tear.

Weigh the column thus filled to an accuracy of 1 g. Determine the dry mass (m_D), in kilograms, of the test portion in the column, in accordance with Equation (2):

$$m_\mathrm{D} = m_\mathrm{W} \cdot w_\mathrm{dm}/100 \qquad (2)$$

where

m_W is the mass of the (moist) test portion in the column (kg).

w_dm is the dry matter content (%);

Fit the outlet-hose to the top section of the column and to the in-line filter (6.2), when an in-line filter is used.

8.4 Start of the test

Saturate the column with water (5.1) either by using the pump (6.4) or by hydrostatic pressure.

NOTE 1 See Annex A for a description of the methods of saturation.

Stop the pump, or take away the hydrostatic pressure, when the material in the column is completely saturated, but the outlet hose remains empty. Leave the saturated material for a period of at least two days, in order to equilibrate the system.

NOTE 2 To facilitate equilibration and the procedure of checking the equilibrium conditions, recirculation of the eluate may be applied (see Annex A).

After the equilibration period, start the pump (again) and set the flow rate such that the linear velocity is approximately (15 ± 2) cm/day through the empty column.

Calculate the flow rate in accordance with Equation (3):

$$q = v_{\mathrm{L}} \times \pi \times d^2 \times 0{,}010\ 4 \tag{3}$$

where

q is the leachant flow rate (ml/h);

v_{L} is the linear velocity of the leachant through the empty column (cm/day);

d is the diameter of the column (cm).

NOTE 3 A linear velocity of 15 cm/day corresponds to a flow rate of 12 ml/h for a column with a diameter of 5 cm, and for a column with a diameter of 10 cm it is equivalent to a flow rate of 48 ml/h.

Connect the outlet hose (6.8) to an eluate collection bottle of appropriate size (6.9). Start the pump and change the collection bottle after a quantity of (0,1 ± 0,02) × the mass of the test portion (m_{W}) of leachant has passed through.

Transfer the supernatant to centrifuge tubes (6.15).

There are two options for solid-liquid separation.

1) Centrifuge the eluate for 30 min at 20 000g to 30 000g using a high-speed centrifuge (6.15).
2) Centrifuge the eluate for 5 h at 2 000g to 2 500g in glass bottles using a lower-speed centrifuge (6.15).

Cooling shall be applied to maintain the temperature at (20 ± 5) °C (see 8.1).

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. The deceleration time shall not exceed 20 min.

Filter the eluate fraction off-line over a 0,45 µm membrane filter (6.2), if no in-line filter was used.

Measure the pH, electrical conductivity and DOC of this eluate portion. Measurement of the turbidity and redox potential are highly recommended.

When an in-line filter is used, this filter shall be regularly checked concerning clogging due to the small filter diameter. If clogging occurs, the filter shall be replaced.

A water lock will be needed on the lid, to avoid back-pressure in the column when the bottle with lid is gas tight.

Keeping the collection bottles under inert atmosphere may be necessary when investigating reducing soil materials, to prevent the occurrence of oxidation reactions.

If only inorganic constituents are measured, the eluate can be filtered using the appropriate membrane filters (6.2) 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.

NOTE 5 For inorganic constituents, it is often preferable to pre-centrifuge the eluate at 2 000g to 3 000g for 20 min using glass bottles with a screw cap and polytetrafluoroethylene inlay (or, if possible, using the leaching bottle directly) prior to filtration.

Under certain circumstances, particularly for alkaline eluates, it is recommended to measure the pH and redox potential of the raw eluate prior to filtration or centrifugation, since these operations may change the pH and redox of the eluate.

Analysis of DOC in the eluate is highly recommended, as this property is relevant both for the release of metals as well as for organic substances.

8.5 Collection of additional eluate fractions

Check the flow rate of the leachant and possible clogging of the in-line filter (if used) as often as needed, but at least three times per week, and adjust to the original linear velocity, in the range of (15 ± 2) cm/day. If clogging occurs, the filter shall be replaced.

Replace the collection bottle with a new one as soon as a quantity of water (5.1) in accordance with Table 1 has passed through. These are fractions 2 to 7. Take care that both criteria (concerning the volume of the actual eluate fraction as well as the cumulative L/S ratio) shall be fulfilled.

