



BSI Standards Publication

**Waste — Characterization of granular solids with
potential for use as construction material —
Compliance leaching test — Up-flow percolation test**

National foreword

This British Standard is the UK implementation of EN 17516:2023.

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 committee manager.

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- Up-flow percolation test

Déchets - Caractérisation des solides granulaires
présentant un intérêt potentiel comme matériaux de
construction - Essai de conformité par lixiviation -
Essai de percolation à écoulement ascendant

Abfall - Charakterisierung von granularen Feststoffen
mit Verwertungspotential als Ersatzbaustoff -
Übereinstimmungsuntersuchung des
Elutionsverhaltens - Perkulationsprüfung im
Aufwärtsstrom

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European foreword

This document (EN 17516:2023) has been prepared by Technical Committee CEN/TC 444 “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of an identical text or by endorsement, at the latest by May 2024 and conflicting national standards shall be withdrawn at the latest by May 2024.

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 has been prepared under a standardization request addressed to CEN by the European Commission. The Standing Committee of the EFTA States subsequently approves these requests for its Member States.

This document specifies an up-flow percolation test to determine the compliance leaching status of granular solid waste with potential for beneficial use as construction product under standardized percolation conditions. The test is equal to the Horizontal up-flow percolation test for construction products (EN 16637-3), which in turn was elaborated based on the up-flow percolation test for characterization of waste (CEN/TS 14405:2004). Modifications have been implemented based on intensive robustness validation work on EN 16637-3 [4], [22]. The reason for implementing this percolation test is to provide a means to avoid double testing of waste-derived aggregates that could have a potential as construction products.

NOTE Waste legislation generally prescribes the use of EN 14405 to describe the leaching behaviour of inorganic and non-volatile organic substances from granular waste materials, whereas construction products legislation generally prescribes the use of EN 16637-3 to describe the leaching behaviour of inorganic and non-volatile organic substances from construction products, including waste materials with a potential as construction products.

Background information on characterization of leaching behaviour of construction products can be found in Technical Reports provided by CEN/TC 351 (i.e. CEN/TR 16098).

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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Introduction

The implementation of a Circular Economy has a high priority on the agenda of the European Commission. To fulfil the needs of a Circular Economy granular solid waste should be used as construction product as much as possible, thus diminishing the amount of landfilling to a minimum and save natural resources.

The release of dangerous substances upon contact with water results in a potential risk to the environment during the intended utilization of waste materials as construction products. The intent of this document together with EN 14405 or the EN 12457 series is to identify the leaching behaviour of granular solid waste with potential for beneficial use as construction product and thereby allow assessments of the release of regulated dangerous substances to soil, surface water and groundwater under intended use conditions in relation to CE marking of construction products derived from waste.

This document describes an up-flow percolation test for granular solid waste with potential for beneficial use as a construction product. It has been elaborated to avoid double testing, i.e. to assess the compliance with both waste and construction products regulations in one run. This test addresses granular solid waste with existing knowledge on long-term leaching behaviour in waste management scenarios obtained i.e. using basic characterization test EN 14405. The specified methods under Clauses 5 to 12 (Principle, Reagents, Equipment, Sample preparation, Test procedure, Evaluation of measurement results, Documentation and Test Report as well as Test Performance) are identical to the corresponding clauses of EN 16637-3 (Horizontal up-flow percolation test) with the exception that the term “construction product” has been changed to terms relevant for this standard such as “granular solid waste with potential to be reused as construction product” or similar.

In the different European countries, tests have been developed to characterize and assess dangerous substances which can be leached from solid matrices (e.g. construction products, contaminated soils, solid waste materials). The intent of these tests is to identify the leaching behaviour of these materials. The complexity of the leaching process makes simplifications necessary. All relevant aspects of leaching behaviour cannot be addressed in one single standard. The test hierarchy for waste is e.g. described in EN 14405, while guidance of appropriate leaching test for determining leaching of dangerous substances from construction products is specified in EN 16637-1.

The test procedure described in this document constitutes a compliance test used to verify compliance to regulatory limit values. Column percolation tests according to EN 14405 is applied for basic characterization of granular solid waste. Column percolation tests according to EN 16637-3 is applied for basic characterization of construction products.

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 general information on the waste (i.e. 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. 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.

Informative Annex A (A-Deviation) is an integral part of this document.

