BS EN 14405:2017

BSI Standards Publication

Characterization of waste — Leaching behaviour test — Up-flow percolation test (under specified conditions)

National foreword

This British Standard is the UK implementation of EN 14405:2017. It supersedes DD CEN/TS 14405:2004 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

Published by BSI Standards Limited 2017

ISBN 978 0 580 87336 2

ICS 13.030.10

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2017.

Amendments/corrigenda issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14405

March 2017

ICS 13.030.10

Supersedes CEN/TS 14405:2004

English Version

Characterization of waste - Leaching behaviour test - Up-flow percolation test (under specified conditions)

Caractérisation des déchets - Essais de comportement à la lixiviation - Essai de percolation à écoulement ascendant (dans des conditions spécifiées)

Charakterisierung von Abfällen - Untersuchung des Elutionsverfahrens - Perkolationsprüfung im Aufwärtsstrom (unter festgelegten Bedingungen)

This European Standard was approved by CEN on 3 February 2017.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 14405:2017 E

Contents

Page

European foreword

This document (EN 14405:2017) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2017, and conflicting national standards shall be withdrawn at the latest by September 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 14405:2004.

The following significant technical changes have been implemented in this new edition of the text:

- the status of the document has been changed from Technical Specification (CEN/TS) into a European Standard;
- based on CEN ISO/TS 21268-3 and CEN/TS 16637-3 the option for analysis of non-volatile organic substances has been added;
- performance data for inorganic substances has been added (see Annex D).

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association

This European Standard has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries.

This European Standard was elaborated on the basis of:

- NEN 7343:1995;
- NT ENVIR 002:1995.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This European Standard specifies an up-flow percolation test to determine the leaching behaviour of inorganic and non-volatile organic substances from granular waste materials under standardized percolation conditions.

NOTE Validation data for non-volatile organic substances are not currently available, but will be added on revision when available.

For the complete characterization of the leaching behaviour of waste under specified conditions the application of other test methods is required (see EN 12920).

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples can contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction.

Consequently these samples should be handled with special care. Gases which can be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations will be followed with respect to all hazards associated with this method.

In the different European countries, tests have been developed to characterize and assess the substances which can be leached from waste materials. The release of soluble substances upon contact with water is regarded as one of the main mechanism of release which results in a potential risk to the environment during life-cycle of waste materials (disposal or re-use scenario). The intent of these tests is to identify the leaching properties of waste materials. The complexity of the leaching process makes simplifications necessary. Not all of the relevant aspects of leaching behaviour can be addressed in one single standard. Procedures to characterize the behaviour of waste materials can generally be divided into three steps, using different tests in relation to the objective. The following test hierarchy is taken from the Landfill Directive[1] and the Decision on Annex II of this Directive[2] for disposal of waste.

a) Basic characterization constitutes a full characterization of the waste by gathering all the necessary information for a safe management of the waste in the short and long term. Basic characterization may provide information on the waste (type and origin, composition, consistency, leachability, etc.), information for understanding the behaviour of waste in the considered management scenario, comparison of waste properties against limit values, and detection of key variables (critical parameters as liquid/solid (*L*/*S*) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters) for compliance testing and options for simplification of compliance testing. Characterization may deliver ratios between test results from basic characterization and results from simplified test procedures as well as information on a suitable frequency for compliance testing. In addition to the leaching behaviour, the composition of the waste should be known or determined by testing. The tests used for basic characterization should always include those to be used for compliance testing.

b) Compliance testing is used to demonstrate that the sample of today fits the population of samples tested before by basic characterization and through that, is used to carry out compliance with regulatory limit values. The compliance test should therefore always be part of the basic characterization program. The compliance test focuses on key variables and leaching behaviour identified by basic characterization tests. Parts of basic characterization tests can also be used for compliance purposes.

---

[1] Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste.

[2] Council Decision 2003/33/EC of 19 December 2002.

c) On-site verification tests are used as a rapid check to confirm that the waste is the same as that which has been subjected to characterization or compliance tests. On-site verification tests are not necessarily leaching tests.

The test procedure described in this document is a basic characterization test and falls in category a).

1 Scope

This European Standard is applicable for determining the leaching behaviour of inorganic and non-volatile organic substances from granular waste (without or with size reduction (see 7.2)). The waste body is subjected to percolation with water as a function of liquid to solid ratio under specified percolation conditions. The waste 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 waste and leachant. This test method produces eluates, which can subsequently be characterized by physical, chemical and ecotoxicological methods according to existing standard methods.

NOTE 1 The mentioned specified percolation conditions are arbitrary and are not necessarily simulating a specific scenario.

NOTE 2 Waste 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 can be difficult to maintain the imposed flow rate. If a waste shows a saturated hydraulic conductivity below 10^{-8} m/s, the test will preferably not be carried out (See C.5 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 2).

NOTE 5 It is not always possible to optimise test conditions simultaneously for inorganic and non-volatile organic substances and optimum test conditions may also vary between different groups of non-volatile organic substances. Test requirements for non-volatile organic substances are generally more stringent than those for inorganic constituents. The test conditions suitable for measuring the release of non-volatile organic substances will generally also be applicable to inorganic substances.

NOTE 6 For ecotoxicological testing, eluates representing the release of both inorganic and non-volatile organic substances are needed. In this standard ecotoxicological testing is meant to include also genotoxicological testing.

NOTE 7 Validation data for non-volatile organic substances are not currently available, but will be added on revision when available.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14346, *Characterization of waste - Calculation of dry matter by determination of dry residue or water content*

EN 14899, *Characterization of waste - Sampling of waste materials - Framework for the preparation and application of a Sampling Plan*

EN 15002, *Characterization of waste - Preparation of test portions from the laboratory sample*

EN 16192, *Characterization of waste - Analysis of eluates*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 5667-3, *Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO 5667-3)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
dry residue
w_{dr}
remaining mass fraction of a sample after a drying process at 105 °C

[SOURCE: EN 14346:2006]

3.2
eluate
solution obtained by a leaching test

3.3
equilibrium condition
condition achieved when the pH deviation during a checking period is below a specified value

Note 1 to entry: See 8.4.

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

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

[SOURCE: IUPAC, 1990]

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, crushing, sawing, coring, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

Note 2 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

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

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

Note 1 to entry: 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 analysis, usually of known weight or volume

[SOURCE: IUPAC, 1990]

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

[SOURCE: IUPAC, 1990]

4 Principle

This standard describes a method to determine the release of substances from waste, 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 waste 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 procedure described in this standard is based on the more stringent test requirements for determining the release of non-volatile organic substances and/or for subsequent ecotoxicological testing. If only the release of inorganic substances is to be measured, less stringent requirements may be adapted for some steps of the procedure.

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

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Distilled water, demineralized water, de-ionized water or water of equivalent purity ($5 < pH < 7,5$) with a conductivity < 0,1 mS/m according to grade 2 specified in EN ISO 3696.

When the release of biodegradable organic compounds is studied, the leachant might be stabilized with a preservative in order to avoid biodegradation, e.g. sodium azide (NaN_3) at a concentration of 0,1 %.

For ecotoxicological tests, preservatives should not be used. Addition of preservatives may also affect the release of inorganic substances.

5.2 Nitric acid, $c(HNO_3) = 1$ mol/l.

5.3 Organic solvent, acetone, $(CH_3)_2CO$, pro analyse

6 Equipment

6.1 General

Check the materials and equipment specified in 6.2.2, 6.2.3 and 6.2.7 before use for proper operation and absence of interfering elements that may affect the result of the test (see 8.7).