Centrifuge or filter each eluate fraction off-line as specified in 8.4. Measure the pH, electrical conductivity, and DOC of each eluate portion. Measurement of the redox potential and turbidity are highly recommended.

At each eluate collection moment, measure the time and volume of the eluate fraction and calculate the L/S-ratio and the average linear velocity of the leachant over the collection period of that fraction. Also measure the actual linear velocity. Report all these values.

Table 1 — Table for collection of eluate fractions

Fraction number	**Fraction volume** (l) (= L/S ratio times dry mass)[a]	**Cumulative L/S ratio** (l/kg)
1	$(0,1 \pm 0,02) \times m_D$	0,1 ± 0,02
2	$(0,1 \pm 0,02) \times m_D$	0,2 ± 0,04
3	$(0,3 \pm 0,05) \times m_D$	0,5 ± 0,08
4	$(0,5 \pm 0,01) \times m_D$	1,0 ± 0,15
5	$(1,0 \pm 0,2) \times m_D$	2,0 ± 0,3
6	$(3,0 \pm 0,2) \times m_D$	5,0 ± 0,4
7	$(5,0 \pm 0,2) \times m_D$	10,0 ± 0,1

[a] In the case of high salt loads (electrical conductivity > 7 500 mS/m), the density of the eluate in the first few fractions is significantly more than 1 g/ml. In that case, the volume of these fractions of eluate shall be measured and used for the calculations instead of the mass.

The test itself is finished when the L/S ratio of 10 l/kg dry matter is reached.

NOTE 1 For specific scenarios (for instance a landfill with top cover), it can be sufficient to know the leaching characteristics up to a pre-determined L/S ratio, for instance L/S = 2. In that case, the test can be stopped after the collection of the fifth eluate fraction.

NOTE 2 For ecotoxicological testing, the fractions 1 to 5 (or parts of these fractions) can be combined for receiving a cumulative L/S ratio of 2. If large amounts of leachate are needed for testing, a cumulative L/S ratio of 10 may be used.

NOTE 3 When no automated eluate collection apparatus is available, a collection scheme can be composed within the ranges that are allowed in the flow rate (15 ± 2) cm/d, and in fraction volume (see Table 1), that enables eluate collection within working hours.

NOTE 4 The execution time of the test can be calculated from Equation (4):

$$t = \left(\mathrm{L/S} \times m_\mathrm{D} \times 1\,000\right) / \left(24 \times q\right) \tag{4}$$

where

t is the execution time of the test, expressed in days (d);

L/S is the final liquid to solid ratio, expressed in litres per kilogram of dry matter (l/kg dry matter);

m_D is the dry mass of the test portion, expressed in kilograms (kg);

q is the leachant flow rate, expressed in millilitres per hour (ml/h).

8.6 Further preparation of the eluates for analysis

If necessary, divide the eluate into an appropriate number of sub-samples for different chemical analyses and store them in accordance with the requirements in ISO 5667-3. If the eluates are to be used for bio-assays, they shall be processed as soon as possible but at least within seven days of storage at a minimum of 4 °C.

8.7 Blank test

In order to check, as far as possible, how the whole procedure is performed, blank tests shall be carried out on a regular basis. A volume of leachant is therefore submitted to the whole procedure (except the sample pre-treatment). For this purpose, the pump shall be started and the empty column, completed with top and bottom sections and with tubing, shall be filled with leachant. After two days, disconnect the pump and the column, empty the column via the bottom section and collect the eluate. Preserve and analyse the blank eluate in accordance with 8.6.

The eluate of this blank test shall fulfil the following minimum requirements: in the eluate of the blank test the concentration of each considered substance shall be less than 10 % of the concentration determined in the first eluate of the tested material. If the blank measurement is below the detection limit and the detection limit is the same or less than that for the eluates, the requirement is also fulfilled. If the requirement is not fulfilled, it is necessary to reduce the contamination.

9 Calculations

Calculate, for each component, the quantities released in all eluate fractions with Equation (5):

$$w_i = \left(V_i \times \phi_i\right) / m_\mathrm{D} \qquad (5)$$

where

i is the index of the eluate fraction (1, 2, …, 7);

w_i is the released quantity of a component per quantity of sample for analysis in the eluate fraction i, expressed in milligrams per kilogram of dry matter (mg/kg dry matter);

V_i is the volume of the eluate fraction i, expressed in litres (l);

ϕ_i is the concentration of the component concerned in the eluate fraction i (mg/l);

m_D is the dry mass of the test portion in the column, expressed in kilograms (kg).

