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

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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 21268-3:2019 cancels and replaces (ISO/TS 21268-3:2007), 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;

— demineralized water has been added as possible leachant;

— the column diameter has been changed from "5 cm or 10 cm" to "5 cm to 10 cm";

— flow rate of 30 cm/d has been added as option based on robustness testing;

— 7.1 and 7.2 has been exchanged to read 7.1 "Particle size" and 7.2 "Sample preparation";

— 11.1 "General", 11.2 "Validation trials performed in Japan" and 12.3 "Validation results obtained in Germany (DIN 19528)" have been added;

— B.2 "Particle size distribution" has been deleted;

— a new informative Annex C "Calculation of centrifugation duration depending on centrifugation speed and rotor dimensions" has been added;

— references in Clause 2 and Bibliography has been updated.

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 (see description of influencing factors in Annex A).

Tests to characterise the behaviour of materials can generally be divided into three categories addressed in ISO 18772[2] and EN 12920[3]. The relationships between these tests are summarized below.

a) "Basic characterization" tests are used to obtain information on the short- and long-term leaching behaviour and characteristic properties of materials. Liquid/solid (L/S) ratios, leachant composition, factors controlling leachability, such as pH, redox potential, complexing capacity, role of dissolved organic carbon (DOC), ageing of material and physical parameters, are addressed in these 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 a): basic characterization tests.

This document was originally elaborated on the basis of CEN/TS 14405:2004[4]. Especially, modifications considering requirements on subsequent ecotoxicological testing and analysis of organic substances have been included. Validation results have been adopted from DIN 19528[5] and from Japanese validation studies[15,16].

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

Part 3:
Up-flow percolation test

1 Scope

This document specifies a test, which is aimed at determining the leaching behaviour of inorganic and organic substances from a soil and soil-like materials. The method is a once-through up-flow percolation test under standardized conditions of flow rate. The material is leached under dynamic hydraulic conditions. The document has been developed to measure the 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[6] and ISO 17616[7]. 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 characterized 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 (L/S) ratio. The test is not suitable for substances that are volatile under ambient conditions.

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 also meant to include genotoxicological testing.

NOTE 5 The test is generally not suitable for soils with hydraulic conductivities below 10^{-8} m/s (see also Annex B). It can be difficult to maintain the designated flow rate already in the range of saturated hydraulic conductivity between 10^{-7} m/s and 10^{-8} m/s.

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 document 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 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 5667-3:2018, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

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

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*

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 purposes of this document, the leachant is specified in 5.1.

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

3.7
test sample
sample, prepared from the *laboratory sample* (3.6), from which *test portions* (3.8) are removed for testing or analysis

3.8
test portion
quantity of material of appropriate size for measurement of the concentration or other properties of interest, taken from the *test sample* (3.7)

Note 1 to entry: 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 to entry: A unit or increment of proper homogeneity, size and fineness, needing no further preparation, can be a test portion.

3.9
soil-like material
excavated soil, dredged materials, manufactured soils, treated soils and fill materials

4 Principle

This document describes a method to determine the release of substances from soil and soil-like material, packed in a column with 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 the substances that are rapidly washed out and the substances that are released under the influence of interaction with the matrix can be deduced from the results. It is intended and assumed that conditions approach local equilibrium between the material and the leachant throughout the test.

The test portion, which originally or after suitable pre-treatment has a particle size less than or equal to 2 mm, is brought into contact with water containing a low concentration (0,001 mol/l) of calcium chloride or demineralised water (5.1) under defined conditions. The leachant is percolated in up-flow direction through the column at a specified flow rate up to a fixed L/S ratio. The eluate is collected in several separate fractions. The standard method is based on the assumption that equilibrium or near-equilibrium is achieved between the liquid and solid phases during the test period. The properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates, and the eluate may be subjected to subsequent ecotoxicological testing.

After the test, the leaching conditions, in terms of pH, electrical conductivity, and optionally, turbidity, dissolved organic carbon (DOC) or redox potential imposed by the material shall be recorded.

NOTE 1 These parameters often control the leaching behaviour of soil materials and are therefore important for evaluation of the test results. DOC, in particular, is crucial in soil and soil-like materials for many inorganic and organic substances.

NOTE 2 The leachant is 0,001 mol/l $CaCl_2$ to minimize the mobilisation of DOC caused by an ionic strength of the leachant which is too low.

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 L/S ratio, in terms of both concentration (mg of the substances released per litre eluate) and release [mg of the substances released cumulatively per kg of material (dry mass)] of the substances.

The procedure described in this document is based on the more stringent test requirements for determining the release of organic substances and/or for subsequent ecotoxicological testing. If only the release of inorganic substances is to be measured, simplifications 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 mol/l $CaCl_2$**.

5.2 Calcium chloride ($CaCl_2 \cdot 2\ H_2O$), analytical grade.

5.3 Sodium azide (NaN_3), analytical grade.

5.4 Nitric acid (HNO^3), analytical grade, made to 0.1 mol/l rinsing solution.

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

6 Apparatus

6.1 Column, made of glass with an internal diameter of 5 cm to 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 substances of interest. 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 1.

NOTE 2 Glass of high quality is usually considered adequate for both metal and organic substances, 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 substances are needed, which emphasises the need to generate integrated eluates.

NOTE 3 When only organic substances are analysed, stainless steel column and fittings can be applied taking into account a certain degree of sorption which can be tested in advance[9]. When only inorganic substances are analysed, column made of plastics can be applied.

NOTE 4 In the case of quartz sand used as filter material, it shall be tested to be free of leachable substances blank free. As necessary it can be washed with demineralized water to remove fines and gently dried afterwards not exceeding 25 °C to avoid enhancing the sorption capacity on the surface of quartz grains. The quartz sand can be treated with acetone first in case of blank values of organic substances under investigation.

NOTE 5 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.

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

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

NOTE This can be tested in preliminary experiments.

6.3 Pre-filters, for the column with a pore size of 1 µm to 20 µm.

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

NOTE This can be tested in preliminary experiments.

6.4 Pump, with an adjustable capacity of between 0 ml/h and 60 ml/h (e.g. peristaltic pump).

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

6.6 pH meter, in accordance with ISO 10523 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, adapted to the analysis to be performed (see ISO 5667-3:2018, Table A.1).

NOTE When both organic and inorganic substances are analysed Perfluoro-Ethylene-Propylene (FEP) can be used. When only inorganic substances are analysed, PTFE or similar tubing materials can be used.

6.9 High-quality **glass bottles** of 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 substances are analysed, alternative bottle materials can be selected [e.g. high density polyethylene (HDPE) or PTFE)].