Calibrate the equipment specified in 6.2.10 to 6.2.13.

6.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

6.2.1 Column

Column made of glass or plastics (e.g. PMMA, PTFE) with an internal diameter of 5 cm to 10 cm and a filling height of about 30 cm ± 5 cm, fitted with filters (see 6.2.6) in bottom and top section. In the top and bottom sections of the column (e.g. made of polychlorotrifluoroethylene (PCTFE)) a filter plate or a thin layer of fine-grained non-reactive material (e.g. fine quartz sand with a particle size range of 1,0 mm to 2,5 mm and SiO_2-content at least 98 % by mass) is applied to ensure proper water flow over the width of the column and as a support for the pre-filter.

Filter plates, quartz sand (SiO_2-content at least 98 % by mass) or glass beads can be washed but when heated to remove any sorbed organic contaminants they shall not be heated to red-heat. They shall also be low in substances (i.e. determinants) which could lead to increased levels in procedural blanks.

NOTE 1 An illustration of the column and its accompanying equipment is given in Annex A.

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

NOTE 3 In case only organic substances are analysed, stainless steel column and fittings can be applied.

6.2.2 Crushing equipment, jaw crusher or a cutting device.

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

6.2.4 Glass or plastic bottles, e.g. high density polyethylene (HDPE)/polypropylene (PP)/polytetrafluoroethylene (PTFE)/polyethyleneterephtalate (PET)

Use 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).

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

Membrane filters for the vacuum or pressure filtration device, fabricated from inert material, which is compatible with the waste. Filter shall be pre-rinsed with demineralized water or similarly clean in order to remove DOC. Filters shall not be used for eluates to be analysed for organic substances.

6.2.6 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 will preferably be used.

6.2.7 Volumetric pump

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

6.2.8 Tubing material

Tubing material inert and adapted to the substances to be analysed (see EN ISO 5667-3). In case only organic substances are to be analysed, stainless steel or glass tubings can be used in contact with the eluate. In case only inorganic substances are analysed PE, PP, PTFE, ETFE, FEP or similar tubing materials can be used.

6.2.9 Centrifuge

(1) Operating at 20 000 *g* to 30 000 *g* using centrifuge tubes of fluorinated ethylene propylene (FEP) or tubes of an alternative material, which is inert with regard to the substances to be measured and suitable to high speed centrifugation.

(2) Alternatively, if a high speed centrifuge is not available, a centrifuge operating at 2 000 *g* to 2 500 *g* using bottles of an appropriate material may be used in combination with increased centrifugation time. Cooling shall be applied to maintain the desired temperature.

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

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

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

6.2.13 Redox potential meter (optional).

7 Sample pretreatment

7.1 General

Sampling shall be performed in accordance with EN 14899 or a standard derived from EN 14899, in order to obtain a representative sample. Sample pretreatment 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 waste material, including ageing effects, the test portion will preferably be representative for the material in the practical situation, also as far as ageing conditions are concerned.

7.2 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 waste material.

NOTE 1 Storage at low temperatures (4 °C) may be required, in order to minimise unwanted alterations of the waste 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 6.2.3).

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 waste

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 % (m/m) to 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 according to EN 15002. Use a sample splitter or apply coning and quartering to split the laboratory sample as specified in EN 15002.

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 according to EN 15002). 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 (see EN 15002).

NOTE 2 Depending on the maximum particle size, the splitting can 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 can occur to an extent, which is affecting the release of some substances 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 will preferably be conducted in an inert atmosphere.

NOTE 6 Crushing, sieving and drying at ≥ 25 °C can lead to loss of semi-volatile components (inorganic and organic). In this case, particle size reduction and drying is best avoided.

7.3 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 shall be used or coning and quartering shall be applied to split the test sample and prepare the test portion(s) (see EN 15002).

7.4 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 °C ± 3 °C according to EN 14346, on a separate test portion. Calculate the dry residue in accordance with Formula 1.

$$w_{\mathrm{dr}} = \frac{m_{\mathrm{d}} \times 100}{m_{\mathrm{r}}} \qquad (1)$$

where

w_{dr} is the dry residue of the waste, in %;

m_{d} is the dry mass of the test portion, in g;

m_{r} is the mass of the undried test portion, in g.

8 Procedure

8.1 Temperature

Carry out the up-flow percolation test at a temperature between 19 °C to 25 °C.

NOTE A constant temperature 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.

8.2 Preparation

Rinse the column, including top and bottom sections and filters (see 6.2.5 and 6.2.6) and bottles (see 6.2.4) with nitric acid (see 5.2) and/or organic solvent (5.3) and water (see 5.1) 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. Alternatively, a heat treatment (e.g. between 450 °C and 500 °C) can be applied to clean temperature resistant equipment of sorbed organic contaminants. Quartz sand and filter layers shall not be heated to red-heat.

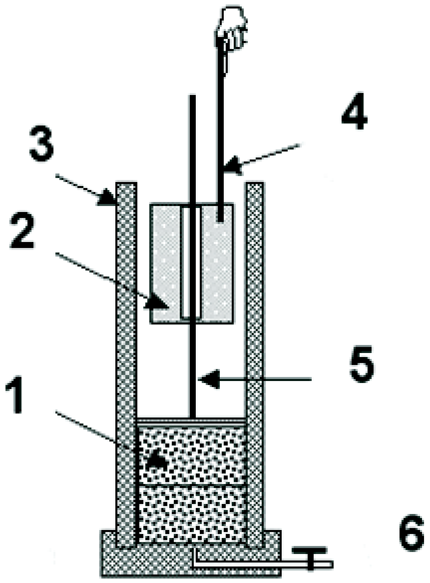
8.3 Packing of the column

Fit the bottom section, equipped with a filter plate or a layer of fine-grained chemically inert material (e.g. fine quartz sand) of approximately 1 cm and a pre-filter (see 6.2.6) 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.



Key

1	layer of compacted waste
2	rammer drilled in the centre
3	column
4	rod for the manipulation of the rammer
5	guide
6	water inlet

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, it is essential to put a 7 cm to 8 cm layer in the column, to pack it and to 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 case.

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 6.2.6), 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 needs to be taken in positioning the pre-filter in place: the filter can 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 Formula 2.

$$m_0 = \frac{\left(m \times w_{\mathrm{dr}}\right)}{100} \qquad (2)$$

where

m_0 is the dry mass of the test portion in the column, in kg;

m is the mass of the (moist) test portion in the column, in kg;

w_{dr} is the dry residue, in %.

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

8.4 Start-up of the test

Saturate the column (6.2.1) with water (5.1) either by using the pump (see 6.2.7) 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 tubing (6.2.8) remains empty. Leave the saturated material for a period of 16 h up to 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 Formula 3.

$$\varphi = v_L \times \pi \times d^2 \times 0{,}0104 \qquad (3)$$

where

ϕ is the leachant flow rate, in ml/h;

v_L is the linear velocity of the leachant through the empty column, in cm/day;

d is the diameter of the column, in 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 6.2.10) 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 tubing(6.2.8) to an eluate collection bottle of appropriate size (see 6.2.4). 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.

NOTE 7 The flow rate may be increased by a factor of 4 if it is demonstrated that such increased flow rate does not lead to pressure build-up and the same results can be obtained at 90 % confidence level.