The concentration ϕ_i referred to in Equation (5), is the concentration originally present in the eluate. The measured value, determined in accordance with 8.6, shall be corrected if the eluate fraction has been diluted and/or if the quantity of preservation fluid added in 8.6 was more than 1 ml per 100 ml of eluate.

Where the concentration of a component in one or more eluate fractions is below the limit of detection, two calculations shall be carried out for this component in these fractions. The upper limit of w_i is calculated by making ϕ_i equal to the limit of determination; the lower limit of U_i is calculated by making ϕ_i equal to 0.

For each component, the cumulatively released quantity (Σw_i) shall be calculated by accumulating the released quantities of the specific component, measured in the different eluate fractions. Where the concentration of a component in one or more eluate fractions is below the lower detection limit, for this component two calculations shall be carried out, to indicate both the upper limit and the lower limit of Σw_i.

10 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) centrifugation speed/force, time and type of vessels used, temperature readings;

c) the test results (e.g. chemical analysis);

d) 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.

11 Test performance

The performance of the test regarding repeatability and reproducibility is dependent on the tested material and also on the testing conditions. Validation needs to be carried out in accordance with ISO 5725-1, -2 and -5.

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

Annex A
(informative)

Suggestions for packing the column, water saturation and establishment of equilibrium conditions

A.1 General

This annex contains suggestions on how to fill and pack the column in the case of specific materials. It also gives a more detailed description of the two methods to conduct water saturation. Furthermore, it gives guidelines on how to equilibrate the column and how to check equilibrium conditions before starting the dynamic process in the column, and after the performance of the test.

A.2 Column filling and packing

In the case of specific materials, guidelines for filling the column and packing the material are the following:

- Non-powdery materials can generally be packed as they are (moist or dry). However, powdery, dry materials should be humidified either at an arbitrary and imposed ratio or referring to the Proctor optimum humidity, if known. The actual water content should be known, in order to be able to determine the dry mass of the test portion in the column (see 8.3).

- Materials can be too wet to pack well in the column. It is usually possible to air-dry the test sample or the test portion. The drying temperature should not exceed 25 °C. Drying may lead to oxidation and/or carbonation. If the material is fresh and is tested as a non-oxidized/non-carbonated material, the drying should be conducted in an inert atmosphere.

- In other cases, it can be possible to pack the (wet) material, but the material may settle even more after the start of the test, causing the formation of headspace. If a system with a piston is used, the piston should be lowered accordingly.

- Some materials can cause problems later, even if they are packed well. These are materials with hydraulic binding properties. Hardening reactions may lead to expansion, leading to a very low permeability, or even to cracking of the column. This means that the material is likely to behave as a monolith in scenario and should be tested with the appropriate standard. However, if it is the intention to study the behaviour of the material in percolating conditions, a system with a piston that can be moved upward should be used. Another solution may be, in some cases, to compact less. Both solutions, however, lead to a worse repeatability.

A.3 Water saturation

In 8.4, two methods are mentioned to saturate the packed column with leachant a): by using the pump or b) by using initial hydrostatic pressure.

