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DRAFT

Leaching behaviour tests – Up-flow percolation test – horizontal standard

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Foreword

This document (prEN xxxxxx) has been prepared by project HORIZONTAL, the management of standards is held by BT/TF 151.

This document is currently submitted for consultation to CEN and ISO bodies.

This European Standard has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries. This European Standard was prepared by project HORIZONTAL with support of BT/TF151.

Explanation about development of horizontal standards

This European Standard received approval from the CEN Technical Board on …….(date).

This European Standard was elaborated on the basis of:

PrEN 14405

This European Standard specifies an up-flow percolation test to determine the leaching behaviour of granular materials under standardized percolation conditions.

For a more complete characterization of the leaching behaviour of materials under specified conditions the application of other test methods may be required (see ENV 12920). For informative references see the Bibliography.

Contents

Introduction

In various European countries tests have been developed to characterize and assess the constituents that can be released from materials. The release of soluble constituents upon contact with water is regarded as a main mechanism of release, which results in a potential risk to the environment during the re-use or disposal of materials. These tests are intended to identify the leaching properties of materials. The complexity of the leaching process makes simplifications necessary. Not all of the relevant aspects of leaching behaviour can be addressed in one standard.

Tests to characterize the behaviour of materials can be divided into three categories. The relationships between these tests are summarized below:

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

"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 characterization tests.

"On-site verification" tests are used as a rapid check to confirm that the material is the same as 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 (1): basic characterization tests.

1   Scope

This European Standard is applicable to determine the leaching behaviour of inorganic constituents from granular material (without or with size reduction (see **6.2**)). The material body is subjected to percolation with water as a function of liquid to solid ratio under specified percolation conditions. The material is leached under hydraulically dynamic conditions. The method is a once-through column leaching test and the test results establish 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.

> NOTE 1 The mentioned specified percolation conditions are arbitrary and are not simulating a specific scenario.
>
> NOTE 2 Materials that show a saturated hydraulic conductivity between 10^{-7} m/s and 10^{-8} m/s can be subjected to this test, but it may be difficult to maintain the imposed flow rate. If a material shows a saturated hydraulic conductivity below 10^{-8} m/s, the test should not be carried out. (See Annex C5 for a definition of 'hydraulic conductivity'.)
>
> NOTE 3 This procedure is generally not applicable to biologically degrading materials and materials reacting with the leachant, leading, for example, to excessive gas emission or excessive heat release.
>
> NOTE 4 This procedure is applicable to materials showing solidification in the column, if the final hydraulic conductivity is within the specified range (see note 1).

2   Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Adjustment needed to appropriate documents for other fields

EN-ISO 5667-3:1995, *Water quality – Sampling – Part 3: Guidance on the preservation and handling of samples (Revision of ISO 5667-3:1985)*

prEN 12506:2000, *Characterization of waste – Analysis of eluates – Determination of pH, As, Cd, Cr^{VI}, Cu, Ni, Pb, Zn, Cl^-, NO_2^-, SO_4^{2-}*

ENV 12920:1998, *Characterization of waste – Methodology guideline for the determination of the leaching behaviour of waste under specific conditions*

prEN 13370:2001, *Characterization of waste – Analysis of eluates – Determination of Ammonium-N, AOX, conductivity, Hg, phenol index, TOC, CN^- (easily liberatable), F*

Draft prEN, WI 292001, *Characterization of waste – Sampling of liquid and granular materials including paste-like materials and sludges – Part 1: A framework for sampling plan preparation*

prEN 14346, *Characterization of waste – Calculation of dry matter by determination of dry residue and water content*

3   Terms and definitions

For the purposes of this European Standard the following terms and definitions apply:

3.1
dry residue w_{dr}
remaining mass fraction in percent of a sample after a drying process at 105 ^{o}C

[prEN 14346]

3.2
eluate
solution obtained by a laboratory leaching test

3.3
equilibrium condition
condition achieved when the pH deviation during a checking period is below a specified value (see **7.4**)

3.4
granular material
solid material that is neither monolithic, liquid, gas nor sludge

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

[IUPAC, 1997]

3.6
leachant
liquid that is brought into contact with the test portion in the leaching procedure

3.7
L/S ratio
ratio between the amount of liquid (L) and of solid (S) in the test

NOTE   L/S is expressed in l/kg dry matter

3.8
sample
quantity that is representative of a certain larger quantity

3.9
test portion
amount or volume of the test sample taken for testing or analysis, usually of known weight or volume

3.10
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

[IUPAC, 1997]

4   Principle

This standard describes a method to determine the release of constituents from material, with or without size reduction, packed in a column with a leachant percolating through it. A continuous vertical up-flow is used, so that the column is water saturated. The test conditions, including the flow rate of the leachant, enable a conclusion to be drawn from the results as to 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 to be tested is packed in a column in a standardized manner. 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 that are characterized physically and chemically according to existing standard methods. In the test, equilibrium conditions at the outlet of the column are verified after an equilibration period by measuring a pH deviation.

The results of the test are expressed as a function of L/S ratio, in terms of both mg of the constituents released per litre of eluate, and mg of the constituents released cumulatively per kg of *material* (dry matter).