1 Scope

This document specifies an up-flow percolation test (PT) which is applicable in compliance testing to determine the leaching behaviour of inorganic and non-volatile organic substances from granular solids with potential for use as construction material. The test is not suitable for substances that are volatile under ambient conditions. The granular solids are subjected to percolation with water as a function of liquid to solid ratio under specified percolation conditions. The method is a once-through column leaching test.

NOTE 1 Volatile organic substances include the low molecular weight substances in mixtures such as mineral oil.

This up-flow percolation test is performed under specified test conditions for granular solids with potential for use as construction material and does not necessarily produce results that mimic specific intended use conditions. This test method produces eluates, which can subsequently be characterized by physical, chemical and ecotoxicological methods according to existing standard methods. The results of eluate analysis are presented as a function of the liquid/solid ratio. The test results enable the distinction between different leaching behaviour.

NOTE 2 It is not always possible to adjust test conditions simultaneously for inorganic and organic substances. Test conditions can also vary between different groups of organic substances. Test conditions for organic substances are generally more stringent than those for inorganic substances. The test conditions are generally described in a way that they fit testing organic substances and are also applicable to inorganic substances depending on the set-up.

NOTE 3 For ecotoxicity testing, eluates representing the release of both inorganic and organic substances are needed. In this document, ecotoxicological testing is meant to include also genotoxicological testing.

NOTE 4 Granular solid waste materials with a low hydraulic conductivity that can cause detrimental pressure build-up are not supposed to be subjected to this test.

NOTE 5 This procedure is generally not applicable to solids that are easily biologically degradable and solids reacting with the leachant, leading to, for example, excessive gas emission or excessive heat release, impermeable hydraulically bound solids or solids that swell in contact with water.

Granular solid waste materials without potential for beneficial use are excluded from the scope.

NOTE 6 Granular solid waste materials without potential for beneficial use can be materials with gas generation or biodegradation during a potential reuse scenario.

This test is applicable to types of granular solid waste of which the general long-term leaching behaviour is known based on previous investigations.

In this document the same test conditions as for EN 16637-3 (CEN/TC 351/WG 1) are applied in order to allow full comparability of testing construction products and waste derived construction products to avoid double testing. The EN 16637-3 test results are eligible in the context of testing granular solids with potential for use as construction material as well.

NOTE 7 If a leaching test according to EN 16637-3 has been performed, additional EN 17516 testing does not need to be carried out.

NOTE 8 The relative standard deviations for inorganic and organic substances are relatively high which is due to low concentration levels in the eluates (see Annex F).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TR 15310 (all parts), *Characterization of waste — Sampling of waste materials*

EN 933-1, *Tests for geometrical properties of aggregates — Part 1: Determination of particle size distribution — Sieving method*

EN 14405, *Characterization of waste — Leaching behaviour tests — Up-flow percolation test (under specified conditions)*

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

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16637-1, *Construction products: Assessment of release of dangerous substances — Part 1: Guidance for the determination of leaching tests and additional testing steps*

EN 16637-3, *Construction products: Assessment of release of dangerous substances — Part 3: Horizontal up-flow percolation test*

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.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp/>

3.1

eluate

solution obtained from a leaching test

[SOURCE: EN 16687:2023, 3.3.2.8, modified – Note 1 to entry deleted]

3.2

granular solid waste

waste composed of solid particles with a particle size smaller than a specified size or grading

3.3

laboratory sample

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

Note 1 to entry: When the laboratory sample is further prepared by subdividing, cutting, sawing, coring, drying, grinding, mixing, curing 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/analysis.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

[SOURCE: EN 16687:2023, 3.2.2.1]

3.4

leachant

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

Note 1 to entry: Usually, demineralized water is used as leachant for laboratory leaching tests.

[SOURCE: EN 16687:2023, 3.3.2.7]

3.5

leaching behaviour

release and change with time in release from a granular solid waste in contact with a leachant as a function of major release controlling factors

Note 1 to entry: Such factors are diffusion, pH, L/S ratio or time.

[SOURCE: EN 16687:2023, 3.3.2.11, modified to refer to granular waste]

3.6

liquid to solid ratio

L/S

ratio between the total volume of liquid (L) percolated through the granular solid waste and of granular solid waste (S) packed into the column

Note 1 to entry: L/S is expressed in l/kg dry matter.

[SOURCE: EN 16687:2023, 3.3.2.15, modified to refer to granular waste]

3.7

local equilibrium

LE

situation where chemical equilibrium exists between a substance in solution and the same substance in the solid phase at any point in the column

Note 1 to entry: Even when local equilibrium exists at all points along the column the equilibrium concentrations may be different at different points.

[SOURCE: EN 16687:2023, 3.3.2.16]

3.8**percolation test****PT****column test**

release test method to determine the release of substances from a granular solid waste packed in a column with a leachant percolating through it

[SOURCE: EN 16687:2023, 3.3.2.5, modified to refer to granular waste]

3.9**release****emission**

liberation of chemical substances (e.g. non-volatile organic compounds, heavy metals, salts) from a granular solid waste into soil, surface water or groundwater into the leachant of a test facility

Note 1 to entry: Release to soil, surface and groundwater is expressed in terms of mass related release (e.g. mg/kg).

Note 2 to entry: The terms “emission” and “release” have fundamentally the same meaning. However, it is often a tradition to use the term “emission” when describing liberation of chemical substances or radiation into air and to use the term “release” when describing the liberation of chemical substances into soil or water.

[SOURCE: EN 16687:2023, 3.3.2.17, modified to refer to granular waste and mass related release]

3.10**release mechanism**

physical-chemical processes that control the release of substances from a granular solid waste into a leachant

Note 1 to entry: In case of granular solid waste the main release mechanisms are washout and solubility control. Diffusion and additional factors like pH or DOC also have influence on the mechanism of the release.

Note 2 to entry: The release mechanism for every substance can be determined using the results of the release test (tank leaching test, percolation test). Determination of the release mechanism is relevant for modelling of the source term and so for determination of the effects on soil and water over a time period.

[SOURCE: EN 16687:2023, 3.3.2.19, modified – Notes edited to refer to percolation only and to refer to granular waste]

3.11**sample**

portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be prescribed in a sampling plan (3.12).

Note 2 to entry: The term “sample” is often accompanied by a prefix (e.g. laboratory sample, test sample) specifying the type of sample and/or the specific step in the sampling process to which the obtained material relates.

[SOURCE: EN 16687:2023, 3.2.1.5]

3.12

sampling plan

predetermined procedure for the selection, withdrawal, on-site pre-treatment, preservation and transportation of samples to be removed from a population

[SOURCE: EN 16687:2023, 3.2.1.6, modified to remove “if necessary” for waste]

3.13

test portion

analytical portion

amount of the test sample taken for testing/analysis purposes, usually of known weight or volume

[SOURCE: EN 16687:2023, 3.2.2.3, modified to “weight” for waste and examples deleted]

3.14

test sample

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

[SOURCE: EN 16687:2023, 3.2.2.2]

3.15

limit of quantification

LOQ

lowest value of an analyte (determinant) that can be determined with an acceptable level of accuracy and precision, generally determined as three times the limit of detection of the method

[SOURCE: EN 16687:2023, 3.3.1.14]

4 Symbols and abbreviations

4.1 Symbols

For the purposes of this document, the following symbols apply.

c_i	concentration of the substance concerned in the eluate fraction i , in $\mu\text{g/l}$;
D_{max}	sieve diameter, in mm; NOTE Common sizes are 31,5 mm, 45 mm or 63 mm.
d_i	inner diameter of the column, in mm;
d_p	is the density of the solid, expressed in kilograms per cubic metre (kg/m^3).
E_i	released quantity of a substance per quantity of sample for analysis in eluate fraction i , in mg/kg dry matter;
h	is the height or layer thickness of the construction, in m;
k_c	constant that represents the speed of the release of a certain substance;
k_e	number of eluates collected in the test;
m_d	dry mass of the test portion, in g;

m_r	mass of the undried test portion, in g;
m_W	mass of the (moist) test portion in the column, in g;
S_{cs}	sieve diameter for test sample preparation (crushing) to reduce the amount of oversized part of the sample to fit the inner column diameter, in mm;
t	execution time of the test, in days;
U_n	measured cumulative release of a substance for cumulative L/S ratio n including fraction $i = 1$ to n , in mg/kg dry matter;
V_i	volume of the eluate fraction i , in l;
V_L	linear velocity of the leachant through the empty column, in mm per day;
w_{dr}	dry residue of the granular solid waste, in %;
ϕ	leachant flow rate, in ml per hour.

4.2 Abbreviations

For the purposes of this document, the following abbreviations apply.

DL	detection limit;
DOC	dissolved organic carbon;
EC	electrical conductivity;
EOTA	European organization for technical assessment;
ETA	European technical assessment;
ETFE	ethylene tetrafluoroethylene;
FEP	fluorinated ethylene propylene;
FPC	factory production control;
GLHC	test method for granular products with low hydraulic conductivity;
HDPE	high-density polyethylene;
hEN	harmonized European Standard;
PAH	polycyclic aromatic hydrocarbon;
PCTFE	polychlorotrifluoroethylene;
PT	percolation test;
PTFE	polytetrafluorethylene;
RDS	regulated dangerous substances;
TPH	total petroleum hydrocarbons.

5 Principle

5.1 General principles

The percolation test described in this document starts with the representative laboratory sample. The methodology for the collection of a representative laboratory sample is in accordance with the procedures described in EN 17087 and EN 15002 considering guidance given in EN 16637-1.

This document describes a method to determine the release of substances from a granular solid waste with potential for beneficial use as construction product, with or without size reduction to a maximum particle size, packed in a column with a leachant percolating through it. Pre-equilibration is applied to approach (local) equilibrium at the start. The column size is related to the amount of eluate needed for subsequent analysis and testing and the size of the largest particles in the test portion. 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 substances are rapidly being washed out and which substances are released under the influence of interaction with the matrix. The method is a once-through column leaching test. It is assumed that conditions approach local equilibrium between the granular solid waste and leachant (for inorganic substances) in the test.

NOTE The results obtained under local equilibrium (LE) can be up-scaled and used in the modelling of in-use conditions for various scenarios.

The eluate is collected in fractions that are characterized physically and chemically and possibly ecotoxicologically according to existing standards. The results of the test are expressed as a function of the L/S ratio, in terms of mg of the substances released cumulatively per kg of granular solid waste or of mg of substance determined per litre of eluate.

The procedure described in this document is based on the more stringent test requirements for determining the release of organic substances and/or for subsequent ecotoxicological testing. If only the release of inorganic substances is to be measured, requirements on equipment (e.g. column and tubing material, centrifugation) are specified in the relevant clauses for some steps of the procedure.

The test procedure described in this document constitutes a compliance test used to verify compliance to regulatory limit values. The column percolation tests according to EN 14405 shall be applied for basic characterization of granular solid waste. The column percolation tests according to EN 16637-3 shall be applied for basic characterization of construction products.

5.2 Number of eluates

By reference the percolation test requires the analysis of seven eluates to determine the release of dangerous substances for an L/S ratio up to 10. These results are needed for characterization of the granular solid waste and allow the determination of the release mechanism according to Annex E.

For specific scenarios, other than characterization, it may be beneficial to combine eluates or adopt other testing schemes based on the one of this document. Examples of such scenarios are:

- a) *Release at a specific L/S ratio.* If it is sufficient to know the cumulative release at a predetermined L/S ratio, for instance $L/S = 2$, the test can be stopped after the collection of the fifth eluate fraction.
- b) *Factory production control.* In the case of FPC it is checked whether the product conforms to the previously determined characteristics (type testing). Different test methods may be used:
 - in the case of testing in accordance with the standard it is sufficient to check the release for the specified L/S ratio and to collect and combine successive eluates in proportion to the volumes, e.g. L/S (0 to 2) l/kg dry matter or L/S (0 to 10) l/kg dry matter;

- following an alternative testing procedure (indirect test method), the release at for instance L/S (0 to 0,5) l/kg dry matter or L/S (0 to 1) l/kg dry matter, may be extrapolated to predict the release after L/S (0 to 10) l/kg dry matter or another appropriate L/S ratio. Also, other leaching tests may be used.

As long as the adapted test scheme is based on the one of this document and the test is performed according to this document, the cumulative release at a specified L/S ratio is equal to the cumulative release based on the reference test performance. The release mechanisms specified in Annex E of this document cannot be determined if less than seven eluates are analysed.

6 Reagents

6.1 General

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

6.2 Leachant

Use as a leachant demineralized water or deionized water or water of equivalent purity with a conductivity $< 0,5$ mS/m or better.

When the release of biodegradable organic compounds is studied, the eluate might be stabilized with a preservative in order to avoid biodegradation.

For ecotoxicological tests, preservatives are not allowed.

6.3 Rinsing solutions

Use as rinsing solution nitric acid, $c(\text{HNO}_3) = 0,1$ mol/l and an organic solvent (e.g. acetone). Make sure that the quality grade of the nitric acid is appropriate for analysis of trace elements.

7 Equipment

7.1 General

Check the materials and equipment specified in 7.2 to 7.17 before use for proper operation and absence of interfering substances that may affect the result of the test.

The equipment specified under 7.6, 7.7, 7.8, 7.14 and 7.15 shall be calibrated.

7.2 Column

Column made of plastics or glass for inorganic substances and glass or stainless steel for organic substances with an inner diameter d_i larger than 50 mm and a length that can accommodate a filling height of $300 \text{ mm} \pm 50 \text{ mm}$. In top and bottom of the column a filter plate, glass beads or a thin layer of quartz sand with a particle size range of 1,0 mm to 2,5 mm is applied to ensure proper water flow over the total width of the column.

Filter plates, quartz sand (SiO_2 -content at least 98 % by mass) or glass beads can be washed but where heated to remove any adsorbed 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 In some cases, it is useful to place a pre-filter at least in the bottom section to prevent the material from leaving the column through the water inlet duct.

A drawing of the column and accompanying equipment is given in Annex C.

Glass of high quality is usually considered adequate for both metals and organic contaminants, particularly, since the pH range usually covered in granular solid waste testing does not reach the conditions where glass itself is attacked. For ecotoxicity testing, eluates with both metals and organic contaminants are needed, which emphasizes the need to generate integrated eluates.

NOTE 2 Usually borosilicate glass is appropriate, unless boron has to be analysed. Sodium glass can change the pH of the eluate.

In case of organic substances to be analysed, it shall be checked that the material does not interfere significantly with the substances to be measured.

7.3 Packing equipment, Rammer with a weight of 125 g in the case of a column with a diameter of 50 mm up to 500 g for a column with a diameter of ≥ 100 mm. Intermediate weights to be linearly interpolated by surface area.

NOTE The limited contact of the rammer is considered not to affect the test results.

7.4 Membrane for off-line filtration of eluates to be analysed for inorganic substances, with a pore size of 0,45 μm (e.g. cellulose acetate, PTFE).

Membranes shall be of inert material and not absorb or adsorb compounds of interest. Filters shall not be used for eluates to be analysed for organic substances.

7.5 Volumetric pump, with an adjustable capacity to be suitable to allow for the flow rate specified according to 9.4.3.

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

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

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

7.9 Tubing material, inert and adapted to the substances to be analysed. In case organic substances are to be analysed, stainless steel or glass and FEP can be used in contact with the eluate. In case inorganic substances are analysed PE, PP, PTFE, ETFE, FEP or similar tubing materials can be used.

NOTE Examples of appropriate tubing material are given in EN ISO 5667-3.

7.10 Eluate bottles

For eluate collection and preservation of eluate samples, use high quality glass bottles with an appropriate volume and with screw cap with PTFE, ETFE or PTFE inlay (in accordance with EN ISO 5667-3). If only inorganic contaminants are to be analysed, alternative bottle materials can be selected, e.g. PE or PP.

NOTE Usually borosilicate glass is appropriate, unless boron has to be analysed. Sodium glass can change the pH of the eluate.

7.11 Crushing equipment

Jaw crusher or a cutting device. The crushing device shall be cleaned thoroughly to avoid cross contamination from previous use.

NOTE Due to crushing, contamination of the sample can occur to an extent which affects the leaching of some constituents of concern e.g. cobalt and tungsten from tungsten carbide equipment or chromium, nickel and molybdenum from stainless steel equipment.

7.12 Sieving equipment (dry sieving), e.g. with sieves 4 mm, 11,2 mm and 22,4 mm nominal screen size.

7.13 Sample splitting equipment

For sub-sampling of laboratory samples, e.g. riffle divider or dividing cross for coning and quartering (optional).

7.14 Redox potential meter (optional)

7.15 Turbidity meter, as specified in ISO 7027-1 [28].

7.16 Centrifuge

Use a centrifuge operating at 20 000 *g* to 30 000 *g* using centrifuge tubes of FEP or tubes of an alternative material, which is inert with regard to both inorganic and organic substances and suitable to high-speed centrifugation.

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

7.17 Filtration

Vacuum filtration device 30 kPa to 70 kPa or pressure filtration device ($\leq 0,5$ MPa).

8 Sample preparation

8.1 General

Sample preparation shall consist of preparation of the test sample from the laboratory sample, preparing a test portion and determining the dry residue of the test sample.

Sampling shall be performed in accordance with the procedures described in the CEN/TR 15310 series and EN 15002, considering guidance given in EN 16637-1 in order to obtain a representative laboratory sample.

NOTE As usual in an accreditation procedure, the testing laboratory reports on sampling only when it performs sampling itself to produce laboratory sample.

8.2 Preparation of the test sample

8.2.1 Principles

A laboratory sample shall be received whose minimum amount depends on the maximum particle size of the granular solid waste, the column diameter chosen and the number of tests to be carried out. The laboratory sample shall be stored in closed packages and kept under conditions which prevent any alterations of the sample.

The laboratory selects the appropriate column (7.2) and determines the sieve diameter S_{cs} for the test sample (see Table 1).

Crushing of the granular solid waste shall be applied no more than absolutely necessary in order to minimize the effect on the waste properties, i.e. crushing is only needed in case of an oversized fraction larger than S_{cs} (see Table 1) or to fill a lack of fines less than 4 mm.

At least 45 % by mass of the test sample shall have a particle size < 4 mm. If the grading of the laboratory sample already contains 100 % by mass particles $< S_{cs}$ (see Table 1) and an amount of particles of at least 45 % by mass < 4 mm, no further crushing or sieving is allowed.

The choice of column width depends on the maximum particle size of the solids to be tested (S_{cs}).

NOTE The choice of column width may also depend on equipment available in the laboratory, the mass of the laboratory sample or the amount of eluate required for chemical analysis or ecotoxicological testing.

No part of the laboratory sample shall be discarded during the sample preparation.

8.2.2 Rules of application

The following stepwise procedure to prepare the test sample shall be applied to fulfil the principles specified in 8.2.1:

- a) If the laboratory sample already contains 100 % by mass of particles $< S_{cs}$ (see Table 1) and an amount of particles of at least 45 % by mass < 4 mm, no further crushing or sieving is allowed (see Annex B for an example).
- b) If the laboratory sample contains an oversized fraction larger than S_{cs} , gently crush this fraction to obtain a test sample with all particles $\leq S_{cs}$ (result: test sample with a maximum particle size to fit the chosen column size).
- c) Crusher jaws should be adjusted to avoid large amounts of particles < 4 mm.
- d) Depending on the number and size of the test portions, the further procedure may be carried out on a representative subsample (see EN 932-1, EN 15002 and EN 16179).
- e) Check by sieving on a 4 mm sieve whether test sample contains an amount of particles < 4 mm larger than 45 % by mass. If this condition is fulfilled the test sample is ready to take test portions for the percolation test.
- f) If the test sample does not contain an amount of at least 45 % by mass passing the 4 mm sieve after step d), gently crush a sufficient and representative portion of $4/S_{cs}$ mm (see Annex B for an example) to an extent that after crushing the completely prepared (recombined) grading contains an amount of particles < 4 mm between 45 % by mass and 55 % by mass. If this condition is fulfilled, the test sample is ready to take test portions for the percolation test.

Table 1 — Column size and corresponding sieve diameter S_{cs}

Inner column diameter d mm	Sieve diameter for test sample preparation (crushing) to reduce the amount of oversized part of the sample to fit the inner column diameter S_{cs} mm
50 – 100	16
70 – 100	22,4
$d \geq 100$ and $d \geq 3D_{max}$	D_{max}^a
^a If the sample contains at least 45 % by mass particles < 4 mm, no crushing is allowed. If this condition does not apply, set S_{cs} to 22,4 mm.	

The particle size distribution of the original granular solid waste should be reported, if available. Regarding the test sample, the maximum particle size and the percentage of particles < 4 mm shall be reported.

8.3 Test portion

At least three test portions shall be taken from the test sample (one for determination of the dry residue, and one for the percolation test itself). The test portions shall be subsampled in line with 8.5.

One test portion shall be reserved to enable repetition in case of failure.

NOTE It is often considered good practice to perform the test in duplicate or triplicate (which would require additional test portions) especially when an inherent heterogeneity of the test material is expected.

The amount of the test portion for the column is depending on the diameter of the column, the packing height, the density of the granular solid waste and the porosity of the packing.

8.4 Drying

If the sample cannot be sieved or crushed, due to its moisture content, it may be dried, but no further than is required for the preparation of the test sample. Freeze drying or drying at room temperature is preferred to oven drying. The drying temperature shall not exceed 40 °C.

NOTE Drying could alter the properties of the sample (e.g. oxidation and/or carbonation). Drying conducted in an inert atmosphere could prevent alteration of the sample properties. Guidance on drying of samples is given in EN 15002 and EN 16179 [8].

8.5 Subsampling

A sample splitter (7.13) shall be used or coning and quartering shall be applied to divide the sample and prepare subsamples.

NOTE Guidance on subsampling is given in EN 15002, EN 16179 [8] and EN 932-1 [9].

8.6 Sieving of samples into fractions

Sieving of samples into particle fractions shall be done by dry sieving only, according to EN 933-1.

8.7 Size reduction of particles

If oversized particles are to be size reduced, the entire oversized fraction shall be crushed with a crushing device (7.11) to grains smaller than the maximum particle size (S_{cs}) required for the test portion. Crushing shall proceed no further than is required for the sample preparation. Heating, loss of dust or contamination from the equipment shall be minimized. On no account, the material shall be finely ground. Non-crushable material (e.g. metallic particles) shall be separated, weighed and the nature of the material recorded.

NOTE Plastic or bituminous samples could be size-reduced after cryogenic treatment. Guidance on particle size reduction is given in EN 15002 and EN 16179 [8].

9 Test procedure

9.1 Temperature

The percolation test shall be carried out at a temperature between 19 °C and 25 °C.

NOTE 1 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.

For waste materials releasing substances that are sensitive to photodegradation, the column and the collection vessels shall be prevented from direct exposure to light, e.g. by wrapping in aluminium foil.

When necessary the vessel(s) for collection of eluate shall be kept cool (e.g. at 4 °C) to limit the degradation of the substances in the eluates. If the test is modified in this way, this deviation shall be reported in the test report.

NOTE 2 For information about whether a substance is photosensitive and sensitive to degradation, see ISO 5667-3.

9.2 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 15934, on a separate test portion. Calculate the dry residue in accordance with Formula (1).

$$w_{dr} = m_d \times \frac{100}{m_r} \% \quad (1)$$

where

w_{dr} is the dry residue of the sample, 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.

9.3 Pre-treatment of the column

Rinse the column with top and bottom section and filters (7.2) and bottles (7.10) with diluted nitric acid (6.3) and/or an organic solvent and water (6.2) respectively. In case of organics acetone should be used. Weigh the dry column, including top and bottom filter plates or layers of sand, to an accuracy of 1 g. Alternatively, a heat treatment (e.g. with a temperature between 450 °C and 500 °C) can be applied to clean temperature resistant equipment of adsorbed organic contaminants. Quartz sand and filter layers shall not be heated to red-heat.

9.4 Packing of the column

9.4.1 General

The column shall be packed as evenly and uniformly as possible, avoiding segregation of different particle sizes. This is generally best accomplished by packing the granular solid waste in the column in stages. Annex D provides packing procedures.

9.4.2 Equilibration

Saturate by hydrostatic pressure (preferably for no more than 6 h) or by pumping (preferably for $4 \text{ h} \pm 2 \text{ h}$).

NOTE See D.4 for a description of the methods of saturation.

Stop the pump, or take away the hydrostatic pressure, when the material in the column is completely saturated, but the outlet hose remains empty. Leave the saturated material for a period between 12 h and 72 h in order to equilibrate the system.

9.4.3 Calculation of the flow rate

After the equilibrium period, start the pump (again) and set the flow rate ϕ such that it corresponds to a linear velocity (V_L) of (300 ± 40) mm per day, calculated for the empty column. Calculate the flow rate in accordance with Formula (2).

$$\phi = V_L \times \pi \times d_i^2 \times 10,4 \times 10^{-6} \quad (2)$$

where

ϕ is the leachant flow rate, in ml per hour;

V_L is the linear velocity of the leachant through the empty column, expressed in mm per day;

d_i is the inner diameter of the column, in mm.

NOTE 1 A linear velocity of 300 mm per day corresponds to a flow rate of 24,5 ml per hour for a column with a diameter of 50 mm. For a column with a diameter of 100 mm, it is equivalent to a flow rate of 98 ml per hour.

NOTE 2 The parameter $10,4 \times 10^{-6}$ includes the factor 0,25 from the calculation of column volume times 0,001 to convert from mm^3 to ml and times the factor $1/24$ to convert from days to hours. Using this factor, the calculated flow rate is expressed in a common unit (ml per hour).

9.5 Collection of eluates

Connect the outlet hose (7.9) to an eluate collection bottle of appropriate size (7.10). Start the pump again and change the collection bottle after a quantity of $(0,1 \pm 0,02)$ times the mass of the test portion (m_d) of leachant has been collected (fraction 1 according to Table 2).

Measure the pH (7.7) and the electrical conductivity (7.8) in accordance with CEN/TR 16192 or EN 17195 and redox potential (7.14, optional) of the eluate. If the pH value of the eluate is higher than 9, or if a later rise in pH is expected, it may be necessary to keep the eluate collection bottle and the following ones under inert (argon or nitrogen) atmosphere or another valid technique to prevent carbonation and precipitation. 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. Refill gas every time the collection bottle is changed. This procedure is repeated for each eluate fraction.

NOTE 1 An air lock could be needed on the lid to avoid back-pressure in the column in case the bottle with lid is gas tight.

NOTE 2 Keeping the collection bottles under inert atmosphere could also be required in case of investigating materials with reducing properties to prevent the occurrence of oxidation reactions.

NOTE 3 The pH, EC, DOC (optional) and redox measurements can be used for verification purposes in case measurements deviate from the expected release behaviour. This will provide the user with the option to accept the results, verify deviations from normal or take corrective measures based on an understanding of the cause of the deviation.

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 (7.16);
- b) there are two options for solid-liquid separation:

centrifuge the eluate for 30 min at 20 000 *g* to 30 000 *g* using a high-speed centrifuge (7.16);

centrifuge the eluate for 5 h at 2 000 *g* to 2 500 *g* in glass bottles using a lower speed centrifuge (7.16).

NOTE 4 Limitation of the maximum centrifugation duration is especially necessary for organic substances. The speed or *g*-force of the centrifuge is dependent on the specific rotor.

NOTE 5 Background information on sorption effects on container walls is given in "Sorption effects interfering with the analysis of polycyclic aromatic hydrocarbons (PAH) in aqueous samples", see [10].

Cooling of the centrifuge shall be applied to maintain the temperature between 19 °C and 25 °C.

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

If only leaching of inorganic substances and DOC 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 (7.4) and a vacuum or pressure filtration device (7.17).

Check the flow rate of the leachant as often as needed (at least after collection of each eluate or three times per week whichever is most frequent) and adjust to the original linear velocity (300 ± 40) mm per day.

Change the collection bottles with a new one as soon as a quantity of water (6.1) 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.

At each eluate collection point, measure time and volume of the eluate fraction and calculate the *L/S* ratio and the average flow velocity of the leachant over the collection period of that fraction. Also, measure the actual flow velocity.

NOTE 6 An automatic eluate collection system can be used.

For samples with low density the volume collected in each of the first two fractions may not be sufficient for chemical analysis. In such cases, the first two volumes may be combined to one fraction prior to chemical analysis.

Table 2 — Table for collection of eluate fractions

Fraction number	Fraction volume ^{a b} l	Cumulative <i>L/S</i> ratio l/kg
1	$(0,1 \pm 0,02) \times m_d$	0,1
2	$(0,1 \pm 0,02) \times m_d$	0,2
3	$(0,3 \pm 0,05) \times m_d$	0,5
4	$(0,5 \pm 0,05) \times m_d$	1,0
5	$(1,0 \pm 0,05) \times m_d$	2,0
6	$(3,0 \pm 0,1) \times m_d$	5,0
7	$(5,0 \pm 0,2) \times m_d$	10,0
^a Numbers in parentheses are the <i>L/S</i> ratios of the fractions in l/kg. ^b Because the density of the eluates is usually close to 1 g/ml, mass can be used instead of volume.		

The test itself is finished when the cumulative *L/S* ratio of $(10 \pm 0,5)$ l/kg dry matter is reached.

For specific scenarios it may be sufficient to know the leaching characteristics up to a pre-determined *L/S* ratio, for instance *L/S* = 2. In that case the test can be stopped after the collection of the fifth eluate fraction or the collection can be performed as one eluate up to *L/S* = 2. In other cases, e.g. for materials functioning as drains, scenario descriptions may require much higher values of the *L/S* ratio. If that is the case, additional fractions may be collected until the relevant range of *L/S* ratios has been covered.

NOTE 7 For ecotoxicological testing, the fractions 1 to 5 (or parts of these fractions) can be combined to receive a cumulative *L/S* ratio of 2. If large amounts of eluates are needed for testing, eluates of the same fractions of several percolation tests can be combined.

NOTE 8 In case no automated eluate collection apparatus is available, a collection scheme can be composed within the ranges that are allowed in flow rate (e.g. (300 ± 40) mm per day) and in fraction volume (see Table 2) that enables eluate collection within working hours.

NOTE 9 The execution time of the test can be calculated from Formula (3).

$$t = \frac{(L / S) \times m_d \times 1\,000}{24 \times \phi} \quad (3)$$

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_d is the dry mass of the test portion, in kg;
- ϕ is the leachant flow rate, in ml per hour.