Start the pump again and change the collection bottle after a quantity of (0,1 ± 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 6.2.12) and make a note of it.

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.

For organic substances proceed as follows:

a) If the turbidity of the eluate is below 100 FNU the eluate shall be analysed as it is. Otherwise, transfer the eluate to centrifuge tubes (6.2.9).

b) There are two options for solid-liquid separation:

   i) centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high speed centrifuge (6.2.9);

   ii) centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower speed centrifuge (6.2.9).

NOTE 8 Limitation of maximum centrifugation duration is necessary especially for organic substances. The speed or g-force of the centrifuge is dependent on the specific rotor.

NOTE 9 Background information on sorption effects on container walls is given in [26].

If necessary, cooling shall be applied to maintain the temperature between 19 °C and 25 °C.

Gentle braking shall be applied in order to avoid re-suspension. Deceleration time shall not exceed 20 min.

If only leaching of inorganic substances is of interest, the eluate does not have to be centrifuged as described above but has to be filtered using the 0,45 µm membrane filters (6.2.5) and a vacuum or pressure filtration device (6.2.5).

8.5 Collection of additional eluate fractions

Check the flow rate of the leachant at least three times per week, and adjust to the original linear velocity (in the range of 15 cm/day ± 2 cm/day).

Because of material-specific situations, it can 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) shall be fulfilled.

For organic substances proceed as follows:

a) If the turbidity of the eluate is below 100 FNU the eluate shall be analysed as it is. Otherwise, transfer the eluate to centrifuge tubes (6.2.9).

b) There are two options for solid-liquid separation:

   i) centrifuge the eluate for 30 min at 20 000 g to 30 000 g using a high speed centrifuge (6.2.9);

   ii) centrifuge the eluate for 5 h at 2 000 g to 2 500 g in glass bottles using a lower speed centrifuge (6.2.9).

NOTE 2 Limitation of maximum centrifugation duration is necessary especially for organic substances. The speed or g-force of the centrifuge is dependent on the specific rotor.

NOTE 3 Background information on sorption effects on container walls is given in [26].

If necessary, cooling shall be applied to maintain the temperature between 19 °C and 25 °C.

Gentle braking shall be applied in order to avoid re-suspension. Deceleration time shall not exceed 20 min.

If only leaching of inorganic substances is of interest, the eluate does not have to be centrifuged as described above but has to be filtered using the 0,45 µm membrane filters (6.2.5) and a vacuum or pressure filtration device (6.2.5).

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/S* ratio times dry mass)** [a] l	**Cumulative *L/S* ratio** l/kg dry matter
1	(0,1 ± 0,02) × m_0	0,1 ± 0,02
2	(0,1 ± 0,02) × m_0	0,2 ± 0,04
3	(0,3 ± 0,05) × m_0	0,5 ± 0,08
4	(0,5 ± 0,1) × m_0	1,0 ± 0,15
5	(1,0 ± 0,2) × m_0	2,0 ± 0,3
6	(3,0 ± 0,2) × m_0	5,0 ± 0,4
7	(5,0 ± 0,2) × m_0	10,0 ± 0,1

[a] In the case of high salt loads (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 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 4 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 5 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 6 The execution time of the test can be calculated from Formula 4.

$$t = \frac{\left(L/S \times m_0 \times 1\,000\right)}{\left(24 \times \varphi\right)} \tag{4}$$

where

t is the execution time of the test, in days;

L/S is the final liquid to solid ratio, in l/kg dry matter;

m_0 is the dry mass of the test portion, kg;

ϕ is the leachant flow rate, in ml/h.

NOTE 7 If the length of the column is 30 cm, the linear velocity is 15 cm/day and the bulk density of the material is 1 500 kg/m^3, the execution time of the test is approximately 30 days for *L/S* = 10 l/kg.

NOTE 8 Under certain circumstances, particularly for alkaline eluates, it is advised to measure the pH and redox potential of the raw eluate prior to filtration or centrifugation, since these operations may change pH and redox of the eluate.

8.6 Further preparation of the eluates for analysis

Measure the pH (see 6.2.12) and conductivity (see 6.2.11) in accordance with EN 16192 (and optionally redox potential E_h in mV (see 6.2.13)) of each eluate fraction, just after collection. If the conductivity of an eluate fraction exceeds 7 500 mS/m, measure the volume (or density) of the eluate and dilute the eluate until the conductivity is below 7 500 mS/m. Ensure that the dilution factor is no more than 10.

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 (e.g. for non-volatile organic substances, elements by ICP analysis, salts by liquid chromatography, etc.), after shaking well. Preserve the eluate sub-samples depending on the elements to be analyzed, according to EN ISO 5667-3. Store the (sub) eluates in sealed bottles, according to EN ISO 5667-3.

Determine the concentrations of substances of interest using the methods of eluate analysis according to EN 16192 and methods for organic substances. Also measure the total concentration of dissolved organic carbon (DOC) using EN 16192.

8.7 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 pretreatment). 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 6.2.11) of these blank eluates. Preserve and analyse both blank eluates in accordance with 8.6.

If the test is performed for comparison with any regulatory standards, these standards shall dictate the criteria 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.

9 Calculations

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

$$U_i = \frac{(V_i \times c_i)}{(m_0)} \tag{5}$$

where

U_i is the released quantity of a component per quantity of sample for analysis in eluate fraction, *i,* in mg/kg dry matter;

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

V_i is the volume of the eluate fraction, *i,* in l;

c_i is the concentration of the component concerned in the eluate fraction *i,* in mg/l;

m_0 is the dry mass of the test portion in the column, in kg.

The concentration c_i referred to in Formula 5, shall be the concentration originally present in the eluate. The measured value determined in accordance with 8.5 shall be corrected if the eluate fraction has been diluted and/or if the quantity of preservation fluid added in 8.6 was more than 1 ml per 100 ml of eluate.

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

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 upper limit and the lower limit of ΣU_i.

10 Performance characteristics

The performance characteristics of the method as determined in a parallel inter-comparison validation between US EPA method 1314 and this standard (EN 14405) are given in Annex D. Table 3 gives the resulting typical values for repeatability and reproducibility limits as their observed ranges. The typical value is derived from the data in Table D.2 by taking the median value and rounding the numbers.

Table 3 — Typical values and observed ranges of the repeatability and reproducibility limits

Results of the validation of the release of substances as a function of liquid to solid ratio in a percolation test on waste	**Typical value** %	**Observed range** %
Repeatability limit, *r*	5	3 - 6
Reproducibility limit, *R*	14	13 - 15
NOTE The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. The repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary field sample.		

NOTE 1 The repeatability limit (*r*) and the reproducibility limit (*R*) as given in Table D.2 and in this table are indicative values of the attainable precision, if the determination of the release of substances as a function of liquid to solid ratio in a percolation test on waste is performed in accordance with this standard.

NOTE 2 A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the determination of the release of substances as a function of liquid to solid ratio in a percolation test on waste. In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Table D.2 and this table.

11 Test report

11.1 General

In order to comply with the present document, the following information shall be documented in such a way that they are immediately available on request. All the deviation from the minimum requirements of this document shall also be documented in the test report. The report shall contain at least the following data.

11.2 General data

— reference to this European Standard;

— date of receipt of the laboratory sample;

— sampling report, according to 7.1;

— identification of the laboratory sample;

— ageing conditions (if relevant);

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

11.3 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.