5   Equipment and reagents

The materials and equipment specified in **5.2** to **5.15** shall be checked before use for proper operation and absence of interfering elements that may affect the result of the test (see **7.7**).

The equipment specified in **5.3**, **5.4, 5.10** and **5.13** shall also be calibrated.

5.1
Column made of glass or plastics (e.g. PMMA, PTFE) with an internal diameter of 5 cm or 10 cm and a filling height of about 30 cm ± 5 cm, fitted with filters (see **5.8**) in bottom and top section. In the top and bottom sections 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   An illustration of the column and its accompanying equipment is given in annex A.

5.2
Adjustable volumetric pump (peristaltic or equivalent). For small columns, running at a linear velocity of 15 cm/day, the capacity must be adjustable between 10 ml/h and 20 ml/h and for wide columns, running at the same linear velocity, the capacity must be adjustable between 40 ml/h and 60 ml/h.

5.3
Analytical balance with an accuracy of at least 0,1 g.

5.4
Conductivity meter with an accuracy of at least 0,1 mS/m.

5.5
Crushing equipment: a jaw crusher or a cutting device.

5.6
Demineralized water with a conductivity of a maximum of 0,1 mS/m.

5.7
Glass or plastics (e.g HDPE / PP / PTFE / PET) bottles with an appropriate volume, and with screw cap, for eluate collection and preservation of eluate samples (rinsed in accordance with EN-ISO 5667-3).

5.8
Membrane filters for in-line or off-line filtration of the eluates, with a pore size of 0,45 μm.

5.9
Nitric acid (pro analyse), 1 mol/l.

5.10
pH meter with an accuracy of at least 0,05 pH units.

5.11
Plastic hose material.

5.12
Pre-filters for the column with a pore size of 1,5 μm to 8 μm.

NOTE   If the eluate is to be used for bio-assays or other specific situations, in which fine particles are important, pre-filters with a larger pore size should be used.

5.13
Redox potential meter (optional).

5.14
Sample splitter for sub-sampling of laboratory samples (optional).

5.15
Sieving equipment with sieves of 4 mm and 10 mm nominal screen size.

6   Sample pre-treatment

6.1 General

Sampling shall be performed in accordance with prEN WI 292001 or a standard, derived from prEN WI 292001, in order to obtain a representative sample. Sample pre-treatment shall consist of sample preparation, taking a test portion and determining the dry residue of the test sample.

NOTE   If it is the intention to determine the leaching behaviour of a *material*, including ageing effects, the test portion should be representative for the material in the practical situation, also as far as ageing conditions is concerned.

6.2 Sample preparation

A laboratory sample shall be obtained of at least 2,5 kg of the material if a small column (diameter 5 cm) is to be used and of at least 10 kg, if a wide column (diameter 10 cm) is to be used. The laboratory sample shall be stored in closed packages and kept under conditions, which prevent any alterations of the *material*.

NOTE 1 Storage at low temperatures (4 °C) may be required, in order to minimise unwanted alterations of the *material*.

To investigate whether the material has to be crushed and to investigate which type of column is to be used, the laboratory sample shall be sieved (see **5.15**).

The use of the small or wide column shall depend on the particle size of the material, according to table 1.

Table 1: Use of column type, related to particle size of material

Fraction < 4 mm	Fraction ≥ 10 mm	Column to be used (with required size-reduction)
≥ 95% (*m/m*)		Small column (without size reduction), or Wide column (without size reduction)
80 – 95% (*m/m*)	≤ 5% (*m/m*)	Small column (with size reduction of the fraction ≥ 4 mm), or Wide column (without size reduction)
≤ 80% (*m/m*)	≤ 5% (*m/m*)	Wide column (without size reduction)
	> 5% (*m/m*)	Wide column (with size reduction of the fraction ≥ 10 mm

Prepare a test sample. Use a sample splitter (see **5.14**) or apply coning and quartering to split the laboratory sample.

The test shall be carried out preferably on a sample in the condition (particle size, moisture content) as it was delivered to the laboratory.

If oversized material is to be size reduced (in accordance with table 1), the entire oversized fraction shall be crushed with a crushing device (see **5.5**). On no account shall the material be finely ground. Non-crushable material (e.g. metallic parts such as nuts, bolts, scrap) in the sample shall be separated and the mass and the nature of the material shall be recorded.

If the sample cannot be sieved or split or crushed because of its moisture content, it is allowed to dry the sample, but no further than is required for the sample preparation. The drying temperature shall not exceed 40 °C.

NOTE 2 Depending on the maximum particle size, the splitting may require size reduction of the coarser particles to comply with the rules of sampling.

NOTE 3 Due to crushing and sieving, contamination of the sample may occur to an extent, which is affecting the release of some constituents of concern, e.g. Co and W from tungsten carbide crushing equipment or Cr, Ni, Mo and V from stainless steel equipment.

NOTE 4 Fibrous materials and plastics can often be size-reduced only after cryogenic treatment.

NOTE 5 Drying may lead to oxidation and/or carbonation. If the material is fresh and has to be tested as a non oxidized / non carbonated material, the drying should be conducted in an inert atmosphere.