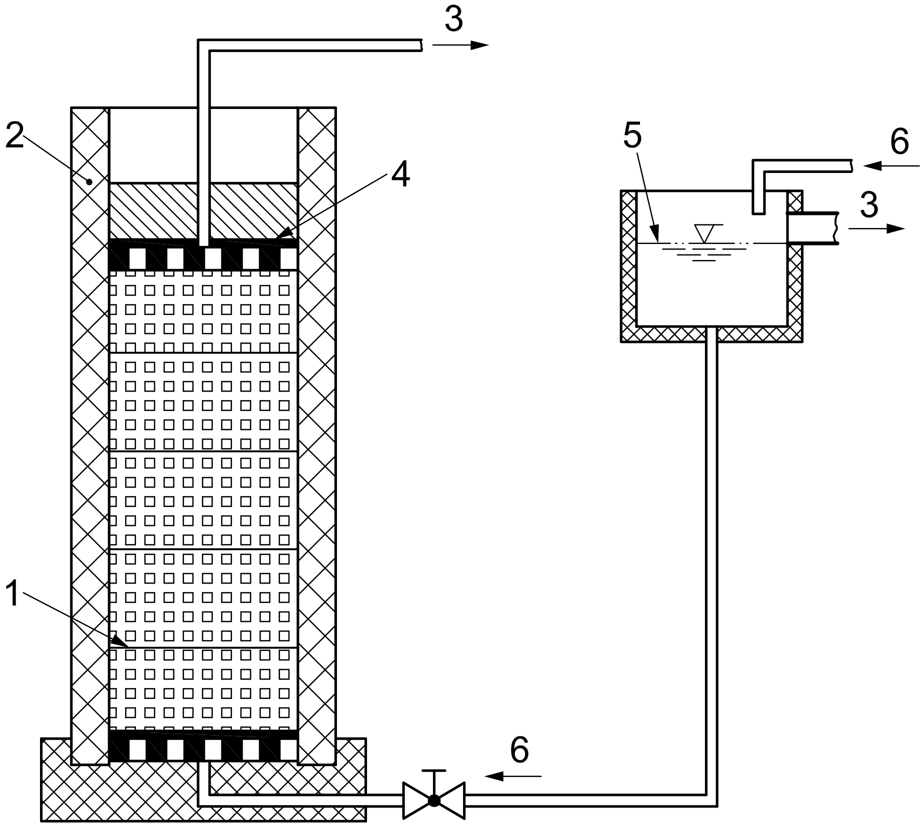
a) Saturating the column by using the pump

Connect the pump (6.4) to the bottom section of the column and pass demineralized water (5.1) through the column from bottom to top (with a linear velocity of the leachant through the empty column of approximately 15 cm/d). Stop the pump when the material in the column is all saturated, but the outlet hose is still empty.

b) Saturating the column by using initial hydrostatic pressure

To avoid the need to watch over the column in order to prevent overflowing, water saturation can also be carried out under an initial hydrostatic pressure of 30 cm (as shown in Figure A.1).

The hydrostatic pressure should be stopped when the material in the column is all saturated, but the outlet hose is still empty. It should be assured that the water level in the inlet is at least as high as the layer of the inert material.



Key

1	layer of compacted waste	4	layer of inert material
2	column	5	water level
3	water outflow	6	water inlet

Figure A.1 — Saturation of the column by initial hydrostatic pressure

Annex B
(informative)

Justification of the choices made in developing the test procedure

B.1 Introduction

During the development of this test procedure, it was necessary to make some choices regarding test conditions. Fixing some conditions means that the test will not provide a general simulation for all kinds of scenarios. One of the original objectives was to ensure local equilibrium conditions[1] between the soil material in the column and the percolating leachant throughout the duration of the test. The existence of local equilibrium would generally enhance the robustness of the test. Another objective was to develop a relatively simple, practical test of moderate duration, operating under fixed conditions and capable of producing results with good reproducibility. It has not been possible to optimise or fulfil all of the objectives simultaneously. The prescribed test conditions are thus the results of several compromises. In view of the desire for a short test duration, it has, for instance, been chosen not to impose a specific requirement for local equilibrium nor the corresponding verification.

In the following, the choices of some of the test conditions are briefly discussed:

- particle size/particle size distribution;
- column dimensions;
- flow mode (up-flow/down-flow);
- flow rate of the leachant;
- nature of the leachant;
- L/S ratio and eluate fractions collected/duration of the test;
- temperature.

For filling and packing of the column, see Annex A.

B.2 Particle size distribution

If possible, the particle size distribution of the material to be tested should remain unchanged. It is generally preferable not to reduce the particle size if it can be avoided. The crushing of a granular material may alter its leaching properties by opening new surfaces and disturb surface layers that may have formed as a result of ageing processes. The particle size/particle-size distribution has an influence both on the potential representativity of the test portion in the column and on the time required to approach the initial equilibrium between the dissolved and the solid phases for various components. Both properties improve with decreasing particle size, i.e. the representativity increases and the pathway for diffusion in the solid phase shortens, thus

---

[1] Local equilibrium is referred to as a condition of chemical equilibrium between the solid phase and the leachant at each cross-section of the column during the actual percolation. This condition is generally not the same along the length of the column. In any one section, it changes with progressing percolation. This condition is not verified during the test. The achievement of local equilibrium is generally favoured by decreasing leachant velocity, decreasing particle size and increasing temperature.

decreasing the time needed to approach equilibrium conditions. As a rule of thumb, the largest particle size should be at least 10 times smaller than the diameter of the column (see 8.5, Table 1 and Table B.1).