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

6.11 Sieving equipment, with sieves of 2 mm or 4 mm nominal screen size.

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

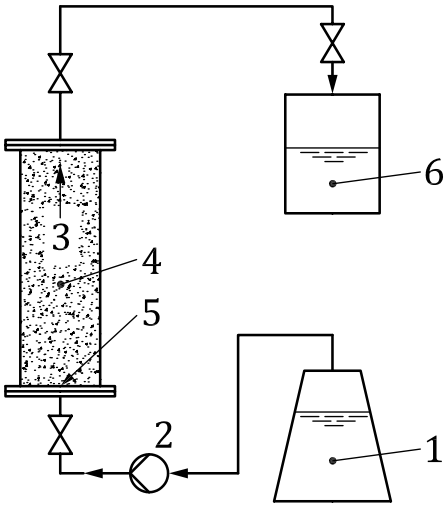
6.13 Redox potential meter (optional).

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

6.15 Centrifuge, refrigerated, operating at 20 000 *g* to 30 000 *g* using centrifuge tubes of appropriate material , which is inert with regard to both inorganic and organic compounds and suitable for high-speed centrifugation (e.g. perfluoroalkoxy alkane (PFA), stainless steel[8].).

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. Guidance on the calculation of the rotor-specific duration of centrifugation is given in Annex C in order to ensure a comparable degree of efficiency of centrifugation at varying rotation speed. Cooling shall be applied to maintain the desired temperature.

6.16 Rammer to support packing of the column as specified in 8.4



Key

1	leachant storage container	4	sample material
2	pump	5	pre-filter and or filter layer
3	column	6	eluate collection bottle

Figure 1 — Schematic view of up-flow percolation test

7 Sample pre-treatment

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 18400-101, ISO 18400-104, ISO 18400-105, ISO 18400-202[10-13] and ISO 23909[14]) 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[12]).

The test shall be carried out on soil or soil-like material sieved to <2 mm (e.g. as described in ISO 11464[9]). 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[9]). 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 (refer also to A.3.6).

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 the test sample

Use a sample splitter (6.12) 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 can 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[14]). 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 Test portion

Take, from the test sample, a test portion with an appropriate volume (e.g. 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.

The inner diameter of the column shall be chosen in such a way that the largest particle size is at least 3 times but preferably 10 times smaller than the diameter of the column.

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 by using an appropriate column dimension within the range specified in 6.1.

NOTE 2 The volume of eluate required depends on the specific purpose and the subsequent chemical analysis and/or ecotoxicological tests to be carried out on the eluate. Analysis for inorganic substances can typically require from 20 ml to 500 ml of eluate, analysis for organic substances from 250 ml to 2 000 ml, depending on the number and type of groups of organic substances to be analysed (DOC from 100 ml to 250 ml), and ecotoxicological testing from 100 ml to 2 000 ml.

7.4 Determination of dry matter content

The whole test sample, complying with the size criterion in 7.1, shall not be further dried. The water 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. The dry mass of the sample shall be determined at (105 ± 5) °C in accordance with ISO 11465 and the dry matter content is calculated in 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 the undried sample, expressed in kilograms (kg).

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

If, for reasons expressed in 7.1, the material was (partly) dried before 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 (22 ± 3) °C.

NOTE A constant temperature of 22 °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. A reduced temperature may result in slower/lower release of organic substances and hence lower concentrations of these compounds in the leachates. If the test is modified in this way, this deviation shall be reported in the test report.

8.2 Preparation of the eluent

Prepare a solution made to 0,001 M $CaCl_2$ by dissolving 0,147 g $CaCl_2$ in water and dilute to 1 000 ml.

In special cases (i.e. measurement of Ca and/or chloride in the eluate are of interest or the sample exhibits an own salt load), water without addition of $CaCl_2$ can also be used. The leachant type used shall be recorded in the test report.

NOTE 1 The application of demineralized water as leachant can induce higher turbidity and lower ionic strength in the eluate for some types of soils (e.g. high content of organic matter) and can cause increased concentrations of analytes adsorbed to colloids.

NOTE 2 For eluates that are not to be used for ecotoxicological testing, sodium azide (NaN_3) can be added to a resulting concentration of 0,1 % in order to reduce microbial degradation of organic substances. However, the addition of NaN_3 is known to only minimize biodegradation if a very high but in turn extremely poisonous concentration in the eluent is applied. Therefore, other appropriate measures can be considered to prevent/reduce biodegradation in the sample or collected eluate (e.g. application of γ-radiation to the sample, dark and air-conditioned room, shorter eluate collection periods, etc). If only inorganic compounds are measured, the addition of NaN_3 is not appropriate.

8.3 Preparation of the column

Rinse the column, including the top and bottom sections (6.1), pre-filters (6.3), tubing material (6.8) 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, 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 substance from the air substance.

8.4 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. Weigh of the rammer, in accordance with Formula (2):

$$r_W = r_C \cdot r_C \cdot 5 \quad (2)$$

where

r_W is the weight of the rammer, expressed in grams (g);

r_C is diameter of the column, expressed in centimeters (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 can 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 is useful.

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) of the test portion in the column, in kilograms, in accordance with Formula (3):

$$m_D = m_W \cdot w_{dm} / 100 \quad (3)$$

where

m_W is the mass of the (moist) test portion in the column, expressed in kilograms (kg);

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

Fit the outlet-hose to the top section of the column.

8.5 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 column for a period of at least 16 to 72 h, in order to equilibrate the system.

After the equilibration period, connect the outlet hose (6.8) to an eluate collection bottle of appropriate size (6.9), start the pump (again) and set the flow rate such that the linear velocity is (15 ± 2) cm/day (as calculated for the empty column). In case faster linear velocity is independent of leaching behaviour of focusing substance and material type shown by conducting preliminary tests or based on literature, it

can be increased up to (30 ± 2) cm/day as an option. The linear velocity used shall be in the test report and justified if applicable.

NOTE 2 The results of a validation study comparing linear velocities between (15 ± 2) and (45 ± 4) cm/day are shown in Reference [15].

Calculate the flow rate in accordance with Formula (4):

$$q = v_L \times \pi \times d^2 \times 0{,}010\,4 \tag{4}$$

where

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

v_L is the linear velocity of the leachant through the empty column, expressed in centimeters per day (cm/day);

d is the diameter of the column, expressed in centimeters (cm).

NOTE 3 For example, 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 49 ml/h.

8.6 Sample Collection and Liquid/Solid separation step

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 eluate corresponding to an L/S ratio of 0,1 ± 0,02 l/kg has passed through.

Transfer the eluate to centrifuge tubes (6.15). The centrifugation containers shall be chosen so as not to adorb (or release) analytes.

There are two options for solid-liquid separation.

a) Centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high-speed centrifuge (6.15).

b) Centrifuge the eluate for 5 h at 2 000 g to 3 000 g in glass bottles using a lower-speed centrifuge (6.15).

Cooling shall be applied to maintain the temperature at (22 ± 3) °C (see 8.1).

NOTE 1 Eluates from column tests on soil frequently exhibit low turbidities (e.g. <100 FNU (Formazin Nephelometric Units)) due to self-filtration. As sorption to centrifuge containers can lead to undesired losses of organic substances, eluates can be directly analysed for these substances if it can be ensured that the turbidity is below 100 FNU and the requirements of the analytical device.