6.3 Test portion

At least two test portions shall be taken from the test sample. To fill the column completely, the quantity of one of the test portions shall be such that its volume after compaction is 0,6 l (in case a column with a diameter of 5 cm is used), or 2,4 l (in case a column with a diameter of 10 cm is used). A sample splitter (see **5.14**) shall be used or coning and quartering shall be applied to split the test sample and prepare the test portion(s).

6.4 Determination of dry residue

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

Determine the dry residue (w_{dr}) at 105 ^{o}C ± 5 ^{o}C according to prEN 14346, on a separate test portion. Calculate the dry residue in accordance with equation 1.

$$w_{dr} = m_d * 100 / m_r \qquad [\%] \tag{1}$$

where:

w_{dr} dry residue of the material [%]

m_r mass of the undried test portion [g]

m_d dry mass of the test portion [g]

7   Procedure

7.1 Temperature

Carry out the up-flow percolation test at a temperature of 20 °C ± 5 °C.

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

7.2 Preparation

Rinse the column, including top and bottom sections and filters (see **5.1**) and bottles (see **5.7**) with nitric acid (see **5.9**) and water (see **5.6**) consecutively. Weigh the dry column, including top and bottom sections, filters and filter plates or layers of fine-grained material, to an accuracy of 1 g.

7.3 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 (see **5.12**) to the column. Fill the column with the test portion, up to a bed height of 30 cm ± 5 cm, in at least five consecutive layers, as follows:

▪ 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 case of a column with a diameter of 5 cm, and of 500 g in case of a column with a diameter of 10 cm. Drop the weight three times on each layer falling 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 1).

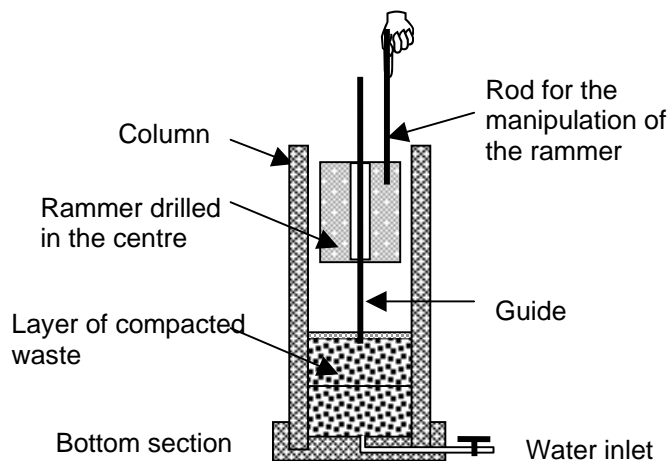▪ For the last layer, check the remaining height and adjust the necessary mass in order to get 30 cm ± 5 cm.

Figure 1 – Filling and packing of the column

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 - 8 cm layer in the column, pack it and calculate the mass necessary to get 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 in that specific cases.

Fit the top section of the column, 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 (see **5.12**), to prevent entrainment of fine particles with the eluate. Fit the top section and pre-filter so that the liquid flow cannot bypass the filter and so that no open space (dead volume) is left above the material.

NOTE 4 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_0) of the test portion in the column, in accordance with equation 2.

$$m_0 = (m * w_{dr}) / 100\% \tag{2}$$

where:

m_0 dry mass of the test portion in the column [kg]

m mass of the (moist) test portion in the column [kg]

w_{dr} dry residue [%]

Fit the outlet-hose to the top section of the column and to the in-line filter (see **5.8**), in case an in-line filter is used.

7.4 Start-up of the test

Saturate the column with water (5.6) either by using the pump (see 5.2) or by hydrostatic pressure (see B.3).

NOTE 1 See annex B for a more detailed description of the methods of saturation.

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

NOTE 2 To facilitate equilibration and the procedure of checking the equilibrium conditions, recirculation of eluate may be applied. See annex B.

After the equilibration period, start the pump (again) and set the flow rate such that the linear velocity is 15 cm/day ± 2 cm/day through the empty column. Calculate the flow rate in accordance with equation 3.

$$\phi = v_L * \pi * d^2 * 0,0104 \tag{3}$$

where:

ϕ leachant flow rate [ml /h]

v_L linear velocity of the leachant through the empty column [cm/day]

d diameter of the column [cm]

NOTE 3 A linear velocity of 15 cm/day corresponds with 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).

Collect a first small portion of eluate, just to measure the pH (but 15 ml at the most). Measure the pH (see **5.10**) of this portion of eluate and make a note of it. Keep the eluate portion.

NOTE 4 It is essential that the pH-electrode and the beaker, in which the small eluate portion is collected and measured, are thoroughly rinsed before use, in order not to contaminate the small eluate portion. This portion will be kept and added to the eluate at a later stage for analysis.

Connect the outlet hose to an eluate collection bottle of appropriate size (see **5.7**). If the pH value of the small eluate portion was higher than 9, or if a later rise in pH is expected, keep the eluate collection bottle and the following ones under inert (argon or nitrogen) atmosphere, to prevent carbonation and precipitation. Refill gas every time the collection bottle is changed.