B.3 Column dimensions

For good reproducibility of the leaching test itself and proper interpretation of the results, the flow pattern within the column should resemble plug flow as closely as possible. Based on experience, this is assumed to be the case for linear velocities (through the empty column) in the range of 0,5 cm/d to 15 cm/d, if the length of the column is at least three to four times the diameter. From the point of view of representativity of the test portion placed in the column and of securing relatively large fractions of eluate for chemical analysis, a large column would be preferable. However, the larger (longer) the column, the longer the time it would take to reach a certain L/S value for the same linear velocity. Experimental data obtained so far do not indicate very significant differences as a function of flow rate within the above-mentioned range. However, the number of materials for which this information has been verified is very limited and can thus not be generalised.

It has therefore been decided to use two options for column size: one with an inner diameter of 5 cm and one with an inner diameter of 10 cm, both with a filling height of (30 ± 5) cm.

Table B.1 — Column dimensions and eluate volumes

	Eluate volume (ml)	
Column diameter (height 30 cm)	5 cm	10 cm
Sample weight (kg) [a]	1,00	4,0
Liquid to solid ratio (l/kg)		
0,1	100	400
0,2	100	400
0,5	300	1 200
1	500	2 000
2	1 000	4 000
5	3 000	12 000
10	5 000	20 000

[a] For a density of 1 700 kg/m³.

B.4 Leachant flow direction

This part of ISO/TS 21268 is intended to describe the leaching properties of granular waste materials being percolated by a leachant under saturated conditions. This is best achieved by passing the leachant through the column in up-flow mode. Application of the up-flow mode further reduces the risk of channelling and preferential flow, provided the flow rates are not excessive.

B.5 Leachant flow rate

The actual flow rate of the leachant in the test (expressed in ml/h) is based on and calculated from an apparent linear velocity, expressed in terms of cm/d (cm/24 h), through the empty column. This is convenient since the equilibrium/non-equilibrium conditions in the soil material/leachant system is related to the linear velocity, and it is independent of the pore volume of the packed column (the actual pore velocity will be considerably higher than the open column velocity). The open column linear velocity is expressed in the same way as, and is comparable to, the rate of infiltration of precipitation into a soil.

The major advantages of a low linear velocity of the leachant are that it is more likely to ensure that the local equilibrium conditions are fulfilled, and that it comes closer to the actual flow conditions occurring under field conditions. It should be noted, however, that the laboratory procedure is not aimed at the same linear velocity as in field conditions, as this might lead to very long test duration. This is also the major disadvantage of low flow rates, particularly when the test is run to an L/S value of 10 l/kg. Conversely, the major advantage of a high leachant flow rate is that it limits the duration of the test.

It should be noted that, for a given soil material, the influences of particle size and flow rate on the achievement of local equilibrium are interrelated. Small particle sizes and low flow rates favour equilibrium, and vice versa.

For this test, a linear leachant velocity of 15 cm/d has been fixed. This enables the test to be carried out to a final L/S = 10 l/kg in approximately 30 d, and to reach L/S = 2 l/kg within approximately one week. Test results indicate that the local equilibrium condition appears to be fulfilled for several components and several materials, but not for all. For the sake of reproducibility, it is therefore important to maintain a relatively constant and precise linear velocity (15,0 ± 2) cm/d when applying this flow rate, compared to the times when very low flow rates are observed in field conditions.

Some (fine-grained) soil materials may have or develop low hydraulic conductivities, which may limit the leachant flow rate that can be obtained without the application of excessive pressure. (Hydraulic conductivity is defined as the rate of discharge of water under laminar flow conditions through a unit cross-sectional area of a porous medium under a unit hydraulic gradient and standard temperature conditions of 20 °C.) If it is deemed necessary or desirable to apply flow rates lower than 15 cm/d, this will be a deviation from the standard procedure and shall be mentioned in the test report.