NOTE 2 Based on Stoke's law, the results of both centrifugation methods are expected to be comparable. Other alternative combinations of centrifugation acceleration and time can be applied given comparable conditions are calculated related to the specification of the rotor (see guidance in Annex C).

Gentle braking of the centrifuge shall be applied in order to avoid resuspension. The deceleration time shall not exceed 20 min.

If only inorganic substances are measured, the centrifugation step can be omitted, the eluate can be filtered using the appropriate membrane filters (6.2) and a vacuum or pressure filtration device (6.3), (see Annex B for an example). 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 3 For inorganic substances, it is often preferable to pre-centrifuge the eluate at 2 000 g to 3 000 g for 20 min using glass bottles with a screw cap and PTFE inlay prior to filtration.

Measure immediately the pH and electrical conductivity of this eluate portion. Measurement of turbidity, redox potential (E_h in mV) and DOC is highly recommended.

NOTE 4 Information on DOC concentration in the eluate is is relevant both for release of inorganic substances, as well as for organic substances.

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-like materials, to prevent the occurrence of oxidation reactions.

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 might change the pH and redox potential of the eluate.

8.7 Collection of additional eluate fractions

Check the flow rate of the leachant and possible clogging of the pre-filter as often as needed, but at least three times per week, and adjust to the original linear velocity, in the range of the defined flow rate (±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 8.6. Measure the pH and electrical conductivity, of each eluate portion. Measurement of the DOC, 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 — 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 ± 0,02) × m_D	0,1 ± 0,02
2	(0,1 ± 0,02) × m_D	0,2 ± 0,04
3	(0,3 ± 0,05) × m_D	0,5 ± 0,08
4	(0,5 ± 0,1) × m_D	1,0 ± 0,15
5	(1,0 ± 0,2) × m_D	2,0 ± 0,3
6	(3,0 ± 0,2) × m_D	5,0 ± 0,4
7	(5,0 ± 0,2) × m_D	10,0 ± 0,5

[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 specific purposes, e.g. ecotoxicological testing, consecutive fractions can be combined to fewer resulting fractions as needed.

NOTE 3 For specific scenarios (for instance, evaluating the long-term leaching characteristics), the L/S ratio for sampling can be set depending on the user's purpose.

NOTE 4 When no automated eluate collection apparatus is available, a collection scheme can be composed within the tolerance ranges that are allowed (flow rate ± 2) cm/d, and in fraction volume (see Table 1), that enables eluate collection within working hours.

NOTE 5 The execution time of the test can be calculated from Formula (5):

$$t = \left(\mathrm{L/S} \times m_{\mathrm{D}} \times 1\,000\right) / \left(24 \times q\right) \tag{5}$$

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).

8.8 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.

8.9 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 2 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 8.8.

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 Calculation

Calculate, for each substance, the quantities released in all eluate fractions with Formula (6):

$$w_i = \left(V_i \times \phi_i\right) / m_{\mathrm{D}} \tag{6}$$

where

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

w_i is the released quantity of a substance 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 substance 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 Formula (6), is the concentration originally present in the eluate. The measured value, determined in accordance with 8.8, shall be corrected if the eluate fraction has been diluted and/or if the quantity of preservation fluid added in 8.8 was more than 1 ml per 100 ml of eluate.

Where the concentration of a substance in one or more eluate fractions is below the limit of detection, two calculations shall be carried out for this substance 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 substance, the cumulatively released quantity (Σw_i) shall be calculated by accumulating the released quantities of the specific substance, measured in the different eluate fractions. Where the concentration of a substance in one or more eluate fractions is below the lower detection limit, for this substance 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 document and include the following details:

a) a reference to this document;

b) address of laboratory, name of responsible person;

c) any information necessary for the complete identification of the sample including size of laboratory sample, test sample and test portion, sample preparation details, full sample storage history, dry matter content of test portions;

d) any information on the test conditions, column size, equilibration time, average flow rate (cm/day), type leachant, L/S ratio of each eluate fraction as well as cumulative L/S ratios, start and end date of the test. If a flow rate of 30 cm/day is used, the justification, i.e. the results of the preliminary testing must be attached to the test report;

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

f) the test results including at least pH, electrical conductivity, measured concentrations (mg/l), and released quantities (mg/kg dry matter) of all eluate fractions, and cumulative released quantities (mg/kg dry matter), limit of detection for each substance;

g) the blank test results;

h) any details that are optional or deviations from the specifications of this document, and any effects which may have affected the results.

11 Analytical determination

11.1 General

Since the analysis step is not included in the scope of this document, the analytical method applied together with the limit of quantification shall be reported.

11.2 Blank test information

The following shall be included in the test report:

— date of the last blank test performed;

— results of the blank test, including the elements considered for the tested material and the levels above which the results can be considered as valid, when compared with the measured concentrations, in mg/l.

12 Performance characteristics

12.1 General

All validation results were obtained in accordance with the principles of ISO 5725-1 and ISO 5725-2.

Some of the values reported for the reproducibility, repeatability, and the number of outliers are relatively high (particularly at low concentrations) and reflect what can currently be achieved in testing laboratories. There are no specific criteria to determine whether these values are acceptable or not. The values for the reproducibility and repeatability can be used to derive uncertainties associated with testing results.

The performance of the test regarding repeatability and reproducibility is dependent on the tested material and also on the testing conditions. Several validation studies were carried out for confirming the robustness, repeatability and reproducibility of this document. A summary of the validation studies is shown in Table 2. An overview of some validation studies is shown in 12.2 and 12.3.

Table 2 — Summary of results of round robin tests and ruggedness tests

No.	Type	Substances	Type of tests	Method	N*	S*	Results	Reference
1	Soil	Inorganic	Robustness test/ring test	ISO/TS 21268-3	34	1	CV values for both (a) concentration and (b)cumulative amount are below 30 %	Yasutaka et al (2017)[15]
2	Soil	Inorganic	Robustness test/ring test	ISO/TS 21268-3, Changing flow rate: 36 ml/h Changing duration time of initial saturation: 16 h	2	2	CV values for both (a) concentration and (b)cumulative amount are below 30 %	Yasutaka et al (2017)[15]
3	Soil	Inorganic	Ruggedness test	Changing flow rate:12 ml/h, 36 ml/h	4	1	Results of experiments with 12 ml/h and 36 ml/h show good agreement.	Naka et al (2017)[16]
4	Soil	Inorganic	Ruggedness test	Changing duration time of initial saturation: 0 h, 16 h, 48 h	4	1	Results of experiments with 16 h and 48 h show good agreement.	Naka et al (2017)[16]
5	Contaminated soil	Inorganic and PAH	Validation ring test	Slightly differing column dimensions but fixed contact time of 5 h, demineralized water as leachant, centrifugation only if the turbidity is >100 FNU	25	1	DIN 19528 has been confirmed as validated based on the results	DIN 19528[5], (see 11.2))
6 (see N298)	Contaminated soil	Organic (PAH, PCB, TPH, Phenols)	Validation ring test	Slightly differing column dimensions but fixed contact time of 5 h, demineralized water as leachant, centrifugation only if the turbidity is >100 FNU	7-16	4	Good results. Validation of DIN 19528 has been widened.	DIN 19528[5], (see 11. 3))
N*: Number of tests, S*: Number of (soil) samples								