11.4 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 analyzed, in accordance with 8.6;

- quantities of preservation fluid added in accordance with 8.6 where this is more than 1 ml per 100 ml eluate;

- analytical report, in accordance with 8.6;

- all measured concentrations.

11.5 Results of the leaching test

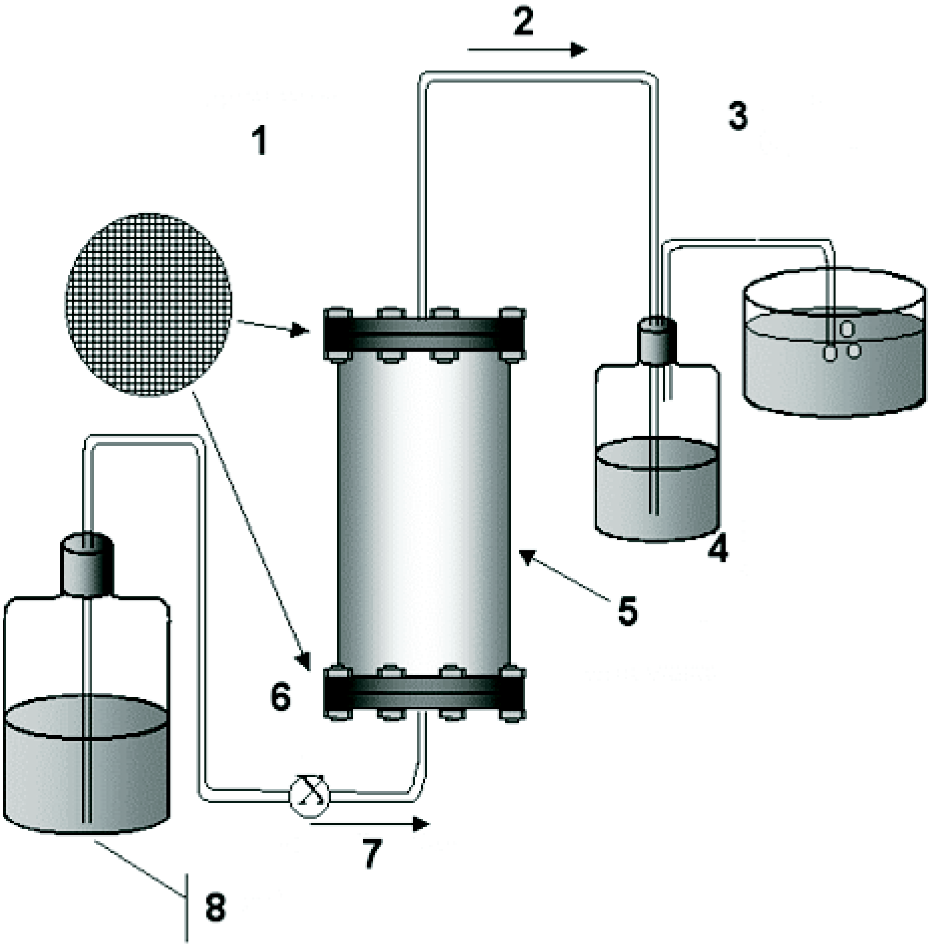
- the measured concentrations per fraction for each component, in mg/l, preferably plotted against the *L/S* ratio;

- the released quantities Ui 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 $\Sigma\underline{U}$i calculated for each component, in mg/kg dry mass. Again, if relevant, lower and upper limits shall be indicated.

Annex A
(informative)

Illustration of the column and accompanying equipment



Key

1 support filter
2 flow direction
3 air lock (optional)
4 eluate collection
5 column filled with waste
6 pump
7 flow direction
8 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 shall be known, in order to be able to determine the dry mass of the test portion in the column (in 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 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 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 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 8.4 two methods are mentioned to saturate the packed column with leachant: by using the pump (1) or by using initial hydrostatic pressure (2).

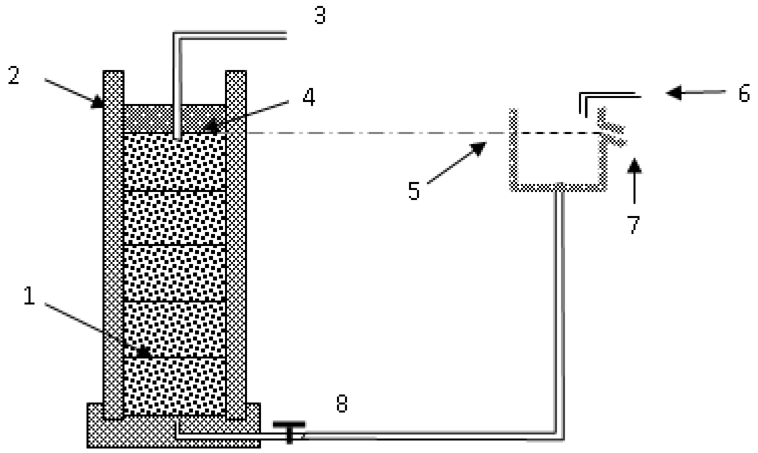
1: Saturating the column by using the pump

Connect the pump (see 6.8) to the bottom section of the column and pass demineralized water (see 5.1) 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.



Key

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

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

B.4 Equilibration and checking of equilibrium conditions

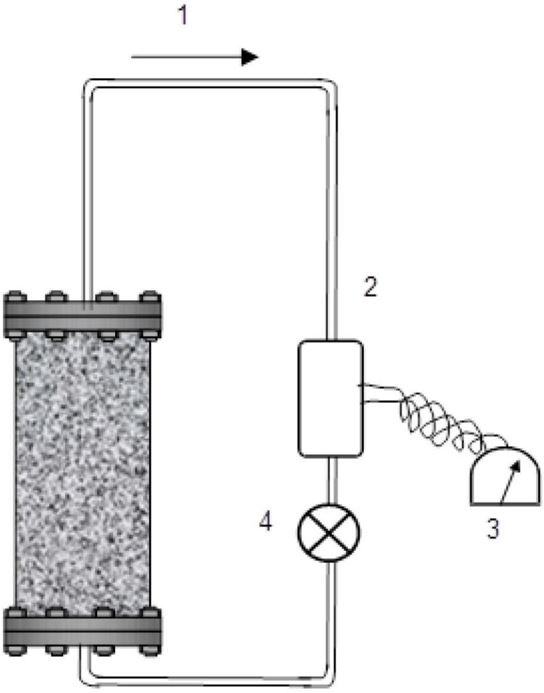
An alternative procedure to establish and control equilibrium conditions during the start-up of the percolation test may be used:

— 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 visualized in Figure B.2);

— Circulate the leachant in closed loop until the pH equilibrium condition is reached (pH deviation in the bottle ≤ 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.



Key

1 water flow
2 bottle for collection and control of the first 0,1 eluate
3 monitoring
4 pump

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 8.4) is not met, the following actions may be taken. A more detailed explanation on equilibrium conditions is given in C.1.

— 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 start-up. 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.

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 EN 12920 that specifies a methodology for the determination of the leaching behaviour of waste under specified conditions. This methodology aims at determining the influence of chemical, physical, geotechnical, mechanical and biological parameters on the release of inorganic substances from waste bodies in the scenario under consideration. EN 12920 distinguishes between parameter specific tests which are intended for measuring intrinsic properties of waste or the effects of specific parameters on the release of inorganic substances from waste 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[3] between the waste 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.

Over the years substantial experience has been gained with column test protocols similar to the present standard both in terms of practical experience in running the test as well as reaction/transport modelling and test data interpretation for long term release predictions [14 - 19].