NOTE 5 A water lock will be needed on the lid of the collection bottle in case an inert atmosphere is applied, to avoid back pressure in the column in case the bottle with lid is gas tight.

NOTE 6 Keeping the collection bottles under inert atmosphere may also be required when investigating reducing materials, to prevent the occurrence of oxidation reactions.

Start the pump again and change the collection bottle after a quantity of $(0,1 \pm 0,02) * $ the mass of the test portion (m_0) of leachant has passed through, the small portion of eluate that was used for pH measurement inclusive. Measure the pH of this second eluate portion (see **5.10**) and make a note of it.

If the pH values measured in the first and second portion of eluate do not deviate more than 0,5 pH unit, continue the test with combining the two small eluate portions. This shall be regarded to be the first eluate fraction, collected at a liquid to solid ratio (L/S ratio) of 0,1 l/kg dry matter. If the equilibrium condition (pH deviation < 0,5 pH unit) is not met, report this fact and consider the "circulation method" of equilibration and/or the other guidelines, as described in annex B4.

Filter the eluate fraction off-line over a 0,45 µm membrane filter (see **5.8**), in case no in-line filter was used.

7.5 Collection of additional eluate fractions

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

Because of material-specific situations, it may be difficult to maintain the original linear velocity (in the range of 15 cm/day ± 2 cm/day). Though, the test result of a specific eluate fraction is regarded to be in accordance with the standard if the average velocity over the total L/S range of that eluate fraction is at least 10 cm/day, and the actual velocity at the end of the collection period of that fraction is at least 5 cm/day.

NOTE 1 A more detailed explanation on flow rate and linear velocity is given in Annex C.

Use new collection bottles as soon as a quantity of eluate according to Table 2 has passed through.
These are fractions 2 to 7. Mind that both criteria (concerning the volume of the actual eluate fraction as well as the cumulative L/S ratio) must be fulfilled. Filter each eluate fraction off-line over a 0,45 µm membrane filter (see **5.8**), if no in-line filter was used.

 At each eluate collection moment measure 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 2 — Table for collection of eluate fractions

Fraction number	Fraction volume [l] (= L/S ratio times dry mass)[1]	Cumulative L/S ratio [l/kg dry matter]
1	$(0,1 \pm 0,02) * m_0$	$0,1 \pm 0,02$
2	$(0,1 \pm 0,02) * m_0$	$0,2 \pm 0,04$
3	$(0,3 \pm 0,05) * m_0$	$0,5 \pm 0,08$
4	$(0,5 \pm 0,1) * m_0$	$1,0 \pm 0,15$
5	$(1,0 \pm 0,2) * m_0$	$2,0 \pm 0,3$
6	$(3,0 \pm 0,2) * m_0$	$5,0 \pm 0,4$
7	$(5,0 \pm 0,2) * m_0$	$10,0 \pm 0,1$

[1] In the case of high salt loads (conductivity > 7500 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 has to 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 2 For specific scenario's (for example a landfill with top cover) it may be sufficient to know the leaching characteristics up to an L/S ratio of 2 l/kg dry matter. In that case the test can be stopped after the collection of the fifth eluate fraction.

NOTE 3 In case no automated eluate collection apparatus is available, a collection scheme can be composed within the ranges that are allowed in linear velocity (15 cm/day ± 2 cm/day) and in fraction volume (see Table 2) that enables eluate collection within working hours.

NOTE 4 The execution time of the test can be calculated from equation 4.

$$t = (L/S * m_0 * 1000) / (24 * \phi) \tag{4}$$

where:

t execution time of the test [days]

L/S   final liquid to solid ratio [l/kg dry matter]

m_0 dry mass of the test portion [kg]

ϕ leachant flow rate [ml /h]

NOTE 5 If the length of the column is 30 cm, the linear velocity is 15 cm/day and the bulk density of the material is 1500 kg/m^3, the execution time of the test is approximately 30 days for L/S = 10 l/kg.

7.6 Further preparation of the eluates for analysis

Measure the pH (see **5.10**) in accordance with prEN 12506 and conductivity (see **5.4**) in accordance with prEN 13370 (and optionally redox potential E_h in mV (see **5.13**)) of each eluate fraction, just after collection. If the conductivity of an eluate fraction exceeds 7500 mS/m, measure the volume (or density) of the eluate and dilute the eluate until the conductivity is below 7500 mS/m. Ensure that the dilution factor is no more than 10.

NOTE 1 If a small column (with a diameter of 5 cm) is used, it may be desirable to dilute the first eluate fractions to get enough eluate to carry out all analyses. The dilution factor for this purpose should be no more than 4. The dilution factor should be reported.

Divide the eluates into an appropriate number of sub-samples for different chemical analysis, after shaking well. Preserve the eluate sub-samples depending on the elements to be analysed, according to EN-ISO 5667-3. Store the (sub) eluates in sealed bottles, according to EN-ISO 5667 - 3.

Determine the concentrations of constituents of interest using the methods of eluate analysis (e.g. prEN 12506 and prEN 13370). Also measure the total concentration of Dissolved Organic Carbon (DOC) using prEN 13370.