12.2 Validation trials performed in Japan

12.2.1 Round robin tests performed in accordance with ISO/TS 21268-3:2007

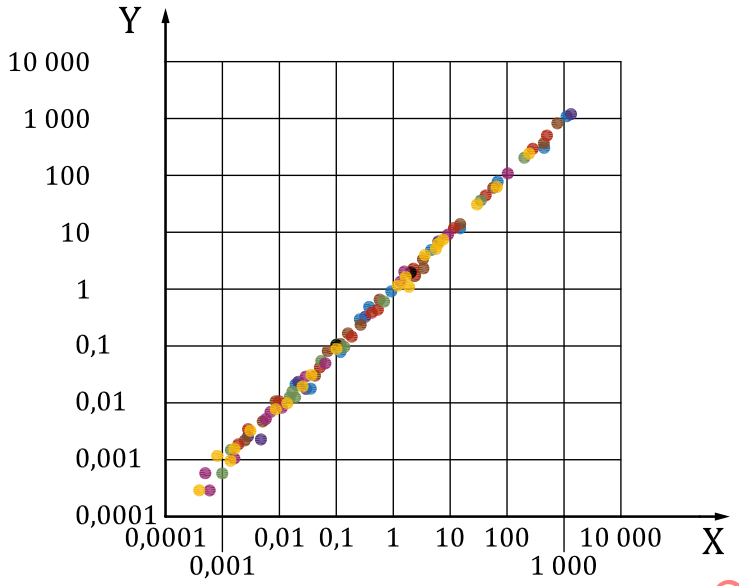
In order to examine the test's reproducibility and repeatability, seventeen laboratories participated in the round robin test based on ISO/TS 21268-3:2007. Nearly all CV (the coefficient of variation) values of the substance's concentrations in each fraction were below 30 % (see Table 3). For the cumulative amounts of inorganic substances, all CV values were below 30 % at all L/S ratios. Detailed information of this validation study was shown in Reference [16].

Table 3 — CV* values (%) of the substance's concentrations in each fraction of validation study[16]

Elements	Number of column experiments	L/S = 0,1	L/S = 0,2	L/S = 0,5	L/S = 1,0	L/S = 2,0	L/S = 5,0	L/S = 10
Cu	34	17	14	13	17	14	23	38
As	34	11	9	8	11	11	11	23
Se	34	22	18	26	26	28	40	71
F	34	22	21	17	17	21	12	11
Ca	34	20	15	16	13	22	8	7
CV*: The coefficient of variation								

12.2.2 Robustness testing and validation results considering equilibration period and flow rate

Three equilibration periods (0 h, 12 h to 16 h, and 48 h) and two flow rates (15 cm/day and 45 cm/day) for four different soils were evaluated and the inorganic substance releases were compared. These results indicated that the equilibration time of 12 h to 16 h and the flow rate of 45 cm/day were optimal for inorganic substances. The reproducibility of column performance tests for soil A and B using the CV has been calculated; more than 90 % of the values were within 30 %, under ISO/TS 21268-3: 2007 test conditions. Detailed information of this validation study was shown in Reference [17].



Key

X original method, concentration [mg/l] (48 h — 12 ml/h)

Y modified method, concentration [mg/l] (12 to 16 h — 36 ml/h)

- L/S = 0,1 l/kg
- L/S = 0,2 l/kg
- L/S = 0,5 l/kg
- L/S = 1 l/kg
- L/S = 2 l/kg
- L/S = 5 l/kg
- L/S = 10 l/kg

Figure 2 — Validation study results comparing two equilibration periods (12 h to 16 h, and 48 h) and two flow rates (15 cm/day and 45 cm/day) for four different soils[16]

12.3 Validation results obtained in Germany (DIN 19528[5])

12.3.1 General

Data on robustness, repeatability and reproducibility of selected inorganic and organic substances are available from German research studies for the validation of DIN 19528[5] based on soils and soil-like materials (Table 5 and Table 8). The test conditions are almost the same to this document except the eluent which was always demineralized water, an additional pre-equilibration was not performed after saturation and the linear flow velocity was 45 cm/day (see Table 4). The grain size of the soil was either <2 mm or partly <10 mm (no crushing was applied during sample preparation). The eluates were analysed directly if the turbidity was <100 FNU or centrifuged prior to analysis if the turbidity exceeded 100 FNU. The results can be adopted to this document taking into account the limitations described.

All validation results were calculated in accordance with the principles of ISO 5725-1 and ISO 5725-2 (see Tables 6, 7 and 9 to 13).

Table 4 — Relevant test conditions applied in the validation study according to DIN 19528

Column diameter/filling height	5 cm to 10 cm (at least 2× max. particle size)/at least 4× diameter
Column	glass, plastic if only inorganics are investigated

Table 4 *(continued)*

Column diameter/filling height	5 cm to 10 cm (at least 2× max. particle size)/at least 4× diameter
Leachant	Demineralised water (grade 3, ISO 3696)
Amount of solid	Depending on the column diameter
Recommended packing methods	rammer, rubber mullet from outside, vibration using sieving machine
Top and bottom section of column	quartz sand layers (grain size 0 mm,6 mm to 1,2 mm), at top min. 2 cm
Pre-equilibration	saturation within two hours and direct start after saturation
L/S (l/kg)	basic characterisation: 4 fractions at 0,3 ± 0,05, 1 ± 0,2, 2 ± 0,4 and 4 ± 0,8 l/kg compliance test: one fraction at 2 ± 0,05 l/kg, or calculation from cumulative release at 2 l/kg from basic characterisation test
Flow rate	calculation of flow rate based on bulk density and fixed contact time of 5 h leads to overall test duration of about 1 week for basic characterization and 2-3 days for compliance test
Liquid/solid separation	Only analysis of organics: no filtration, centrifugation only if turbidity ≥100 FNU (20 000 *g*, 30 min or appropriate, at least 2 000 *g*, 5 h with cooling), otherwise no further eluate pre-treatment This centrifugate may also be taken for the analysis of inorganics. Only analysis of inorganics: filtration through 0,45 µm membrane may be applied (e.g. syringe filters) only if needed for protection of measurement devices regarding allowed particle amount, otherwise no further eluate pre-treatment

12.3.2 Results for validation trial 1

Table 5 — Characteristics of the test material RM BO (contaminated soil-like material, <10 mm)

Parameter	Dimension	RM BO
Humidity	% by weight	0,51
pH-value	—	8,51
Electrical conductivity	µS/cm	242,1
Carbonate content	% by weight	3,77
Loss on ignition	% by weight	1,14
Particle density	g/cm^3	2,652
Grain size distribution (dry sieving, no crushing):		
10 mm – 6,3 mm	% by weight	2,37
6,3 mm – 2 mm	% by weight	9,92
2 mm – 0,63 mm	% by weight	17,88
0,63 mm – 0,2 mm	% by weight	54,65
0,2 mm – 0,063 mm	% by weight	13,81
<0,063 mm	% by weight	1,38

Table 6 — Performance characteristics for metals

Sub-stance	Sample	unit	l	n	n_A	n_{AP} %	$\bar{x}$ (µg/l)	s_R	CV_R %	s_r	CV_R %
Cr	Fraction 1	µg/l	21	39	4	9,30	7,47	4,69	62,72	0,90	12,1
	Fraction 2	µg/l	14	24	7	22,58	2,58	1,20	46,45	0,10	3,7
	Fraction 3	µg/l	11	18	0	0,00	4,06	2,40	59,00	0,80	19,6
	Fraction 4	µg/l	6	11	1	8,33	2,37	1,51	63,79	0,64	26,8
	Cumulative release at L/S 2 l/kg	mg/kg	5	9	4	30,77	0,01	0,003	45,63	0,000 2	3,4
Cu	Fraction 1	µg/l	25	48	11	18,64	36,31	11,85	32,64	3,06	8,42
	Fraction 2	µg/l	29	56	2	3,45	25,37	7,90	31,13	2,77	10,91
	Fraction 3	µg/l	30	57	2	3,39	20,87	7,64	36,63	3,14	15,06
	Fraction 4	µg/l	31	58	0	0	13,15	4,72	35,92	2,39	18,13
	Cumulative release at L/S 2 l/kg	mg/kg	27	52	6	10,34	0,05	0,02	37,67	0,004	8,93
Pb	Fraction 1	µg/l	3	6	1	14,29	0,23	0,15	67,79	0,09	38,31
	Fraction 2	µg/l	7	11	0	0	3,46	4,82	139,09	0,21	6,14
	Fraction 3	µg/l	8	13	4	23,53	4,40	7,12	161,94	0,28	6,30
	Fraction 4	µg/l	8	15	0	0	7,69	10,69	138,98	0,86	11,24
	Cumulative release at L/S 2 l/kg	mg/kg	4	7	0	0	0,001	0,002	101,20	0,000 3	18,73
Zn	Fraction 1	µg/l	25	44	0	0	47,78	58,94	123,37	31,81	66,59
	Fraction 2	µg/l	21	39	0	0	31,03	39,35	127,13	15,77	50,82
	Fraction 3	µg/l	18	33	7	17,5	20,69	17,54	84,80	10,33	49,95
	Fraction 4	µg/l	19	35	6	14,63	19,39	21,34	110,09	17,15	88,48
	Cumulative release at L/S 2 l/kg	mg/kg	16	31	2	6,06	0,06	0,06	105,88	0,02	32,83
Ni	Fraction 1	µg/l	22	39	2	4,88	9,80	7,65	78,05	4,22	43,10
	Fraction 2	µg/l	13	24	0	0	6,34	5,39	85,09	2,15	33,89
	Fraction 3	µg/l	10	17	4	19,05	3,64	2,42	66,55	1,81	49,66
	Fraction 4	µg/l	9	17	2	10,53	4,22	3,62	85,61	0,98	23,29
	Cumulative release at L/S 2 l/kg	mg/kg	10	19	0	0	0,01	0,01	100,00	0,003	18,73

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 7 — Performance characteristics for PAH

Substance	Sample	unit	l	n	n_A	n_{AP} %	$\bar{x}$ (µg/l)	s_R	CV_R %	s_r	CV_R %
Sum PAH	Fraction 1	µg/l	25	47	0	0,00	104,63	70,40	67,28	15,37	14,69
	Fraction 2	µg/l	25	48	0	0,00	85,36	57,34	67,17	7,62	8,93
	Fraction 3	µg/l	24	46	2	4,17	64,99	45,79	70,46	10,29	15,84
	Fraction 4	µg/l	24	45	2	4,26	26,69	30,57	114,55	4,71	17,64
	Cumulative release at L/S 2 l/kg	mg/kg	25	46	0	0,00	0,15	0,09	62,74	0,02	10,26
Naph-thaline	Fraction 1	µg/l	19	33	0	0,00	2,13	1,55	72,51	0,32	14,99
	Fraction 2	µg/l	10	17	4	19,05	0,40	0,30	75,81	0,07	18,25
	Fraction 3	µg/l	12	20	2	9,09	0,37	0,34	92,11	0,05	13,56
	Fraction 4	µg/l	12	20	2	9,09	0,18	0,18	99,70	0,04	23,38
	Cumulative release at L/S 2 l/kg	mg/kg	7	12	0	0,00	0,001	0,001	50,84	0,000 2	11,79
Anthracene	Fraction 1	µg/l	20	37	4	9,76	0,66	0,50	75,83	0,05	8,15
	Fraction 2	µg/l	21	40	2	4,76	0,53	0,39	73,49	0,13	23,99
	Fraction 3	µg/l	21	39	2	4,88	0,39	0,37	94,31	0,10	25,84
	Fraction 4	µg/l	17	33	2	5,71	0,12	0,11	92,19	0,03	26,53
	Cumulative release at L/S 2 l/kg	mg/kg	18	34	0	0,00	0,001	0,001	65,02	0,000 2	15,94
Pyrene	Fraction 1	µg/l	25	47	0	0,00	2,09	1,40	67,13	0,19	9,06
	Fraction 2	µg/l	25	48	0	0,00	2,63	1,38	52,33	0,31	11,91
	Fraction 3	µg/l	24	46	2	4,17	3,01	1,58	52,53	0,34	11,15
	Fraction 4	µg/l	25	47	0	0,00	2,22	1,00	45,26	0,25	11,28
	Cumulative release at L/S 2 l/kg	mg/kg	24	44	2	4,35	0,01	0,003	53,24	0,000 5	8,99
Chrysene	Fraction 1	µg/l	16	40	0	0,00	0,08	0,06	75,93	0,01	9,35
	Fraction 2	µg/l	20	39	0	0,00	0,17	0,14	78,55	0,04	21,78
	Fraction 3	µg/l	20	36	4	10,00	0,25	0,18	71,41	0,05	19,00
	Fraction 4	µg/l	20	38	0	0,00	0,15	0,10	66,31	0,03	23,46
	Cumulative release at L/S 2 l/kg	mg/kg	15	27	0	0,00	0,000 4	0,000 2	58,31	0,000 1	30,64

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 7 *(continued)*

Substance	Sample	unit	l	n	n_A	n_{AP} %	$\bar{x}$ (µg/l)	s_R	CV_R %	s_r	CV_R %
Benzo-a-pyrene	Fraction 1	µg/l	3	5	0	0,00	0,01				
	Fraction 2	µg/l	11	18	2	10,00	0,02	0,02	66,96	0,01	39,40
	Fraction 3	µg/l	16	30	0	0,00	0,08	0,06	71,88	0,03	39,06
	Fraction 4	µg/l	17	33	0	0,00	0,04	0,03	79,31	0,01	28,28
	Cumulative release at L/S 2 l/kg	mg/kg	2	4	0	0,00	0,000 13	0,000 01	9,70	0,000 02	12,33

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

12.3.3 Results for validation trial 2

The validation trial was based on two different reference soils which were mixed with contaminated soil to four test materials. Demineralized water was applied as leachant. The compliance test option of DIN 19528 was performed for all materials, i.e. one fraction up to L/S 2 l/kg was collected.

Table 8 — Characteristics of the test materials

Test material	Soil type	pH [-]	C_{org} [weight%]	Cation exchange capacity CEC_{eff} [mmol/kg]
Soil TL	clayey loam	4,97	3,52	118
Soil MS	medium sand	8,48	0,64	8,3

Both soils were sieved at 2 mm, no crushing of oversized material.

The CEC was expressed as charge concentration.

Sample codes as listed in Tables 9 to 13:

— TL-PAH/PCB soil (clayey loam) contaminated with PAH and PCB, <2mm;

— TL-PH/TPH/PAH soil (clayey loam) contaminated with phenols, TPH and PAH, <2mm;

— MS-PAK/PCB soil (medium sand) contaminated with PAH and PCB, <2mm;

— MS-PH/TPH/PAH soil (medium sand) contaminated with phenols, PAH and PCB, <2mm.

Table 9 — Performance characteristics for basic parameters for validation trial 2

Substance	Soil	unit	l	n	n_A	n_{AP}	$\bar{x}$ (µg/l)	s_R	CV_R	s_r	CV_r
Turbidity	TL-PAH/PCB	FNU	11	22	4	15,4 %	9,8	10,9	111,3 %	2,4	24,1 %
	TL-PH/TPH/PAH	FNU	11	22	4	15,4 %	11,1	5,4	48,7 %	3,9	34,8 %
	MS-PAH/PCB	FNU	13	25	2	7,4 %	16,3	14,1	86,7 %	6,8	41,7 %
	MS-PH/TPH/PAH	FNU	10	19	0	0,0 %	5,4	3,2	58,4 %	2,1	39,4 %
DOC	TL-PAH/PCB	mg/l	12	24	0	0,0 %	47,2	24,5	51,9 %	6,4	13,6 %
	TL-PH/TPH/PAH	mg/l	8	16	0	0,0 %	406,2	67,7	16,7 %	59,1	14,6 %
	MS-PAH/PCB	mg/l	12	23	2	8,0 %	16,2	2,9	18,1 %	1,1	6,9 %
	MS-PH/TPH/PAH	mg/l	8	16	0	0,0 %	453,3	37,5	8,3 %	23,0	5,1 %
pH	TL-PAH/PCB		12	24	0	0,0 %	7,45	0,36	4,8 %	0,15	2,1 %
	TL-PH/TPH/PAH		8	16	2	11,1 %	7,02	0,41	5,9 %	0,08	1,1 %
	MS-PAH/PCB		13	25	0	0,0 %	7,84	0,42	5,4 %	0,11	1,4 %
	MS-PH/TPH/PAH		9	18	0	0,0 %	7,38	0,28	3,8 %	0,20	2,7 %

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 10 — Performance characteristics for PAH for validation trial 2

Substance	Soil	l	n	n_A	n_{AP}	$\bar{x}$ (µg/l)	s_R	CV_R	s_r	CV_r
Naphthalene	TL-PAH/PCB	13	24	0	0,0 %	7,0	5,3	75,3 %	2,4	33,8 %
	TL-PH/TPH/PAH	12	23	0	0,0 %	346,7	165,3	47,7 %	44,4	12,8 %
	MS-PAH/PCB	10	17	0	0,0 %	0,9	1,4	152,4 %	0,8	86,5 %
	MS-PH/TPH/PAH	13	24	0	0,0 %	482,1	180,7	37,5 %	46,1	9,6 %
Acenaphthene	TL-PAH/PCB	15	28	0	0,0 %	4,2	2,7	64,8 %	1,2	29,5 %
	TL-PH/TPH/PAH	12	23	0	0,0 %	7,8	3,7	47,6 %	0,6	8,1 %
	MS-PAH/PCB	12	23	4	14,8 %	18,6	22,4	120,8 %	2,5	13,6 %
	MS-PH/TPH/PAH	12	23	0	0,0 %	12,7	3,6	28,3 %	1,1	8,4 %
Fluorene	TL-PAH/PCB	15	28	0	0,0 %	2,5	1,8	74,1 %	0,8	30,8 %
	TL-PH/TPH/PAH	12	23	0	0,0 %	25,7	10,0	38,8 %	2,6	10,1 %
	MS-PAH/PCB	10	20	5	20,0 %	0,3	0,3	107,7 %	0,0	13,5 %
	MS-PH/TPH/PAH	13	24	0	0,0 %	44,1	15,0	33,9 %	3,0	6,7 %
Phenanthrene	TL-PAH/PCB	14	25	2	7,4 %	1,9	1,9	99,9 %	0,5	28,5 %
	TL-PH/TPH/PAH	12	23	0	0,0 %	23,4	13,1	55,8 %	4,9	21,1 %
	MS-PAH/PCB	10	18	6	25,0 %	0,1	0,1	59,6 %	0,0	21,3 %
	MS-PH/TPH/PAH	13	24	0	0,0 %	38,1	17,9	47,0 %	9,0	23,7 %

Table 10 *(continued)*

Substance	Soil	l	n	n_A	n_{AP}	$\bar{x}$ (µg/l)	s_R	CV_R	s_r	CV_r
Anthracene	TL-PAH/PCB	16	29	0	0,0 %	1,4	0,9	69,6 %	0,4	26,5 %
	TL-PH/TPH/PAH	12	23	0	0,0 %	5,5	2,6	46,0 %	0,9	16,0 %
	MS-PAH/PCB	12	22	4	15,4 %	0,5	0,5	96,8 %	0,3	65,0 %
	MS-PH/TPH/PAH	13	24	0	0,0 %	13,0	5,6	42,8 %	0,1	0,9 %
Fluoranthene	TL-PAH/PCB	14	25	2	7,4 %	0,6	0,3	58,9 %	0,1	21,6 %
	TL-PH/TPH/PAH	12	22	0	0,0 %	4,1	1,7	41,2 %	0,5	12,3 %
	MS-PAH/PCB	14	26	4	13,3 %	4,1	3,2	76,7 %	0,6	13,5 %
	MS-PH/TPH/PAH	13	23	0	0,0 %	5,6	3,1	55,4 %	0,6	10,0 %
Pyrene	TL-PAH/PCB	13	23	6	20,7 %	0,2	0,1	57,7 %	0,0	7,3 %
	TL-PH/TPH/PAH	12	22	0	0,0 %	2,5	1,2	46,5 %	0,3	11,8 %
	MS-PAH/PCB	15	27	4	12,9 %	2,1	1,6	77,3 %	0,3	13,9 %
	MS-PH/TPH/PAH	11	21	0	0,0 %	3,5	1,6	47,1 %	0,4	11,5 %
Benzo(a) anthracene	TL-PAH/PCB	6	10	3	23,1 %	0,23	0,02	6,5 %	0,01	2,2 %
	TL-PH/TPH/PAH	10	18	0	0,0 %	0,19	0,14	72,4 %	0,06	31,8 %
	MS-PAH/PCB	12	22	4	15,4 %	0,14	0,12	85,3 %	0,04	30,1 %
	MS-PH/TPH/PAH	10	18	0	0,0 %	0,30	0,27	88,1 %	0,06	20,2 %
Chrysene	TL-PAH/PCB	6	11	6	35,3 %	0,01	0,01	35,7 %		0,0 %
	TL-PH/TPH/PAH	9	16	2	11,1 %	0,10	0,05	51,5 %	0,05	44,6 %
	MS-PAH/PCB	11	22	2	8,3 %	0,10	0,07	70,3 %	0,04	34,7 %
	MS-PH/TPH/PAH	10	18	0	0,0 %	0,24	0,19	80,1 %	0,04	15,8 %
Benzo(b) fluoranthene	TL-PAH/PCB	6	11	0	0,0 %	0,04	0,05	145,7 %	0,05	145,7 %
	TL-PH/TPH/PAH	7	11	2	15,4 %	0,06	0,04	70,9 %	0,01	14,5 %
	MS-PAH/PCB	10	19	2	9,5 %	0,06	0,05	74,2 %	0,03	51,6 %
	MS-PH/TPH/PAH	5	9	2	18,2 %	0,04	0,03	68,4 %	0,01	15,8 %
Benzo(k) fluoranthene	TL-PAH/PCB	4	6	0	0,0 %	0,04	0,05	117,5 %	0,05	117,5 %
	TL-PH/TPH/PAH	5	9	0	0,0 %	0,06	0,05	91,1 %	0,02	33,9 %
	MS-PAH/PCB	6	11	2	15,4 %	0,04	0,03	82,1 %	0,02	41,0 %
	MS-PH/TPH/PAH	2	4	2	33,3 %	0,01	0,01	54,5 %	0,01	45,5 %
Benzo(a) pyrene	TL-PAH/PCB	4	6	2	25,0 %	0,05	0,05	100,0 %		0,0 %
	TL-PH/TPH/PAH	6	10	2	16,7 %	0,07	0,04	57,4 %	0,01	11,8 %
	MS-PAH/PCB	7	13	2	13,3 %	0,04	0,04	92,7 %	0,02	43,9 %
	MS-PH/TPH/PAH	5	9	2	18,2 %	0,03	0,02	64,0 %	0,00	16,0 %
Benzo(g,h,i) perylene	TL-PAH/PCB	2	3	2	40,0 %	0,01		0,0 %		0,0 %
	TL-PH/TPH/PAH	3	5	0	0,0 %	0,05	0,02	46,0 %	0,02	46,0 %
	MS-PAH/PCB	6	11	2	15,4 %	0,03	0,02	80,0 %	0,01	46,7 %
	MS-PH/TPH/PAH	2	3	0	0,0 %	0,17	0,21	127,5 %	0,21	127,5 %
Dibenzo(a,h) anthracene	TL-PAH/PCB	2	3	0	0,0 %	0,02	0,02	75,0 %	0,02	75,0 %
	TL-PH/TPH/PAH	2	4	1	20,0 %	0,01		0,0 %		0,0 %
	MS-PAH/PCB	2	3	0	0,0 %	0,04	0,04	81,4 %		0,0 %
	MS-PH/TPH/PAH	2	3	0	0,0 %	0,02	0,02	114,3 %	0,02	114,3 %
Inde-no(1,2,3-cd) pyrene	TL-PAH/PCB	4	6	0	0,0 %	0,02	0,02	73,9 %	0,01	43,5 %
	TL-PH/TPH/PAH	4	6	2	25,0 %	0,06	0,06	116,4 %		0,0 %
	MS-PAH/PCB	5	9	0	0,0 %	0,09	0,13	143,3 %	0,02	16,7 %
	MS-PH/TPH/PAH	4	7	2	22,2 %	0,06	0,07	125,0 %	0,01	25,0 %

Table 10 *(continued)*

Substance	Soil	l	n	n_A	n_{AP}	$\bar{x}$ (µg/l)	s_R	CV_R	s_r	CV_r
Sum 15 PAH	TL-PAH/PCB	15	28	0	0,0 %	16,7	12,9	77,3 %	5,5	32,7 %
	TL-PH/TPH/PAH	10	19	0	0,0 %	478,7	255,6	53,4 %	58,9	12,3 %
	MS-PAH/PCB	15	28	2	6,7 %	31,1	35,9	115,6 %	3,1	9,9 %
	MS-PH/TPH/PAH	11	21	0	0,0 %	654,8	259,9	39,7 %	43,2	6,6 %

l = number of labs after outlier elimination;
n = number of single results after outlier elimination;
n_A = number of outliers;
n_{AP} = percentage of outliers;
$\bar{x}$ = mean value;
s_R = reproducibility standard deviation;
CV_R = reproducibility coefficient [%];
s_r = repeatability standard deviation;
CV_r = repeatability coefficient [%].

Table 11 — Performance characteristics for PCB for validation trial 2

Parameter	Soil	l	n	n_A	n_{AP}	$\bar{x}$ (µg/l)	s_R	CV_R	s_r	CV_r
PCB 28+31	TL-PAK/PCB	11	19	0	0,0 %	0,025	0,015	60,0 %	0,004	16,0 %
	MS-PAK/PCB	9	16	6	27,3 %	0,082	0,062	75,6 %	0,010	12,2 %
PCB 52	TL-PAK/PCB	12	22	2	8,3 %	0,123	0,100	81,3 %	0,014	11,4 %
	MS-PAK/PCB	11	19	6	24,0 %	0,617	0,507	82,2 %	0,090	14,6 %
PCB 101	TL-PAK/PCB	12	22	2	8,3 %	0,045	0,041	91,1 %	0,008	17,8 %
	MS-PAK/PCB	12	21	4	16,0 %	0,248	0,221	89,1 %	0,151	60,9 %
PCB 153	TL-PAK/PCB	11	19	0	0,0 %	0,029	0,041	141,4 %	0,021	72,4 %
	MS-PAK/PCB	12	21	4	16,0 %	0,078	0,066	84,6 %	0,037	47,4 %
PCB 138	TL-PAK/PCB	10	17	4	19,0 %	0,013	0,006	46,2 %	0,004	30,8 %
	MS-PAK/PCB	10	17	8	32,0 %	0,089	0,081	91,0 %	0,007	7,9 %
PCB 180	TL-PAK/PCB	3	6	0	0,0 %	0,018	0,012	66,7 %	0,009	50,0 %
	MS-PAK/PCB	12	21	0	0,0 %	0,052	0,081	155,8 %	0,066	126,9 %
SUM PCB	TL-PAK/PCB	11	21	2	8,7 %	0,232	0,198	85,3 %	0,029	12,5 %
	MS-PAK/PCB	10	18	6	25,0 %	1,111	0,910	81,9 %	0,146	13,1 %

l = number of labs after outlier elimination;
n = number of single results after outlier elimination;
n_A = number of outliers;
n_{AP} = percentage of outliers;
$\bar{x}$ = mean value;
s_R = reproducibility standard deviation;
CV_R = reproducibility coefficient [%];
s_r = repeatability standard deviation;
CV_r = repeatability coefficient [%].

Table 12 — Performance characteristics for TPH for validation trial 2

Parameter	Soil	l	n	n_A	n_{AP}	$\bar{x}$ (µg/l)	s_R	CV_R	s_r	CV_r
TPH C_{10}-C_{22}	TL-PH/TPH/PAH	8	16	2	11,1 %	411,9	121,2	29,4 %	77,0	18,7 %
	MS-PH/TPH/PAH	10	19	0	0,0 %	738,1	450,6	61,0 %	188,5	25,5 %
TPH C_{10}-C_{40}	TL-PH/TPH/PAH	9	17	0	0,0 %	863,0	831,4	96,3 %	60,3	7,0 %
	MS-PH/TPH/PAH	10	19	0	0,0 %	828,2	507,4	61,3 %	205,6	24,8 %

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Table 13 — Performance characteristics for phenols for validation trial 2 (all concentrations in µg/l):

Parameter	Soil	l	n	n_A	n_{AP}	$\bar{x}$	s_R	CV_R	s_r	CV_r
Phenol	TL-PH/TPH/PAH	7	13	0	0,0 %	526	410	78,1 %	75,9	14,4 %
	MS-PH/TPH/PAH	7	13	0	0,0 %	1286	1036	80,6 %	263	20,4 %
2-Methylphenol (o-Kresol)	TL-PH/TPH/PAH	7	13	0	0,0 %	9,5	4,2	43,9 %	1,7	18,3 %
	MS-PH/TPH/PAH	6	11	2	15,4 %	1 415	567	40,1 %	85,3	6,0 %
3-Methylphenol (m-Kresol)	TL-PH/TPH/PAH	7	14	0	0,0 %	818	477	58,4 %	136	16,7 %
	MS-PH/TPH/PAH	6	11	2	15,4 %	2 158	806	37,4 %	155	7,2 %
4-Methylphenol (p-Kresol)	TL-PH/TPH/PAH	5	9	0	0,0 %	19,1	17,0	88,7 %	3,1	16,0 %
	MS-PH/TPH/PAH	5	9	2	18,2 %	1 629	272	16,7 %	43,5	2,7 %
2,6-Dimethyl phenol	TL-PH/TPH/PAH	4	7	0	0,0 %	4,2	6,2	148,8 %	0,9	20,8 %
	MS-PH/TPH/PAH	6	11	0	0,0 %	248	349	140,8 %	7,4	3,0 %
3,4-Dimethyl phenol	TL-PH/TPH/PAH	4	7	2	22,2 %	3,3	1,9	58,6 %	0,6	19,5 %
	MS-PH/TPH/PAH	6	11	0	0,0 %	605	294	48,5 %	80,2	13,2 %
Sum phenols	TL-PH/TPH/PAH	6	12	0	0,0 %	1 285	934	72,7 %	359	27,9 %
	MS-PH/TPH/PAH	6	11	0	0,0 %	8173	5 637	69,0 %	2489	30,5 %

l = number of labs after outlier elimination;

n = number of single results after outlier elimination;

n_A = number of outliers;

n_{AP} = percentage of outliers;

$\bar{x}$ = mean value;

s_R = reproducibility standard deviation;

CV_R = reproducibility coefficient [%];

s_r = repeatability standard deviation;

CV_r = repeatability coefficient [%].

Annex A
(informative)

Suggestions for packing the column, water saturationand 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 as follows:

— 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 lower 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.

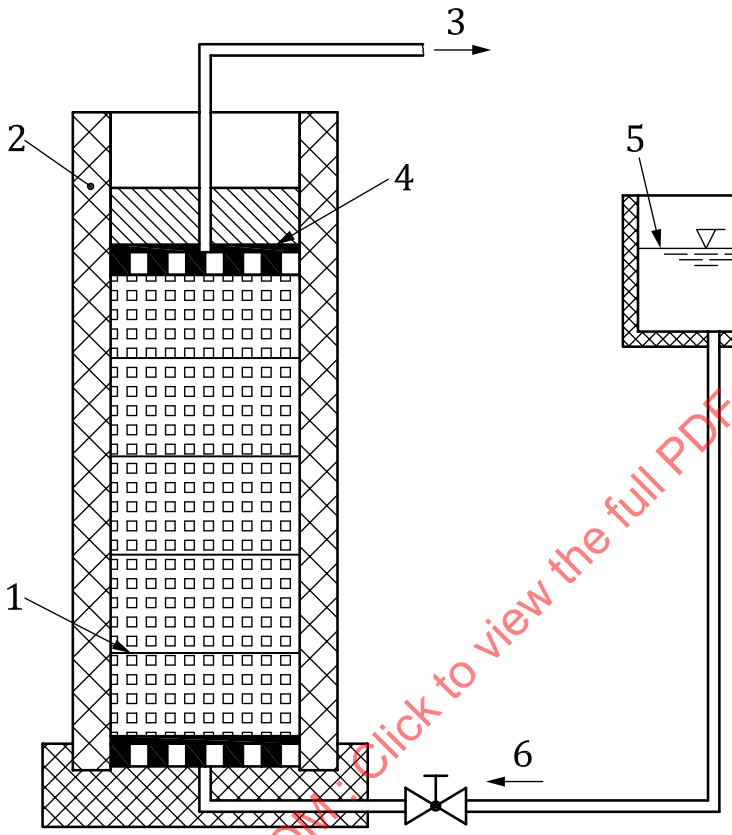
a) **Saturating the column by using the pump**

Connect the pump (6.4) to the bottom section of the column and pass 0,001 mol/l $CaCl_2$ (8.2) or demineralized water (5.1) through the column from bottom to top. 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 adjusted to the top of the soil in the column (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 soil or soil-like material	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 General

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-like 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 optimize 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 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, and for some materials also to 30 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 generalized.

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.