Test users with specific test needs, e.g. to ensure that the local equilibrium assumption is fulfilled, are hence referred to the C.5.

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;

---

[3] 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.

— temperature.

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

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 7.2 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 generalized.

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 ± 5 cm. The conditions prescribing which column size to be used for a particular sample of waste has been presented in Table 1.

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

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

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 h), through the empty column. This is convenient since the equilibrium / non- equilibrium conditions in the waste 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 an utilisation scenario of waste materials.

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 waste 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 waste 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 waste materials 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 ± 2 cm/day) when applying this flow rate compared to the sometimes very low flow rates observed in field conditions.

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

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

The following eluate fractions are collected: *L/S* = 0,0 l/kg to 0,1 l/kg, 0,1 l/kg to 0,2 l/kg, 0,2 l/kg to 0,5 l/kg, 0,5 l/kg to 1,0 l/kg, 1,0 l/kg to 2,0 l/kg, 2,0 l/kg to 5,0 l/kg and 5,0 l/kg to 10 l/kg. The sizes 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 l/kg to 1 l/kg or 0 l/kg to 2 l/kg. In addition, the *L/S* of many of the field leaching scenarios (landfills and utilisation applications), which could be modelled on the basis of percolation test results, will often require many years to reach a value of *L/S* = 1 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 waste with a density of 1 Mg/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 waste with a similar density and percolation rate, *L/S* ratios of 2 l/kg and 10 l/kg will be attained after 200 years and 1 000 years, respectively.

It should be noted, that the collection of the eluate as fractions is well suited to describe the amount of substances 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 analyzed at those L/S values.

C.7 Nature of the leachant

For most waste types the composition of the eluate at *L/S* values below 10 l/kg is controlled predominantly by the composition of the waste. It makes little difference whether the leachant consists of demineralized water or (real or artificial) rainwater. For the sake of simplicity demineralized water has therefore been chosen as the leachant in this European Standard.

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

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 (19 °C to 25 °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 range, 19 °C to 25 °C, has been chosen to represent common indoor conditions and to be consistent with the temperature requirements of EN 12457 parts 1 to 4.

C.9 Beneficial use of the test results

The test results can be used in predicting the release of substances from a waste 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 waste under specified conditions this test can generally not be used alone: the application of other test methods may be required (see EN 12920).

Under certain conditions the test may also provide information on pore water concentrations in scenarios in which waste and aqueous solutions are in equilibrium, both in the short- and long-term (after wash-out of the salts), if the modelling step of EN 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 parts 1 to 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 parts 1 to 4, which may lead to different end results. In cases where the changes in pH during the test is limited, the results obtained at cumulative $L/S = 10$ correspond well with pH dependence test (EN 14429 or EN 14997) results obtained at material’s pH.

Annex D
(informative)

Repeatability and reproducibility data

D.1 Materials used in the interlaboratory comparison study

The interlaboratory comparison of the release of substances as a function of liquid to solid ratio in a percolation test on waste was carried out with 10 American and 1 European laboratory on 2 materials. The materials selected for the interlaboratory comparison were chosen to represent waste (detailed information can be found in the final report on the interlaboratory comparison study mentioned [20, 21]).

NOTE Validation data for non-volatile organic substances are not currently available, but will be added on revision when available.

Table D.1 provides a list of the types of materials chosen for testing and the selected components.

Table D.1 — Material types tested and components analyzed in the interlaboratory comparison of - Determination of the release of substances as a function of liquid to solid ratio in a percolation test on waste

Grain size class	Sample code	Material type tested
Very fine grained (< 0,5 mm)	JaFS	Foundry sand
Fine grained (< 2 mm)	CFS	Contaminated soil from a smelter facility

D.2 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2 after log normal transformation of the test results. The average values, the repeatability standard deviation (RSD_r) and the reproducibility standard deviation (RSD_R) were obtained (Table D.2).

Table D.2 — Results of the interlaboratory comparison studies of determination of the release of substances as a function of liquid to solid ratio in a percolation test on waste

CUMULATIVE RELEASE				Concentration range				Labs		Collected	Filtered	Used			Concentration range		
EPA Method	**Matrix**	**Parameter**	*L/S* l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	*p*	Out-liers	Total number	Number < DLT	Total used	EN	*L/S*	Min. mg/kg	Max. mg/kg	RSD_r %
1314	CFS	Al	0,2	0,0095	0,0281	12	34	7		24	0	24	14405	0,1	0,007	0,008	6
			0,5	0,0222	0,0530	10	23	7		24	0	24		0,2	0,014	0,015	3
			1,0	0,0418	0,0815	7	18	7		24	0	24		0,5	0,031	0,034	5
			1,5	0,0602	0,119	9	17	7		24	0	24		1,0	0,060	0,065	5
			2,0	0,0790	0,144	9	17	7		24	0	24		2,0	0,109	0,119	4
			4,5	0,174	0,265	6	15	7		24	0	24		5,0	0,263	0,279	3
			5,0	0,192	0,287	5	15	7		24	0	24		10,1	0,518	0,558	4
			9,5	0,346	0,496	4	14	7		24	0	24					
			10,0	0,363	0,523	5	14	7		24	0	24					
1314	CFS	Ba	0,2	0,0066	0,0113	7	16	7		24	0	24	14405	0,1	0,005	0,005	6
			0,5	0,0207	0,0295	5	12	7		24	0	24		0,2	0,011	0,012	6
			1,0	0,0480	0,0772	3	15	7		24	0	24		0,5	0,034	0,034	1
			1,5	0,0768	0,137	3	19	7		24	0	24		1,0	0,074	0,075	1
			2,0	0,106	0,199	3	21	7		24	0	24		2,0	0,157	0,159	1
			4,5	0,268	0,529	3	24	7		24	0	24		5,0	0,427	0,432	1
			5,0	0,303	0,598	3	24	7		24	0	24		10,1	0,905	0,914	1
			9,5	0,633	1,238	3	23	7		24	0	24					
			10,0	0,672	1,313	3	23	7		24	0	24					
1314	CFS	B	0,2	0,299	0,553	9	20	7		24	0	24	14405	0,1	0,18	0,19	5
			0,5	0,639	1,07	5	17	7		24	0	24		0,2	0,34	0,37	5

CUMULATIVE RELEASE				Concentration range				Labs		Collected	Filtered	Used			Concentration range		
EPA Method	Matrix	Parameter	L/S l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	p	Out-liers	Total number	Number < DLT	Total used	EN	L/S	Min. mg/kg	Max. mg/kg	RSD_r %
			1,0	0,917	1,45	4	14	7		24	0	24		0,5	0,67	0,70	3
			1,5	1,06	1,63	4	13	7		24	0	24		1,0	0,90	0,92	1
			2,0	1,14	1,75	4	13	7		24	0	24		2,0	1,09	1,10	1
			4,5	1,40	2,09	3	12	7		24	0	24		5,0	1,38	1,41	1
			5,0	1,44	2,14	3	12	7		24	0	24		10,1	1,68	1,72	1
			9,5	1,72	2,45	3	11	7		24	0	24					
			10,0	1,74	2,48	3	11	7		24	0	24					
1314	CFS	Ca	0,2	74,0	103	2	11	7		24	0	24	14405	0,1	46,2	49,5	4
			0,5	179	278	2	12	7		24	0	24		0,2	95,4	100	3
			1,0	407	599	3	9	7		24	0	24		0,5	258	265	1
			1,5	658	929	2	9	7		24	0	24		1,0	558	565	1
			2,0	916	1 254	2	9	7		24	0	24		2,0	1 185	1 202	1
			4,5	2 196	2 897	1	8	7		24	0	24		5,0	3 133	3 155	1
			5,0	2 461	3 238	1	8	7		24	0	24		10,1	6 416	6 487	1
			9,5	4 826	6 290	1	8	7		24	0	24					
			10,0	5 051	6 639	2	8	7		24	0	24					
1314	CFS	Cd	0,2	2,33	4,65	8	22	7		24	0	24	14405	0,1	2,5	2,8	6
			0,5	4,33	8,36	7	17	7		24	0	24		0,2	4,1	4,5	5
			1,0	7,58	13,8	6	16	7		24	0	24		0,5	7,2	7,7	4
			1,5	10,9	19,1	6	15	7		24	0	24		1,0	11,9	12,4	2
			2,0	14,3	24,2	6	14	7		24	0	24		2,0	21,1	21,9	2
			4,5	29,7	44,9	4	11	7		24	0	24		5,0	42,7	43,4	1