7.7 Blank test

In order to check, as far as possible, how the whole procedure is performed, carry out blank tests on a regular basis. Submit two volumes of leachant to the whole procedure (except the sample pre-treatment). Start the pump, until the empty column, complete with top and bottom sections and with tubing, is filled with leachant. After two days disconnect the pump and the column, empty the column via the bottom section and collect the first blank eluate. Connect the pump again, and fill the empty column and fittings with new leachant. After two other days empty the column in the same way and collect the second blank eluate. Measure the conductivity (see **5.4**) of these blank eluates. Preserve and analyse both blank eluates in accordance with **7.6**.

If the test is performed for comparison with any regulatory standards, these standards shall dictate the criteris the analytical results of the blank test have to fulfil. Recalculate the standard values to concentration values, corresponding with an L/S ratio of 1 l/kg.

If the test is not performed for regulatory purposes, the average concentrations in the first 4 eluate fractions (up to a cumulative L/S ratio of 1 l/kg) of the test actually performed, form the criteria.

Ensure that the eluates of this blank test fulfil the following minimum requirements:

- in the first eluate of the blank test the concentration of each considered element shall be less than 10% of the relevant criterion, mentioned before;

- the conductivity of the second blank eluate shall be < 0,2 mS/m.

If these requirements are not fulfilled, reduce the contamination.

8   Calculations

For each component the quantities released in all eluate fractions shall be calculated as in equation 5.

$$U_i = (V_i * c_i) / (m_0) \tag{5}$$

where:

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

U_i released quantity of a component per quantity of sample for analysis in eluate fraction i [mg/kg dry matter]

V_i volume of the eluate fraction i [l]

c_i concentration of the component concerned in the eluate fraction i [mg/l]

m_0 dry mass of the test portion in the column [kg]

The concentration c_i referred to in equation 5, shall be the concentration originally present in the eluate. The measured value determined in accordance with **7.5** shall be corrected if the eluate fraction has been diluted and/or if the quantity of preservation fluid added in **7.5** 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 <u>upper limit</u> of U_i shall be calculated by making c_i equal to the limit of detection; the <u>lower limit</u> of U_i shall be calculated by making c_i equal to 0.

For each component the cumulatively released quantity (ΣU_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 <u>upper limit</u> and the <u>lower limit</u> of ΣU_i.

9   Test report

The report shall contain at least the following data:

a)   General

— reference to the present standard;

— date of receipt of the laboratory sample;

— sampling report, according to 6.1.;

— a complete identification of the laboratory sample;

— ageing conditions (if relevant);

— pre-treatment (e.g. method of size-reduction, drying, sub-division) and storage conditions.

b)   Leaching test conditions

— date of the test (beginning and end);

— dry residue of the test sample (w_{dr});

— dry mass of the test portion in the column (m_0);

— compaction method;

— identification of the test equipment and instruments used, including the diameter of the column (5 cm or 10 cm);

— actual flow rate during the course of the test, and any significant changes in flow rate;

— cumulative L/S ratios of the eluate fractions collected;

— temperature range during the performance of the test;

— the pH values, measured during the start-up of the test, after the equilibration period;

— any deviation from the test method and the reason of this deviation together with all circumstances that have influenced the results.

c)   Analytical report

— pH and conductivity of the eluate fractions collected;

— any dilutions that were carried out;

— methods of preservation of the eluate fractions for the different elements to be analysed, in accordance with **7.6**.;

— quantities of preservation fluid added in accordance with **7.6** where this is more than 1 ml per 100 ml eluate;

— analytical report, in accordance with **7.6.**;

— all measured concentrations.

d)   Results of the leaching test

— the measured concentrations per fraction for each component, in mg/l, preferably plotted against the L/S ratio;

— the released quantities U_i calculated per fraction for each component, in mg/kg dry mass; for analysis results below the limit of detection for the components concerned both the lower limit and the upper limit of the released quantity shall be indicated;

— the cumulatively released quantities ΣU_i calculated for each component, in mg/kg dry mass. Again, if relevant, lower and upper limits shall be indicated.

10 Test performance

The performance of the test regarding repeatability and reproducibility is dependent on the tested material and also on the testing conditions. When the standard was adopted by CEN, the test specified in this standard was not validated and no data where available on robustness, repeatability and reproducibility.

Annex A
(informative)

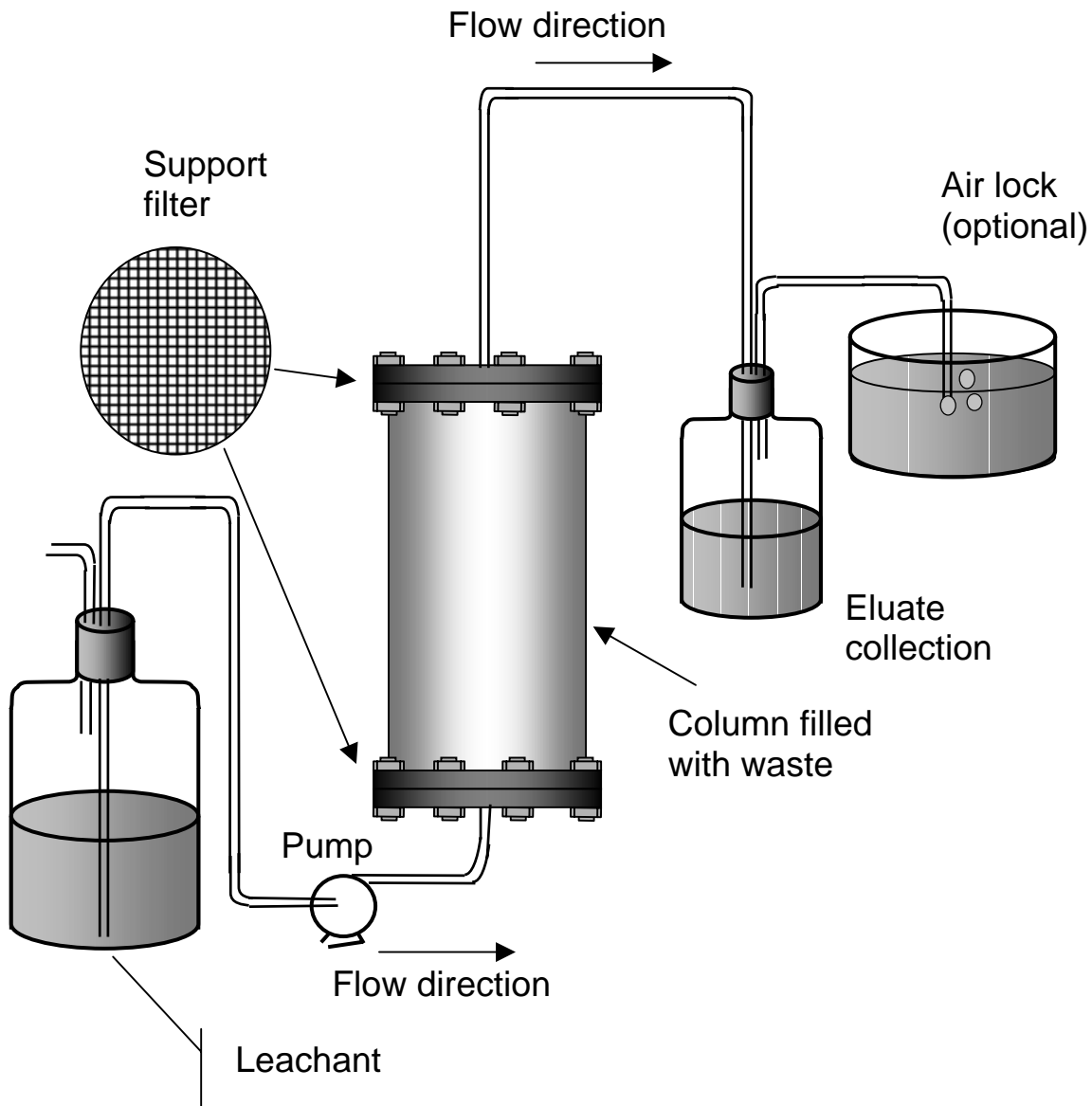## Illustration of the column and accompanying equipment

Flow direction

Support filter

Air lock (optional)

Eluate collection

Column filled with waste

Pump

Flow direction

Leachant

Figure A.1 - Example of a column and accompanying equipment

Annex B
(informative)

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

B.1   General

This annex contains suggestions on how to fill and pack the column in 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.

B.2   Guidelines on column filling and packing

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

— 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 moisture content must be known, in order to be able to determine the dry mass of the test portion in the column (in section 7.3).

— Materials may 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 40 °C. Drying may lead to oxidation and/or carbonation. If the material is fresh and has to be tested as a non oxidized / non carbonated material, the drying should be conducted in an inert atmosphere.

— In other cases it may 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 may cause problems later, even if 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.

B.3   Water saturation

In section 7.4 two methods are mentioned to saturate the packed column with leachant: by using the pump (1) or by using initial hydrostatic pressure (2).

1: Saturating the column by using the pump

Connect the pump (see **5.2**) to the bottom section of the column and pass demineralized water (see **5.6**) through the column from bottom to top (with a linear velocity of the leachant through the empty column of approximately 15 cm/day). Stop the pump when the material in the column is all saturated, but the outlet hose is still empty.
.

2: 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 done under an initial hydrostatic pressure of 30 cm (as is shown in figure B.1).

The hydrostatic pressure should be stopped when the material in the column is all saturated, but the outlet hose is still empty.
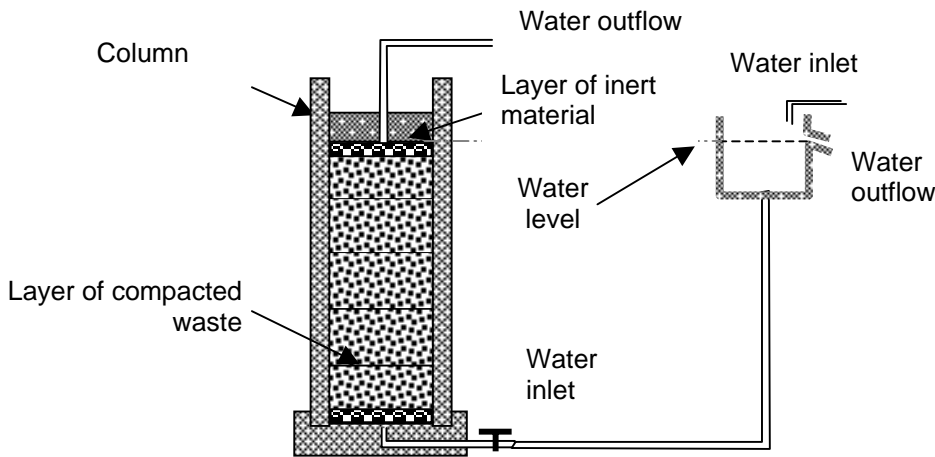

Figure B.1 – Saturation of the column by initial hydrostatic pressure

B.4   Equilibration and checking of equilibrium conditions

An alternative procedure to establish and control equilibrium conditions during the start-up of the percolation test may be used:

— Connect the inlet of the pump and the outlet of the column to a bottle containing a volume of leachant corresponding to a liquid to solid ratio of 0,1 (as is visualised in figure B.2).

— Circulate the leachant in closed loop until the pH equilibrium condition is reached (pH deviation in the bottle $\leq 0,5$ over a period of least 8 h, e.g. between $t\,0 + 64$ h and $t\,0 + 72$ h). If the equilibrium condition is not met after three days, extend the equilibration period to four days, and check again.

— Remove the solution in the bottle as the first eluate (L/S 0,1).

— Connect the inlet of the pump to the leachant supply and the outlet of the column to a new bottle for collection of the second eluate fraction.
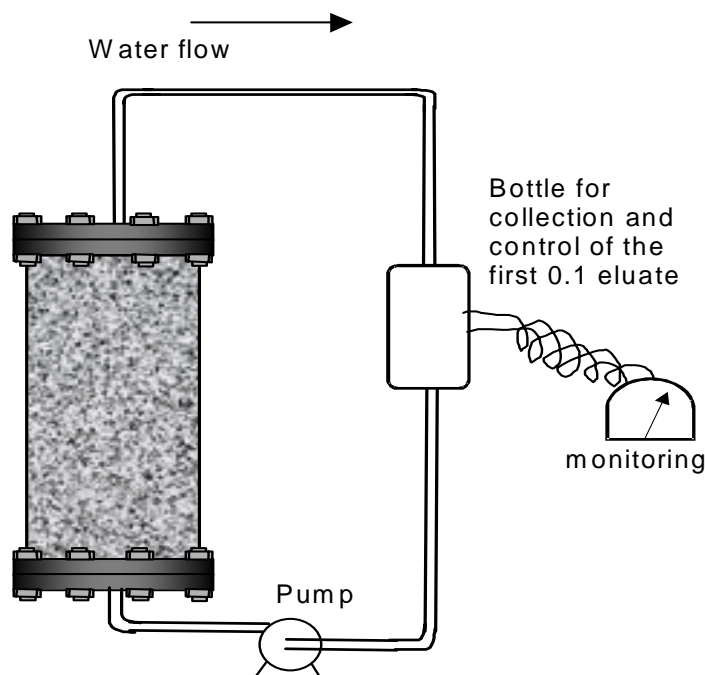
Figure B.2 — Apparatus for alternative procedure to establish and control equilibrium conditions during start-up

If the equilibrium condition during the start-up of the percolation test (as described in **7.4**) is not met, the following actions may be taken. A more detailed explanation on equilibrium conditions is given in Annex C1.

- There may be no equilibrium yet. The procedure may be performed again, waiting for a longer period than three days, or using the alternative equilibration procedure (as described above).

- The material may be highly reactive, and the difference in pH is caused by the production of acid gases during equilibration. The first eluate fraction is influenced by these acid gases, and the release of this fraction should be put between brackets. This should be stated in the report.

- It may be unclear what the reason is for the pH deviation. In that case there are two options. Either make a statement in the report and continue the test, or abandon the test.

If a final eluate in equilibrium is needed two options are possible:

1. Continue percolating to get a last volume of percolate corresponding to a L/S ratio of 0,1. Then put the inlet hose in the collection bottle: the solution should circulate again in the column until equilibrium is reached;

2. Leave the saturated material for a minimum period of three days in order to equilibrate the system and remove the interstitial water if necessary under a vacuum or a pressure of nitrogen.

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Annex C
(informative)

Background for the choices made in developing this percolation test

C.1   Introduction

This test has been developed within the scope of the European Standard ENV 12920 that specifies a methodology for the determination of the leaching behaviour of material under specified conditions. This methodology aims at determining the influence of chemical, physical, geotechnical, mechanical and biological parameters on the release of inorganic constituents from material bodies in the scenario under consideration. ENV 12920 distinguishes between parameter specific tests which are intended for measuring intrinsic properties of material or the effects of specific parameters on the release of inorganic constituents from material bodies and simulation tests which are intended for simulating the combined effects of several parameters on the release.

During the development of this test procedure it has been 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 *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 has been 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. Only a verification of the initial equilibrium[2] in the column before the start up of the pump is mandatory.

Test users with specific test needs, e.g. to ensure that the local equilibrium assumption is fulfilled, are hence referred to the C.5 or the standard: "Characterization of waste – Leaching behaviour tests – Percolation test for simulating conditions of specific scenarios" (WI 292035, in preparation by CEN/TC 292/WG 6), which allows a freer choice of test conditions to fit specific purposes and requirements.

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.

The filling and packing of the column have already been discussed in detail in annex B.

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[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.

[2] Initial equilibrium is referred to as a condition of chemical equilibrium between the solid phase and the leachant at the start of the test. This applies to the full length of the column and is checked at the beginning of the test.

C.2 Particle size/particle size distribution

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 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 table 1 in § **6.2** and **C.3**). 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

C.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/day to 15 cm/day if the length of the column is at least 3 to 4 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 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 cm $\pm$ 5 cm. The conditions prescribing which column size to be used for a particular sample of material has been presented in the table in section **6.2** of the standard.

C.4 Flow mode (up-flow/down-flow)

This standard is intended to describe the leaching properties of granular *material*s 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.

C.5 Flow rate of the leachant

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/day (cm/24 hrs), through the empty column. This is convenient since the equilibrium / non- equilibrium conditions in the *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 comparable to the rate of infiltration of precipitation into a landfill or a utilisation scenario of *material*s.

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 *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 a given *material* with a given particle size distribution there should in principle exist an upper limit for the leachant flow rate, below which the local equilibrium assumption is always fulfilled. This can be used to check the equilibrium situation: If similar columns with identical *material*s are run at two different leachant flow rates and the results are similar, the local equilibrium requirement is likely to have been fulfilled at both flow rates.

For this test, a linear leachant velocity of 15 cm/day has been fixed. This enables the test to be carried out to a final L/S = 10 l/kg in approximately 30 days and to reach L/S = 2 l/kg within approximately one week. Test results indicate that the local equilibrium condition appear 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 cm/day $\pm$ 2 cm/day) when applying this flow rate compared to the some times very low flow rates observed in field conditions.

Some (fine-grained) *material*s 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 ^{o}C.) If it is deemed necessary or desirable to apply flow rates lower than 15 cm/day, this will be a deviation from the standard procedure and must be mentioned in the test report. Alternatively, the standard: "Characterization of waste – Leaching behaviour tests – Percolation test for simulating conditions of specific scenarios" (WI 2292035 in preparation by CEN/TC 292/WG 6), which allows adjustment of flow rates and other test conditions to specific needs, could be applied.

C.6   L/S ratio and eluate fractions collected/duration of the test

The following eluate fractions are collected: L/S = 0,0-0,1 l/kg, 0,1-0,2 l/kg, 0,2-0,5 l/kg, 0,5-1,0 l/kg, 1,0-2,0 l/kg, 2,0-5,0 l/kg and 5,0-10 l/kg. The size of the fractions are 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 – 1 l/kg or 0 – 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 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 material with a density of 1 ton/m^3 through which water (e.g. infiltrating rainwater) is percolating at a rate of 200 mm/year, a 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 material 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 1000 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.

C.7   Nature of the leachant

For most material types the composition of the eluate at L/S values below 10 l/kg is controlled predominantly by the composition of the material. 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 standard.

For specific purposes it may be desirable to use other leachants than de-mineralised water. If this is done, it will be a deviation from the standard procedure and it must be mentioned in the test report. Alternatively, the standard: "Characterization of waste – Leaching behaviour tests – Percolation test for simulating conditions of specific scenarios" (WI 292035 in preparation by CEN/TC 292/WG 6), which allows adjustment test conditions to specific needs, could be applied.

C.8   Temperature

Equilibrium reactions are temperature-dependent and care should therefore be taken to ensure an leachant/eluate temperature as constant as possible and as close as possible to the prescribed value (20 °C $\pm$ 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 °C $\pm$ 5 °C, has been chosen to represent common indoor conditions and to be consistent with the temperature requirements of EN 12457 part 1-4.

D.9   Beneficial use of the test results

The test results can be used in predicting the release of constituents from a material in a specific scenario, as a function of time, by means of modelling under the hydraulic, geotechnical, hydrological, chemical, physical and biological conditions of the scenario. As a part of a complete characterization of the leaching behaviour of material under specified conditions this test can generally not be used alone: the application of other test methods may be required (see ENV 12920).

Under certain conditions the test may also provide information on pore water concentrations in scenarios in which material and aqueous solutions are in equilibrium, both in the short- and long-term (after wash-out of the salts) if the modelling step of ENV 12920 is applied.

Results from the up-flow percolation test may be comparable to those obtained by carrying out the batch compliance test EN 12457 part 1-4 either at L/S=2 of at L/S=10 l/kg. However, users have to be careful, as the sequence of leaching events in the dynamic percolation test may differ from the static conditions in a batch test such as EN 12457 part 1-4, which may lead to different end results.

Bibliography

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

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