B.6 Nature of the leachant

For most soil materials, the composition of the eluate at L/S values below 10 l/kg is controlled predominantly by the composition of the soil. It makes little difference whether the leachant consists of de-mineralised water or (real or artificial) rainwater. For the sake of simplicity, de-mineralised water has therefore been chosen as the leachant in this part of ISO/TS 21268. In view of the known mobilisation of dissolved organic matter (DOC) when using demineralised water, unlike conditions occurring in usual field conditions, a very low addition of $CaCl_2$ of 0,001 M is foreseen to reduce this artificial effect.

For specific purposes, it may be desirable to use leachants other than de-mineralised water. If this is done, it will be a deviation from the standard procedure and it shall be mentioned in the test report.

B.7 L/S ratio

The following eluate fractions are collected: L/S = 0,0 l/kg to 0,1 l/kg, 0,1 l/kg to 0,2 l/kg, 0,2 l/kg to 0,5 l/kg, 0,5 l/kg to 1,0 l/kg, 1,0 l/kg to 2,0 l/kg, 2,0 l/kg to 5,0 l/kg and 5,0 l/kg to 10 l/kg. The size of the fractions is seen to increase with increasing L/S, and the most detailed description is given of the first part of the leaching process. This is due to the fact that a large part of the easily leachable components are leached quite extensively within L/S = 0 l/kg to 1 l/kg or 0 l/kg to 2 l/kg. In addition, the L/S of many of the field leaching scenarios (landfills and utilisation applications), which could be modelled on the basis of percolation test results, will often require many years to reach a value of L/S = 1 l/kg or 2 l/kg.

To place the L/S ratios in perspective, two simple scenario calculations can be considered. For a 2 m thick layer of soil with a density of 1 t/m^3 through which water (e.g. infiltrating rainwater) is percolating at a rate of 200 mm/a, an L/S ratio of 2 l/kg and 10 l/kg will be attained in 20 years and 100 years, respectively. For a 20 m thick layer of soil with a similar density and percolation rate, L/S ratios of 2 l/kg and 10 l/kg will be attained after 200 years and 1 000 years, respectively.

It should be noted that the collection of the eluate as fractions is well suited to describe the amount of constituents leached at a given L/S. This procedure is, however, less suited to describe the actual eluate composition at various values of L/S, since the concentrations are measured as averages over increasing ranges of L/S. If a description of eluate composition at specific L/S values is desired, small eluate samples could be collected and analysed at those L/S values.

B.8 Temperature

Equilibrium reactions are temperature-dependent and care should therefore be taken to ensure a leachant/eluate temperature as constant as possible, and as close as possible to the prescribed value [20 ± 5] °C. The safest way to achieve this will be to ensure that the room temperature remains within the required range day and night during the entire test period. Alternatively, temperature control may be achieved in the column by applying a heated/cooled water jacket or a similar device.

The temperature (20 ± 5) °C has been chosen to represent common indoor conditions.

B.9 Beneficial use of the test results

Under certain conditions, the test results can be used to predict the total release of constituents from a material in a specific scenario, as a function of time, by means of modelling the hydraulic conditions of the scenario. The test also gives information on pore water concentrations in scenarios in which material and aqueous solutions are in equilibrium, both in the short term and in the long run (after wash-out of the salts).

Bibliography

[1] Dutch standard NEN 7343: 1995, *Leaching characteristics of solid earthy and stony building and materials; Leaching tests; Determination of the release of inorganic components from granular materials with the column test*

[2] *Solid Waste, granular inorganic material: Column Test,* Nordtest Method NT ENVIR 002, Nordtest, 1995

[3] Harmonisation of leaching/extraction tests, 1997. *Studies in Environmental Science*, Volume 70. Eds H.A. van der Sloot, L. Heasman, Ph. Quevauviller, Elsevier Science, Amsterdam, 292 pp.

[4] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[5] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[6] ISO 5725-5:1998, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

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

[8] Dijkstra, J.J., H.A. van der Sloot, R.N.J. Comans (2002): *Process identification and model development of contaminant transport in MSWI bottom ash.* Waste Management 2002, 22(5):pp. 531-541.

[9] K. Hund-Rinke, W. Kördel, D. Hennecke, A. Eisenträger and St. Heiden (2002). 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), pp. 43-50 (2002)

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

[11] CEN/TS 14405, *Characterisation of waste — Leaching behaviour tests — Up-flow percolation test (under specified conditions)*

[12] ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

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

ICS 13.080.05

Price based on 18 pages