CUMULATIVE RELEASE				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**			**Concentration range**		
EPA Method	**Matrix**	**Parameter**	*L/S* l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	*p*	Out-liers	Total number	Number < DLT	Total used	EN	*L/S*	Min. mg/kg	Max. mg/kg	RSD_r %
			5,0	32,5	48,2	4	11	7		24	0	24		10,1	64,9	66,6	1
			9,5	52,1	70,5	2	8	7		24	0	24					
			10,0	53,9	72,6	2	9	7		24	0	24					
1314	CFS	Cu	0,2	0,265	0,909	11	38	7		24	0	24	14405	0,1	0,35	0,41	9
			0,5	0,562	1,78	12	32	7		24	0	24		0,2	0,60	0,66	5
			1,0	1,06	3,04	9	28	7		24	0	24		0,5	1,17	1,26	4
			1,5	1,59	4,27	8	26	7		24	0	24		1,0	1,96	2,08	3
			2,0	2,12	5,35	7	25	7		24	0	24		2,0	2,60	2,86	5
			4,5	4,68	9,77	6	21	7		24	0	24		5,0	5,25	5,65	4
			5,0	5,16	10,5	6	20	7		24	0	24		10,1	9,15	9,76	3
			9,5	8,74	14,9	4	16	7		24	0	24					
			10,0	9,08	15,3	4	15	7		24	0	24					
1314	CFS	Mo	0,2	0,0037	0,0072	15	18	7		24	0	24	14405	0,1	0,004	0,004	6
			0,5	0,0079	0,0118	7	10	7		24	0	24		0,2	0,006	0,007	7
			1,0	0,0114	0,0161	5	9	7		24	0	24		0,5	0,011	0,012	3
			1,5	0,0145	0,0208	5	10	7		24	0	24		1,0	0,015	0,016	2
			2,0	0,0174	0,0247	5	9	7		24	0	24		2,0	0,021	0,022	2
			4,5	0,0316	0,0452	6	10	7		24	0	24		5,0	0,044	0,045	2
			5,0	0,0346	0,0495	5	11	7		24	0	24		10,1	0,080	0,083	2
			9,5	0,0603	0,0867	5	12	7		24	0	24					
			10,0	0,0631	0,0908	4	12	7		24	0	24					
1314	CFS	Pb	0,2	0,102	0,199	7	24	7		24	0	24	14405	0,1	0,11	0,12	7

CUMULATIVE RELEASE				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**			**Concentration range**		
EPA Method	**Matrix**	**Parameter**	*L/S* l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	*p*	Out-liers	Total number	Number < DLT	Total used	EN	*L/S*	Min. mg/kg	Max. mg/kg	RSD_r %
			0,5	0,293	0,471	6	18	7		24	0	24		0,2	0,19	0,21	3
			1,0	0,601	0,991	5	17	7		24	0	24		0,5	0,44	0,47	4
			1,5	0,959	1,57	4	15	7		24	0	24		1,0	0,79	0,86	5
			2,0	1,37	2,11	3	15	7		24	0	24		2,0	1,25	1,43	7
			4,5	3,51	4,92	2	13	7		24	0	24		5,0	3,33	3,68	5
			5,0	3,96	5,46	2	13	7		24	0	24		10,1	6,97	7,53	4
			9,5	7,51	9,80	2	11	7		24	0	24					
			10,0	7,77	10,3	2	11	7		24	0	24					
1314	CFS	Tl	0,2	0,237	0,492	10	20	7		24	0	24	14405	0,1	0,26	0,28	5
			0,5	0,529	0,836	6	14	7		24	0	24		0,2	0,44	0,46	3
			1,0	0,797	1,23	4	13	7		24	0	24		0,5	0,71	0,74	2
			1,5	1,01	1,53	4	12	7		24	0	24		1,0	1,05	1,05	1
			2,0	1,18	1,76	4	11	7		24	0	24		2,0	1,50	1,53	1
			4,5	1,77	2,48	3	10	7		24	0	24		5,0	2,20	2,28	2
			5,0	1,85	2,58	3	10	7		24	0	24		10,1	2,82	2,97	3
			9,5	2,39	3,28	3	10	7		24	0	24					
			10,0	2,42	3,35	3	10	7		24	0	24					
1314	CFS	Zn	0,2	9,20	19,1	8	22	7		24	0	24	14405	0,1	9,4	10,5	6
			0,5	17,5	37,7	9	21	7		24	0	24		0,2	17,0	18,6	5
			1,0	31,1	66,1	8	21	7		24	0	24		0,5	32,7	36,1	5
			1,5	45,1	93,2	7	20	7		24	0	24		1,0	57,3	61,2	3
			2,0	59,5	119	6	18	7		24	0	24		2,0	105	111	2

CUMULATIVE RELEASE				Concentration range				Labs		Collected	Filtered	Used			Concentration range		
EPA Method	**Matrix**	**Parameter**	L/S l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	p	Out-liers	Total number	Number < DLT	Total used	EN	L/S	Min. mg/kg	Max. mg/kg	RSD_r %
			4,5	125	220	5	15	7		24	0	24		5,0	211	221	2
			5,0	136	236	5	14	7		24	0	24		10,1	309	326	3
			9,5	218	334	4	11	7		24	0	24					
			10,0	226	343	4	11	7		24	0	24					
1314	JaFS	Al	0,2	0,0137	0,0194	8	15	7		24	0	24	14405	0,1	0,010	0,011	8
			0,5	0,0298	0,0348	8	12	7		24	0	24		0,2	0,018	0,020	5
			1,0	0,0413	0,0516	9	14	7		24	0	24		0,5	0,026	0,028	5
			1,5	0,0512	0,0620	8	12	7		24	0	24		1,2	0,038	0,043	7
			2,0	0,0606	0,0728	8	11	7		24	0	24		2,3	0,049	0,057	8
			4,5	0,0966	0,117	6	9	7		24	0	24		5,5	0,084	0,093	6
			5,0	0,106	0,126	6	9	7		24	0	24		10,8	0,136	0,144	3
			9,5	0,178	0,204	7	12	7		24	0	24					
			10,0	0,186	0,210	7	13	7		24	0	24					
1314	JaFS	Ba	0,2	0,0246	0,0293	4	9	7		24	0	24	14405	0,1	0,006	0,008	12
			0,5	0,0521	0,0581	5	8	7		24	0	24		0,2	0,015	0,018	10
			1,0	0,0756	0,0824	6	6	7		24	0	24		0,5	0,032	0,035	6
			1,5	0,0924	0,101	6	7	7		24	0	24		1,2	0,052	0,059	7
			2,0	0,109	0,119	6	8	7		24	0	24		2,3	0,086	0,097	6
			4,5	0,173	0,200	6	8	7		24	0	24		5,5	0,162	0,184	6
			5,0	0,183	0,212	6	8	7		24	0	24		10,8	0,236	0,261	5
			9,5	0,245	0,300	6	11	7		24	0	24					
			10,0	0,250	0,304	6	11	7		24	0	24					

CUMULATIVE RELEASE				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**			**Concentration range**		
EPA Method	**Matrix**	**Parameter**	*L/S* l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	*p*	Out-liers	Total number	Number < DLT	Total used	EN	*L/S*	Min. mg/kg	Max. mg/kg	RSD_r %
1314	JaFS	B	0,2	34,8	41,9	8	28	7		24	0	24	14405	0,1	18,0	21,8	10
			0,5	56,1	60,4	5	12	7		24	0	24		0,2	30,4	34,9	7
			1,0	67,8	73,7	5	8	7		24	0	24		0,5	50,9	53,8	3
			1,5	72,4	80,2	5	8	7		24	0	24		1,2	60,0	66,0	5
			2,0	75,3	83,8	5	8	7		24	0	24		2,3	67,0	74,4	6
			4,5	82,2	91,7	5	7	7		24	0	24		5,5	75,4	83,9	6
			5,0	83,0	92,6	5	7	7		24	0	24		10,8	81,9	91,2	5
			9,5	88,0	98,0	4	6	7		24	0	24					
			10,0	88,3	98,4	4	6	7		24	0	24					
1314	JaFS	Ca	0,2	65,8	84,8	9	21	7		24	0	24	14405	0,1	47,9	53,4	6
			0,5	77,0	100	7	16	7		24	0	24		0,2	78,5	83,4	3
			1,0	87,7	108	7	12	7		24	0	24		0,5	91,4	94,5	2
			1,5	95,8	117	6	9	7		24	0	24		1,2	102	106	2
			2,0	104	126	6	8	7		24	0	24		2,3	119	126	3
			4,5	136	173	5	10	7		24	0	24		5,5	163	170	2
			5,0	142	187	5	10	7		24	0	24		10,8	219	225	1
			9,5	190	320	4	20	7		24	0	24					
			10,0	195	326	4	20	7		24	0	24					
1314	JaFS	Cu	0,2	0,246	0,463	12	31	7		24	0	24	14405	0,1	0,16	0,19	9
			0,5	0,481	0,838	10	24	7		24	0	24		0,2	0,29	0,34	8
			1,0	0,781	1,15	8	20	7		24	0	24		0,5	0,58	0,64	5
			1,5	0,952	1,34	7	19	7		24	0	24		1,2	0,82	0,95	8

CUMULATIVE RELEASE				Concentration range				Labs		Collected	Filtered	Used			Concentration range		
EPA Method	Matrix	Parameter	L/S l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	p	Out-liers	Total number	Number < DLT	Total used	EN	L/S	Min. mg/kg	Max. mg/kg	RSD_r %
			2,0	1,08	1,48	6	19	7		24	0	24		2,3	1,10	1,27	7
			4,5	1,51	2,00	5	20	7		24	0	24		5,5	1,61	1,78	6
			5,0	1,57	2,07	5	20	7		24	0	24		10,8	2,04	2,27	6
			9,5	1,97	2,56	5	21	7		24	0	24					
			10,0	2,01	2,60	5	21	7		24	0	24					
1314	JaFS	Mg	0,2	12,1	15,7	9	25	7		24	0	24	14405	0,1	8,3	9,5	7
			0,5	14,3	18,1	6	19	7		24	0	24		0,2	13,4	14,6	4
			1,0	15,7	19,4	6	14	7		24	0	24		0,5	15,6	16,6	3
			1,5	16,8	20,7	6	11	7		24	0	24		1,2	17,4	18,7	4
			2,0	18,0	22,1	5	10	7		24	0	24		2,3	20,4	22,1	4
			4,5	24,1	28,6	5	9	7		24	0	24		5,5	27,1	29,1	4
			5,0	24,8	30,6	5	10	7		24	0	24		10,8	34,6	36,5	3
			9,5	30,4	48,2	4	17	7		24	0	24					
			10,0	30,9	48,9	4	17	7		24	0	24					
1314	JaFS	Mn	0,2	0,0545	0,0659	9	28	7		24	0	24	14405	0,1	0,024	0,031	12
			0,5	0,0662	0,0771	7	23	7		24	0	24		0,2	0,039	0,046	9
			1,0	0,0740	0,0841	7	20	7		24	0	24		0,5	0,046	0,052	8
			1,5	0,0799	0,093	7	19	7		24	0	24		1,2	0,049	0,059	9
			2,0	0,0874	0,102	7	18	7		24	0	24		2,3	0,057	0,068	8
			4,5	0,116	0,141	7	18	7		24	0	24		5,5	0,074	0,086	9
			5,0	0,121	0,149	7	17	7		24	0	24		10,8	0,089	0,103	8
			9,5	0,146	0,196	7	15	7		24	0	24					

CUMULATIVE RELEASE				Concentration range				Labs		Collected	Filtered	Used			Concentration range		
EPA Method	**Matrix**	**Parameter**	*L/S* l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	*p*	Out-liers	Total number	Number < DLT	Total used	EN	*L/S*	Min. mg/kg	Max. mg/kg	RSD_r %
			10,0	0,148	0,198	7	15	7		24	0	24					
1314	JaFS	Mo	0,2	0,0202	0,0308	9	36	7		24	0	24	14405	0,1	0,012	0,013	7
			0,5	0,0388	0,0469	6	21	7		24	0	24		0,2	0,021	0,023	5
			1,0	0,0463	0,0559	6	15	7		24	0	24		0,5	0,034	0,038	5
			1,5	0,0529	0,0627	5	14	7		24	0	24		1,2	0,044	0,050	6
			2,0	0,0587	0,0682	5	13	7		24	0	24		2,3	0,057	0,063	6
			4,5	0,0774	0,0885	4	10	7		24	0	24		5,5	0,075	0,084	7
			5,0	0,0797	0,0918	4	10	7		24	0	24		10,8	0,092	0,101	5
			9,5	0,0947	0,115	5	11	7		24	0	24					
			10,0	0,0959	0,118	4	11	7		24	0	24					
1314	JaFS	Pb	0,2	0,0183	0,0257	11	16	7		24	0	24	14405	0,1	0,04	0,04	3
			0,5	0,0541	0,0744	9	12	7		24	0	24		0,2	0,07	0,07	2
			1,0	0,107	0,136	8	11	7		24	0	24		0,5	0,11	0,13	4
			1,5	0,150	0,189	8	10	7		24	0	24		1,2	0,17	0,19	7
			2,0	0,184	0,244	8	11	7		24	0	24		2,3	0,22	0,25	8
			4,5	0,324	0,440	9	12	7		24	0	24		5,5	0,34	0,36	4
			5,0	0,347	0,475	9	12	7		24	0	24		10,8	0,48	0,52	4
			9,5	0,513	0,722	10	15	7		24	0	24					
			10,0	0,529	0,736	9	15	7		24	0	24					
1314	JaFS	Zn	0,2	0,260	0,376	11	35	7		24	0	24	14405	0,1	0,29	0,34	8
			0,5	0,419	0,530	9	29	7		24	0	24		0,2	0,47	0,52	5
			1,0	0,530	0,676	8	29	7		24	0	24		0,5	0,59	0,65	4

CUMULATIVE RELEASE				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**			**Concentration range**		
EPA Method	**Matrix**	**Parameter**	*L/S* l/kg	Min. mg/kg	Max. mg/kg	RSD_r %	RSD_R %	*p*	Out-liers	Total number	Number < DLT	Total used	EN	*L/S*	Min. mg/kg	Max. mg/kg	RSD_r %
			1,5	0,587	0,767	8	28	7		24	0	24		1,2	0,70	0,78	6
			2,0	0,633	0,846	8	27	7		24	0	24		2,3	0,82	0,93	7
			4,5	0,787	1,04	7	22	7		24	0	24		5,5	1,01	1,17	8
			5,0	0,805	1,07	7	21	7		24	0	24		10,8	1,16	1,33	7
			9,5	0,913	1,23	7	19	7		24	0	24					
			10,0	0,923	1,24	7	19	7		24	0	24					

RSD_r is repeatability standard deviation

RSD_R is reproducibility standard deviation

p is the number of labs remaining in statistical evaluation

NOTE 1 All laboratories conducted 3 replicates of each test with one of the laboratories (the reference laboratory) conducting 6 replicates.

NOTE 2 No outlier analysis was performed.

NOTE 3 No data were removed from analysis (i.e., "filtered") as a result of comparison of eluate concentrations to high blank concentrations.

NOTE 4 No data were "filtered for cause" due to procedural issues (e.g., pH measurements, *L/S* ratios out of target range).

NOTE 5 NA indicates that RSD_r and RSD_R were not calculated since all data is below DLT.

NOTE 6 For the CEN test only the RSD_r can be quantified.

Bibliography

[1] EN 12457-1:2002, *Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 1: One stage batch test at a liquid to solid ratio of 2 l/kg for materials with high solid content and with particle size below 4 mm (without or with size reduction)*

[2] EN 12457-2:2002, *Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 2: One stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (without or with size reduction)*

[3] EN 12457-3:2002, *Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 3: Two stage batch test at a liquid to solid ratio of 2 l/kg and 8 l/kg for materials with high solid content and with particle size below 4 mm (without or with size reduction)*

[4] EN 12457-4:2002, *Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 4: One stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 10 mm (without or with size reduction)*

[5] EN 12920:2006+A1:2008, *Characterization of waste - Methodology for the determination of the leaching behaviour of waste under specified conditions*

[6] EN 14429:2015, *Characterization of waste - Leaching behaviour test - Influence of pH on leaching with initial acid/base addition*

[7] EN 14997:2015, *Characterization of waste - Leaching behaviour test - Influence of pH on leaching with continuous pH control*

[8] CEN/TS 16637-3:2016, *Construction products - Assessment of release of dangerous substances - Part 3: Horizontal up-flow percolation test*

[9] CEN ISO/TS 21268-3:2009, *Soil quality - Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials - Part 3: Up-flow percolation test (ISO/TS 21268-3:2007)*

[10] 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*

[11] ISO 11464:2006, *Soil quality — Pretreatment of samples for physico-chemical analysis*

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

[13] NT ENVIR 002:1995, *Solid waste — Granular inorganic material — Column test*

[14] FÄLLMAN A.M., AURELL B. Leaching tests for environmental assessment of inorganic substances in wastes, Sweden. *Sci. Total Environ.* 1996, **178** pp. 71–84

[15] D. GOETZ and W. GLÄSEKER, Percolation method for the leaching of aggregates, Report of AK 6.4.1. "Leaching techniques for aggregates" of the Research Society for Highway Engineering and Transport, Published in Strasse + Autobahn 10/94, pp 605-609

[16] VAN DER SLOOT H.A., HEASMAN L., QUEVAUVILLER P.H., eds. *Harmonization of leaching/extraction tests, 1997. Studies in Environmental Science*. Elsevier Science, Amsterdam, Vol. 70, 1997

[17] WAHLSTRÖM M. Nordic recommendations for leaching tests for granular waste materials. *Sci. Total Environ.* 1996, **178** pp. 95–102

[18] Protocol of up-flow percolation test: pre-standard application within the scope of European standards – BRGM-report RP-50359-FR (ADEME agreement) – 2000

[19] DIJKSTRA J.J., VAN DER SLOOT H.A., COMANS R.N.J. Process identification and model development of contaminant transport in MSWI bottom ash. *Waste Manag.* 2002, **22** (5) pp. 531–541

[20] A.C. GARRABRANTS, D.S. KOSSON, R. DELAPP, P. KARIHER, P.F.A.B. SEIGNETTe, H.A. VAN DER SLOOT, L. STEFANSKI and M. BALDWIN, Interlaboratory Validation of the Leaching Environmental Assessment Framework (LEAF) Method 1314 and Method 1315, EPA 600/R-12/624, September 2012

[21] H.A. VAN DER SLOOT, D.S. KOSSON, A. GARRABRANTS, R. COMANS and O. HJELMAr, Parallel validation of CEN/TC 292 characterisation leaching methods with US EPA validation, CEN/TC 292/WG 6 N 631, June 2012

[22] IUPAC — INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY, ANALYTICAL CHEMISTRY DIVISION COMMISSION ON ANALYTICAL NOMENCLATURE. Nomenclature for sampling in analytical chemistry (Recommendations 1990). *Pure Appl. Chem.* 1990, **62** (6) pp. 1193–1208

[23] Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste

[24] Council Decision 2003/33/EC of 19 December 2002

[25] US EPA SWA-846. EPA method 1314, Liquid-solid partitioning (LSP) as a function of liquid-to-solid ratio for constituents in solid materials using an up-flow percolation column procedure, 2012; http://www.epa.gov/wastes/hazard/testmethods/sw846/index.htm

[26] KRÜGER O., KALBE U., MEIßNER K., SOBOTTKA S. Sorption effects interfering with the analysis of polycyclic aromatic hydrocarbons (PAH) in aqueous samples. *Talanta*. 2014 May, **122** pp. 151–156

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Copyright in BSI publications

All the content in BSI publications, including British Standards, is the property of and copyrighted by BSI or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use.

Save for the provisions below, you may not transfer, share or disseminate any portion of the standard to any other person. You may not adapt, distribute, commercially exploit, or publicly display the standard or any portion thereof in any manner whatsoever without BSI's prior written consent.

Storing and using standards

Standards purchased in soft copy format:

- A British Standard purchased in soft copy format is licensed to a sole named user for personal or internal company use only.
- The standard may be stored on more than 1 device provided that it is accessible by the sole named user only and that only 1 copy is accessed at any one time.
- A single paper copy may be printed for personal or internal company use only.

Standards purchased in hard copy format:

- A British Standard purchased in hard copy format is for personal or internal company use only.
- It may not be further reproduced – in any format – to create an additional copy. This includes scanning of the document.

If you need more than 1 copy of the document, or if you wish to share the document on an internal network, you can save money by choosing a subscription product (see 'Subscriptions').

Reproducing extracts

For permission to reproduce content from BSI publications contact the BSI Copyright & Licensing team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email subscriptions@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Useful Contacts

Customer Services
Tel: +44 345 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 345 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK