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BRITISH STANDARD

BS 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)

The European Standard EN 12457-2:2002 has the status of a British Standard

ICS 13.030.10; 13.030.20

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British Standards

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National foreword

This British Standard is the official English language version of EN 12457-2:2002.

The UK participation in its preparation was entrusted by Technical Committee B/508, Waste management, to Subcommittee B/508/3, Characterisation of waste, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

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This British Standard, having been prepared under the direction of the Building and Civil Engineering Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 15 October 2002

Summary of pages

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EUROPEAN STANDARD
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EN 12457-2

September 2002

ICS 13.030.10; 13.030.20

English version

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)

Caractérisation des déchets - Lixiviation - Essai de conformité pour la lixiviation des déchets fragmentés et des boues - Partie 2: Essai en bâchée unique avec un rapport liquide-solide de 10 l/kg et une granularité inférieure à 4mm (sans ou avec réduction de la granularité)

Charakterisierung von Abfällen - Auslaugung - Übereinstimmungsuntersuchung für die Auslaugung von körnigen Abfällen und Schlämmen - Teil 2: Einstufiges Schüttelverfahren mit einem Flüssigkeits-/Feststoffverhältnis von 10 l/kg für Materialien mit einer Korngröße unter 4 mm (ohne oder mit Korngrößereduzierung

This European Standard was approved by CEN on 17 August 2002.

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 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 Management Centre has the same status as the official versions.

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Contents

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Foreword

This document (EN 12457-2:2002) has been prepared by Technical Committee CEN /TC 292, "Characterisation of waste", 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 June 2003, and conflicting national standards shall be withdrawn at the latest by June 2003.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This document has been developed primarily to support the requirements for compliance testing within the EU and EFTA countries.

This document was elaborated on the basis of :

DIN 38414-S4:1984

AFNOR X-31 210:1992

NEN 7343:1992

ÖNORM S 2072:1990

No existing European Standard is superseded by the publication of this European Standard.

Annexes A, B, C, D and E are informative.

This document includes a Bibliography.

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Introduction

In the different European countries, tests have been developed to characterise and assess the constituents which can be leached from waste materials. The release of soluble constituents upon contact with water is regarded as a main mechanism of release which results in a potential risk to the environment during the reuse or disposal of waste materials. 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 standard. Tests to characterise waste materials and their behaviour can generally be divided into three categories :

> (1) "Basic Characterisation" tests are used to obtain information on the short and long term leaching behaviour and characteristics properties of waste materials. Liquid/solid (L/S) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters are addressed in these tests ;
>
> (2) "Compliance" tests are used to determine whether the waste complies with specific reference values. The tests focus on key variables and leaching behaviour identified by basic characterisation tests ;
>
> (3) "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 the compliance test(s).

The procedures described in this European Standard fall in category 2 : compliance tests.

The four procedures described in the four following European Standards are based on different liquid to solid (L/S) ratios and different particle sizes because these parameters, among others, play an important role in the leaching process:

EN 12457-1, *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).*

EN 12457-2, *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).*

EN 12457-3, *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).*

EN 12457-4, *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).*

Each part specifies a distinct procedure. The specific features of each part are described in the scope and principle of each part. For given wastes the results can be different for the different procedures. There are six annexes to this European Standard giving useful information on the selection of the appropriate procedure, reference documents and guidance on the limitations of these procedures.

The choice of the procedure depends on the degree and type of information required for compliance testing. This choice has to be made by the organisation establishing the compliance requirements.

This European Standard specifies a compliance test. For basic characterisation, a methodology for the determination of the leaching behaviour of waste has been developed within TC 292 and formulated in ENV 12920.

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1 Scope

This part of four European Standards specifies a compliance test providing information on leaching of granular wastes and sludges under the experimental conditions specified hereafter, and particularly a liquid to solid ratio of 10 l/kg dry matter. It applies to waste which has a particle size below 4 mm without or with size reduction (as specified in 4.3.2)

This European Standard has been developed to investigate mainly inorganic constituents from wastes. It does not take into account the particular characteristics of non-polar organic constituents nor the consequences of microbiological processes in organic degradable wastes.

The test procedure specified in this European Standard produces an eluate which shall subsequently be characterised physically and chemically according to appropriate standard methods.

NOTE 1 This procedure cannot be applicable to materials with a water content or such a water affinity that a good mixing of the solid with the predetermined quantity of liquid is not achievable.

NOTE 2 This procedure cannot be applicable to materials reacting with the leachant, leading, for example, to excessive gas emission, a solidifying effect or an excessive heat release.

NOTE 3 By crushing the material, new surfaces are exposed which may lead to a change in leaching properties.

This test cannot be used alone to determine the leaching behaviour of a waste, as specified in ENV 12920 .

This European Standard does not address issues related to health and safety.

2 Normative references

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

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

ENV 13370, *Characterization of waste – Analysis of eluates – Determination of Ammonium–N, AOX, conductivity, Hg, phenol index, TOC,* CN^- *easily liberatable,* F^-.

EN 12880, *Characterisation of sludges – Determination of dry residue and water content.*

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*.

EN ISO 5667-3, *Water Quality - Sampling - Part 3 : Guidance on the preservation and handling of samples (ISO 5667-3:1994).*

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

ISO 11465, *Soil Quality - Determination of dry matter and water content on a mass basis - Gravimetric method*.

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3 Terms and definitions

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

3.1
leaching test
test during which a material is put into contact with a leachant and some constituents of the material are extracted

3.2
leachant
liquid used in a leaching test

NOTE For the purpose of this European Standard the leachant is water as specified in 4.2.15

3.3
eluate
solution recovered from a leaching test

3.4
single batch leaching test
leaching test in which a fixed amount of material is leached in one step with a fixed amount of leachant

3.5
serial batch leaching test
leaching test for two or more subsequent extractions of the same portion of material with a fresh amount of leachant

3.6
liquid to solid ratio L/S (abbreviation : L/S)
ratio between the total amount of liquid (L in litre), which in a leaching test is in contact with the waste, and the dry mass of the sample (S in kg of dry matter) abbreviated L/S and expressed in l/kg

3.7
liquid waste
waste that completely flows out of a calibrated opening, down to the upper level of the opening within a limited period of time less than 8 h (annex B informative)

3.8
sludge
mixture of liquid and solids separated from various types of liquids as a result of natural or artificial processes

NOTE In the field of water treatment, the prevailing standards (EN 1085 and EN 12880) use the word water instead of the word liquid.

3.9
monolithic waste
material which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time

3.10
granular waste
waste that is neither monolithic, liquid, gas nor sludge

3.11
dry matter content ratio (abbreviation : DR)
ratio expressed in percent between the mass of the dry residue, determined according to ISO 11465, or EN 12880 for sludges, and the corresponding raw mass

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3.12
moisture content ratio (abbreviation MC)

ratio expressed in percent between the mass of water contained in the material and the corresponding dry mass of the material

NOTE The basis for the calculation of the moisture content is the mass of the dry residue in this European Standard, as specified in ISO 11465 (for the determination of the water content of soil). It is to be noted that in EN 12880 (for the determination of water content of sludges), the water content is calculated on the basis of the raw mass.

3.13
laboratory sample

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

NOTE 1 When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding 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 sample collection but it is the initial sample from the point of view of the laboratory.

NOTE 2 Several laboratory samples can 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.

3.14
test sample

sample, prepared from the *laboratory sample,* from which *test portions* are removed for testing or for analysis (IUPAC definition)

3.15
test portion

amount or volume of the *test sample* taken for analysis, usually of known weight or volume (IUPAC definition)

4 Compliance test

4.1 Principle

The sample material, which originally or after pre-treatment has a particle size below 4 mm as defined in 4.3.2, is brought into contact with water (4.2.15) under defined conditions. This European Standard is based on the assumption that equilibrium or near-equilibrium is achieved between the liquid and solid phases during the test duration (see 5.2.1). The solid residue is separated by filtration. The properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluates (ENV 12506, ENV 13370 and others under development).

After the test the leaching conditions in terms of pH, conductivity and optionally redox potential dictated by the waste are recorded.

NOTE These parameters often control the leaching behaviour of wastes and are therefore important for checking the leaching test.

4.2 Equipment and reagents

4.2.1 Glass or high density polyethylene(HDPE)/polypropylene(PP) bottles in accordance with EN ISO 5667-3, with a nominal volume of 1 litre, glass bottles having caps of inert material, for example PTFE (polytetrafluoroethylene). Rinsing is compulsory.

NOTE 1 For inorganic constituents HDPE/PP bottles are preferred, except for samples analysed for mercury.

NOTE 2 The volume of 1 litre is selected in combination with the mass M_D of 90 gram as specified in 4.3.4 in order to minimise head space. In case of material with low density, deviation from this requirement can be necessary while still attempting to minimise headspace. This deviation should be documented in the test report.

4.2.2 An end-over-end tumbler (5 rpm - 10 rpm) or roller-table inducing rotation of the bottle at about 10 rpm.

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NOTE Other shaking or mixing devices can be used provided that they are proven to be equivalent.

4.2.3 Filtering apparatus, either a vacuum filtration device (between 30 kPa and 70 kPa) (300 mbar to 700 mbar) or a high pressure filtration apparatus (< 0,5 MPa) (5 bar). Rinsing is compulsory.

NOTE 1 Water ejection pump generally operates in the vicinity of 50 kPa.

NOTE 2 When volatile components are to be analysed, vacuum filtration should not be used.

4.2.4 Pre-rinsed or similarly clean 0,45 µm membrane filters for filtration (e.g. rinsed with 0,1 mol/l HNO_3 as described in 4.2.16 and water as described in 4.2.15).

NOTE Depending on the test requirements, PTFE filter material can be required. Filter made of PTFE are hydrophobic. Before filtration of eluate, they should be wetted using ethanol or methanol provided it doesn't influence the results of analysis.

4.2.5 Crushing equipment : jaw crusher or cutting device.

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.

4.2.6 Sieving equipment with sieves of 4 mm nominal screen size.

NOTE Due to sieving, 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.

4.2.7 A centrifuge operating between 3 000 and 4 000 g.

4.2.8 A device for measuring electrical conductivity.

4.2.9 A pH meter according to ENV 12506.

4.2.10 Two thermometers for air and leachant temperature measurement.

4.2.11 A redox potential meter (optional).

4.2.12 A balance with accuracy of at least 0,1 g.

4.2.13 Measuring cylinders for volume determination with 1 % accuracy.

4.2.14 A sample splitter or utensils for cone and quartering for sub-sampling of test samples (optional).

4.2.15 Distilled water, demineralised water, de-ionised water or water of equivalent purity (5 < pH < 7,5) with a conductivity < 0,5 mS/m according to grade 3 specified in EN ISO 3696.

4.2.16 Nitric acid (HNO_3) 0,1 mol/l (analytical grade).

4.3 Sampling strategy and sample preparation

4.3.1 Sampling strategy

Obtain a laboratory sample of at least 2 kg of the material. Use a sample splitter (4.2.14) or apply coning and quartering to split the sample.

NOTE 1 Sampling should have been performed according to the framework for the preparation of a sampling plan for liquid and granular waste materials including paste-like materials and sludges, under development by CEN/TC 292, in order to obtain a representative laboratory sample.

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

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4.3.2 Particle size reduction

The tests shall be made on material with a grain size of at least 95 % (mass) less than 4 mm. Therefore the laboratory sample shall be sieved (4.2.6). If oversized material exceeds 5 % (mass) the entire oversized fraction shall be crushed with a crushing equipment (4.2.5). On no account shall the material be finely ground. Non-crushable material (e.g. metallic parts such as nuts, bolts, scrap) in the sample shall be separated and the weight and nature of the material shall be recorded. The method of size-reduction applied shall be documented and recorded in the test report. Irrespective of any necessary size reduction, the separate fractions with the exception of the non-crushable material and the material that may be used according to note under 5.4, shall be mixed to constitute the test sample. If the laboratory sample cannot be crushed or sieved because of its moisture content, it is allowed, only in this case, to dry the laboratory sample. The drying temperature shall not exceed 40 °C.

NOTE 1 Fibrous materials and plastics can often be size-reduced after cryogenic treatment.

NOTE 2 Any drying step can change other properties of the waste. Care should be taken to minimise such changes

NOTE 3 In order to minimise a possible contamination during sieving, fragmentation and splitting, it is recommended, before preparing the test sample, to process a portion of the laboratory sample through the devices for sieving, fragmentation, splitting, and to discard such material thereafter. This recommendation does not cover the possible contamination described in the notes under 4.2.5 and 4.2.6.

NOTE 4 Important differences may occur in the leaching test results for a given material depending on the crushing procedure and the waste material being crushed. Particle size related differences may be made evident by determining the particle size distribution. It is to be noted that in the case of very narrow size distribution, such differences in the leaching result may be enhanced especially in the upper part of the size range.

4.3.3 Determination of the dry matter content ratio and the moisture content ratio

The whole test sample, complying with the size criterion in 4.3.2, shall not be further dried. The mass of the dry residue shall be determined at 105 °C ± 5 °C according to ISO 11465 or for sludges according to EN 12880. This determination shall be made in parallel on a test portion different from the test portion for leaching. The dry matter content ratio is calculated as follows :

$$DR = 100 \times M_D/M_W \tag{1}$$

where

DR is the dry matter content ratio (%) ;

M_D is the mass of the dried test portion (kg) ;

M_w is the mass of undried test portion (kg).

The moisture content ratio (MC in %) is calculated as follows :

$$MC = 100 \times (M_W - M_D)/M_D \tag{2}$$

NOTE 1 The basis for the calculation of the moisture content is the mass of the dry residue in this European Standard, as specified in ISO 11465 (for the determination of the water content of soil). It is to be noted that in EN 12880 (for the determination of water content of sludges), the water content is calculated on the basis of the raw mass.

NOTE 2 The above moisture content determination could be not accurate enough in some cases (e.g. large amount of volatile or unstable compounds). In such cases a direct determination of the true water content should be performed and the moisture content calculated accordingly.

4.3.4 Preparation of test portion

Prepare from the test sample a test portion with a total mass M_W (measured with an accuracy of 0,1 g) containing 0,090 kg ± 0,005 kg of dry mass (M_D). Use a sample splitter (4.2.14) or apply coning and quartering to split the sample.

$$M_W = 100 \times M_D/DR \tag{3}$$

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where

DR is the dry matter content ratio (%) ;

M_D is the mass of the dried test portion (kg) ;

M_w is the mass of undried test portion (kg).

NOTE In view of the minimum requirements of sample volume for analytical purposes, it can be necessary to use a larger test portion size and a correspondingly larger volume of leachant. This deviation from this European Standard should be documented in the test report.

5 Procedure

5.1 Testing conditions

The compliance test for leaching shall be carried out at room temperature (20 ± 5) °C.

5.2 Description of the procedure

5.2.1 Leaching step

- Place the test portion with the total mass M_w corresponding to 0,090 kg ± 0,005 kg of dry mass M_D in a bottle (4.2.1).
- Add an amount of leachant (L) establishing a liquid to solid ratio (L/S) = 10 l/kg ± 2 % during the extraction. Care should be taken to obtain good mixing of solid and liquid.

$$L = (10 - MC/100) \times M_D \quad (4)$$

Where

L is the volume of leachant used (in l) ;

M_D is the dry mass of the test portion (in kg) ;

MC is the moisture content ratio (in %).

- Place the capped bottle in an agitation device (4.2.2).
- Agitate for 24 h ± 0,5 h.
- During the extraction care should be taken to prevent settlement of solids in the bottle.
- Excessive abrasion leading to significant particle size reduction shall be avoided.

NOTE Some wastes generate gas when they are wetted. Examples are waste incineration fly ash and sand blasting waste which may contain metallic particles. If gas emission occurs, careful opening of the bottle a few times during the leaching can prevent too high pressure. Such opening should be documented in the test report.

5.2.2 Liquid-solid separation step

- Allow the suspended solids to settle for 15 min ± 5 min.
- Filter the eluate almost completely over a 0,45 µm membrane filter using a vacuum or pressure filtration device (4.2.3). Rinsing of the filter with water or another solvent is not allowed after filtration.

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NOTE 1 When volatile components have to be analysed, vacuum filtration should not be used.

When filtration as specified above is not possible in less than one hour with an eluate flow rate of at least 30 ml/cm^2/hour, a liquid-solid separation procedure, specific for the considered case, shall be applied and reported in details in the test report. A recommended procedure is given in annex E. The specific procedure shall not include the use of additives.

NOTE 2 It is always recommended to try first to filtrate and then to centrifugate.

NOTE 3 Such specific liquid-solid separation procedure can include settling, pre-filtration on coarser filter, centrifugation, filtration on large size membrane filter, filtration at high pressure, filtration at increasing high pressure following a first period without pressure, etc.

NOTE 4 Examples of additives (which cannot be used) are calcium chloride, ammonium salt……etc

- Measure the volume of filtered eluate VE.
- Measure immediately conductivity (in mS/m), temperature and pH (and optionally redox potential E_h in mV) of the eluate.

5.3 Further preparation of the eluate for analysis

5.3.1 Divide the eluate into an appropriate number of sub-samples for different chemical analysis and store them according to the requirements in EN ISO 5667-3.

5.3.2 Determine the concentrations of constituents of interest using the methods of eluate analysis (ENV 12506, ENV 13370 and others).

NOTE In cases where high contents of dissolved solids (conductivity > 75 mS/cm) are leached, acidification of the eluates can lead to precipitation of salts. This can be avoided by diluting such samples 5 to 10 times prior to acidification. This additional dilution should be taken into account in the calculations. A quantity of the undiluted sample should be acidified and retained in case the 5 to 10 dilution takes the sample below the detection limit for some determinands. Such procedure should be documented in the test report.

5.4 Blank test for the verification of the leaching procedure

Blank tests shall be carried out. A 0,95 litre volume of leachant is submitted to the whole procedure (except the sieving step, the fragmentation step and the splitting step but including the eluate analysis step).

The eluate of this blank test shall fulfill the following minimum requirements : in the eluate of the blank test, the concentration of each considered element shall be less than 20 % of the concentration determined in the eluate of the tested waste or less than 20 % of the concentration in the eluate of a limit value to which the measurement result is to be compared. The elements to be considered are all the elements which are to be determined in the eluate of the tested waste.

If the above requirements are not fulfilled, it is necessary to reduce the contamination.

The blank test results shall not be substracted from the results of the waste leaching test.

NOTE The above provisions for the blank test do not take into account the sieving step, the fragmentation step and the splitting step. In order to minimise the possible contamination during these three steps, it is recommended to process a portion of the laboratory sample through the sieving device, the fragmentation device and through the splitting device before the preparation of the test sample and to discard such material thereafter. This provision does not cover the situation described in the notes under 4.2.5 and 4.2.6.

6 Calculation

The analysis of the eluate(s) produced by the leaching test provides the concentrations of the constituents in the eluate(s), expressed in mg/l. The final results are expressed as the amount of constituent leached relative to the total mass of the sample, in mg/kg of dry matter.

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Calculate the quantity of a constituent leached from the material, based on the dry mass of the original material, from equation :

$$A = C \times [(L/M_D) + (MC/100)] \qquad (5)$$

Where

A is the release of a constituent at a L/S = 10 (in mg/kg of dry matter) ;

C is the concentration of a particular constituent in the eluate (in mg/l) (5.3.2) ;

L is the volume of leachant used (in l) ;

MC is the moisture content ratio as defined in 3.12, expressed as a percentage of the dry mass (4.3.2) and calculated as in 4.3.3 ;

M_D is the dry mass of the test portion (in kg) (4.3.4).

7 Documentation and test report

In order to comply with this European Standard, the following information shall be documented in such a way that they are immediately available on request. In the following list, at least, the items marked with an asterisk (*) shall be included in the test report. All the deviation from the minimum requirements of this European Standard shall also be documented in the test report

7.1 Sample preparation

- reference to this European Standard* ;
- nature of the waste (e.g. granular waste, sludge)* ;
- reference of the laboratory sample* and of the accompanying documents* stipulated in the sampling standard ;
- date of reception in the laboratory of the laboratory sample* ;
- address of the laboratory and name(s) of the responsible person(s)* ;
- mass of the laboratory sample* ;
- date of the preparation of the test portion for the determination of the dry matter content ratio ;
- date of the preparation of the test portion for the leaching test ;
- storage conditions between the three above dates ;
- description of the preparation of the test portion with references to the paragraphs of this European Standard especially : method of size reduction*, including the eventual drying, when required, fraction above 4 mm*, fraction of non-crushable material*, mode of splitting ;
- storage conditions between preparation of the test portion and the implementation of the leaching test.

7.2 Production of eluate

- raw mass M_W (kg) of the test portion* ;
- moisture content ratio MC (%) of the waste material* ;
- address of the laboratory and name(s) of the responsible person(s)* ;

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- date* of the test producing the eluate ;
- description of the extraction with references to the paragraphs of this European Standard, especially the type, the size and the material of the bottle being used, the type and the setting point of the agitation device ;
- temperature at which the test was conducted ;
- volume of leachant added for the extraction L (l)* ;
- description of the liquid-solid separation procedure with references to the paragraphs of this European Standard, especially nature and characteristics of the filter and in the case of a specific procedure, complete and detailed description of the procedure* ;
- duration between the end of the agitation and the beginning of the separation ;
- duration of the complete separation and duration of the eventual centrifugation step ;
- volume of filtered eluate VE (l) ;
- eluate filtration flow rate (ml/cm^2/hour) ;
- results of immediate measurements (pH* value, conductivity*, temperature* and when required redox……) ;
- conditioning method and storage conditions for the further analysis of the eluate, especially acidification, dilution…etc ;

7.3 Analytical determination

Since the analysis step is not included in the scope of this European Standard, the analytical report specified in the standards dedicated to such analysis shall be incorporated, including the date of analysis, the storage conditions until the analysis is performed, the quantification limit of the analytical method being used, the address of the laboratory and name(s) of operator(s)

7.4 Calculations and blank test information

- results of the calculation of the leached quantities in mg/l and in mg/kg of dry mass with references to the paragraphs of this European Standard* ;
- date of the last blank test performed* ;
- results of the last blank test, including the elements considered for the tested material and the levels above which the results can be considered as valid*.

8 Performance characteristics

8.1 Objective of the validation

In a European wide validation study according to ISO 5725-5, the performance characteristics of the compliance leaching tests EN 12457-2 for inorganic components were established. The uncertainty in the end result of a leaching test is composed of contributions from:

- the origin of the material (variation in production processes);
- the method of sampling in the field (differences in representativeness);
- the sample pre-treatment (reduction of the field sample into laboratory sample(s) and preparation of the test portion from the laboratory sample before the leaching test);
- the leaching test itself and the experimental parameter variations as allowed by the tolerances;

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- the chemical analysis (uncertainty in the determination of concentration in the eluates).

In the interlaboratory exercise to establish the uncertainty of the compliance leaching test, the contributions of the first two items listed above were not included. The results of the validation study are relevant to all activities carried out on the laboratory sample including its preparation from the primary field sample.

8.2 Validation study

The validation of EN 12457-2 was carried out with 12 - 14 European laboratories on seven types of waste materials. One of the wastes was tested according to the four parts of EN 12457. The wastes selected for the validation were chosen to represent a range of wastes as broad as possible, because the standard will find general application across many different types of waste materials. In the validation work of parts 2 and 4, all the wastes were tested. In the validation work of parts 1 and 3, only a selection from these 7 wastes were tested (detailed information can be found in the final report on the Validation study mentioned in the Bibliography).

In the validation study the following starting points were used:

- The laboratory samples were all taken from one large batch of the different wastes according to the normal practice. The normal size reduction and the normal repeated mixing were carried out as needed to obtain representative laboratory samples from the large batch sample. However in the case of metallurgical slag, a separate laboratory sample was provided to assess the difference between size reduction down to 4 mm performed in the laboratory and size reduction down to 4 mm performed in the course of the preparation of the laboratory samples.

- The experimental plan was designed by CEN/TC 292 WG 2 on the basis of each laboratory being given two laboratory samples of each waste to be tested. This is in accordance with ISO 5725-5:1998, clause 5 dedicated to heterogeneous material (e.g. sand or aggregate samples etc.). However, in order to verify the contribution of the analysis of the eluate to the overall variability of the leaching test, the participating laboratories were asked to perform a single complete leaching test on each laboratory sample and to analyse the eluates in duplicate.

- The wastes examined cover all the grain size classes to which the compliance leaching test applies: powdered wastes and sludges (0 µm to about 125 µm), fine-grained materials (0 mm to 4 mm) and coarse-grained materials (0 mm to greater than 4 mm) after the required size reduction.

- Not only was testing carried out on familiar waste/component combinations (where considerable experience with the testing procedure has already been reported in the technical literature) but also on less familiar and potentially difficult waste-component combinations. Wastes were chosen in this latter category where it was expected that one or more of the requirements would not be easily fulfilled (for example heterogeneity in metallurgical slag, biological instability of sewage sludge). These combinations were applied in the validation trial to give insight into the level of uncertainty that might arise for these matrices.

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

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Table 1 — Waste types tested and components analysed in the validation of EN 12457-2.

Grain size class	Waste type tested and European Waste Catalogue (EWC) reference number	Components analysed[a]
Powder/sludge	MSWI Filtercake (FCM) EWC 19 01 05	F, Cl, NO2*, Ba, Cr VI
	Sewage sludge (SEW) EWC 19 08 05	Co, Ni, NH4*, TOC, SO4
	Contaminated soil (COS) EWC 17 05 03	As, Pb, Co, Cd, Ni
	Chemical sludge (CHS) EWC 06 05 02	Cr VI, Cr, F*, Cl, Cd*
Coarse-grained	Sand blasting waste (SBW) EWC 12 01 16	Ba, Cu*, Mo F, Zn
	Incinerator bottom ash (MBA) EWC 19 01 11	Ba, Cu, Mo, Sb, SO4
	Metallurgical slag (MES) EWC 10 04 01	Sb, Ba, B, As* , Pb

[a] The components marked with * were analysed but were not included in the determination of the test performance characteristics for reasons justified in Table 2.

8.3 Validation results

The statistical evaluation was conducted according to ISO 5725-5:1998, clause6 providing "robust methods for data analysis": The average values, the repeatability standard deviation ($s_{r\ test}$) and the reproducibility standard deviation (s_R) were obtained (Table 2). In order to compare and contrast the contribution of the analysis of the eluate to the overall uncertainty in the leaching test, Table 2 lists the repeatability standard deviation for the eluate analysis $s_{r,anal}$ as obtained in the validation study.

The repeatability is determined as an interval around a measurement result (i.e. "repeatability limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by the same laboratory using its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The repeatability limit was calculated using the relationship : $r_{test} = f \cdot \sqrt{2} \cdot s_{r,test}$ with the critical range factor $f = 2$.

For instance, for the first line of Table 2, the repeatability limit around a measurement result of 4,69 mg As/kg is ± 0,49 mg As/kg (i.e. ± 10,4 % of 4,69)

NOTE 1 The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value $f = 2$ used in the factor $f \cdot \sqrt{2}$ corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value $f = 2$ corresponds to the usual value $k = 2$ of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). However it can be necessary to use a larger value for f in situation as described below in 8.3 and 8.4.

The reproducibility, like repeatability is also determined as an interval around a measurement result (i.e. "reproducibility limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another test result obtained by another laboratory, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by two different laboratories using their own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The reproducibility limit was calculated using the relationship : $R = f \cdot \sqrt{2} \cdot s_R$ with the critical range factor $f = 2$.

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For instance the first line of Table 2, the reproducibility limit around a measurement result of 4,69 mg As/kg is ± 3,85 mg As/kg (i.e. ± 82,0 % of 4,69)

NOTE 2 The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value f = 2 used in the factor f · √2 corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value f = 2 corresponds to the usual value k = 2 of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the dispersion limit is equal to $k \cdot s_R$ with the usual value k = 2, resulting in a dispersion limit lower than the reproducibility limit (i.e. a ratio of √2). However it can be necessary to use a larger value f · √2 (or k) in situation as described below in 8.3 and 8.4.

The statistical evaluation of clause 6 of ISO 5725-5:1998 relies, among others, on two basic principles:

- a quasi normal distribution for the differences calculated for each pair of results: this is not generally the case in the validation program;

- an assumption that the extreme results are given by "poor quality" laboratories and, consequently, the robust method calculates the repeatability and the reproducibility on the basis of the "good quality" laboratories without being influenced by the results of the "poor quality" laboratories. In addition it is assumed that the group of such extreme values is not too important (as indicated in 6.1.3 of ISO 5725-5:1998).

However, in the case of heterogeneous materials, the concept of a distinction between “poor” and “good” laboratories includes not only the quality of operation of the laboratory in accordance with the applied standardised method, but also the heterogeneity between the laboratory samples. The consequence is that each and every laboratory has the same chance of receiving a laboratory sample that produces extreme results

Consequently, in case of relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Tables 2 and 4 (this means that the value chosen for the critical range factor f is larger than 2 as well as for the coverage factor k for dispersion). This is because the extreme results may have been obtained in accordance with the present standard and/or be caused by the variability within, or in between, the laboratory samples.

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Table 2 — Results of the validation studies of EN 12457-2

EN 12457-2			Repeatability standard deviation	Reproducibility standard deviation	Repeatability limit (comparing two measurements)	Reproducibility limit (comparing two measurements)	Number of labs	Eluate analysis repeatability standard deviation
Sample	Element	Average						
Code		mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
COS	As	4,69	3,7	29,3	10,4	82,0	11	3,4
COS	Pb	33,19	4,9	7,4	13,7	20,7	11	3,4
COS	Cd	19,71	3,9	16,6	10,9	46,5	11	4,1
COS	Ni	4,70	4,1	14,7	11,5	41,2	11	3,1
COS	Co	4,31	5,0	19,0	14,0	53,2	11	4,1
Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
SEW	TOC	31544	3,4	19,9	9,5	55,7	13	2,4
SEW	SO_4	505	25,7	25,7	72,0	72,0	13	2,8
SEW[d]	NH_4	2882	11,4	51,0	31,9	143	13	2,0
SEW	Ni	2,5	8,7	14,5	24,4	40,6	13	3,0
SEW	Co	0,51	9,7	23,3	27,2	65,2	13	1,6
Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
CHS	Cr^{VI}	5320	3,0	13,7	8,4	38	11	1,8
CHS	F	6,2	4,1	34,7	11,5	97	7	3,1
CHS	Cr	5059	3,4	22,0	9,5	62	11	1,0
CHS	Cl	5390	9,8	25,2	27	71	11	2,1
CHS[a,b]	Cd	0,029	*[-[f]]*	73	*[-[f]]*	204	3	
Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
FCM	Cl	2752	2,37	7,07	6,6	19,8	13	1,8
FCM	F	7,30	7,84	28,28	22	79	12	3,4
FCM[a]	NO_2	2,12	-[f]	124	-[f]	347	3	5,2
FCM	Ba	0,64	4,79	17,3	13,4	49	13	2,2
FCM	Cr^{VI}	11,46	4,20	23,7	11,8	66	13	1,6
Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
MBA	Mo	0,48	17,7	26,7	50	75	12	7,3
MBA	Sb	0,29	19,1	36,0	53	101	12	5,5
MBA	SO_4	1517	15,6	39,6	44	111	14	3,9
MBA	Ba	1,62	11,9	37,0	33	104	13	2,6
MBA	Cu	4,57	18,3	22,8	51	64	14	1,8

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Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
MESr [b,g]	As	0,047	38,4	39,7	108	111	10	10
MESr	Sb	0,76	30,9	34,9	87	98	13	2,2
MESr	Ba	6,20	8,4	26,1	24	73	13	1,9
MESr	B	1,96	15,3	31,0	43	87	12	3,6
MESr [c]	Pb	1,20	31,1	81,2	87	227	13	4,4
Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
MES[c]	As	0,087	51,0	83,5	143	234	11	3,4
MES	Sb	0,67	15,5	38,7	43	108	13	4,6
MES	Ba	6,2	17,9	27,9	50	78	13	1,7
MES	B	2,45	19,1	44,5	53	125	13	24
MES[c]	Pb	1,36	36,7	55,1	103	154	13	3,4
Sample	Element	mg/kg	$s_{r,test}$ %	s_R %	r_{test} %	R %	N	$s_{r,\ anal}$ % [e]
SBW	Ba	7,20	34,82	50,69	97	142	11	3,6
SBW[b]	Cu	0,19	26,70	109,58	75	307	9	9,0
SBW[c]	Mo	1,12	77,28	106,39	216	298	11	3,1
SBW	F	17,50	5,10	45,49	14,3	127	9	3,4
SBW[c]	Zn	5,31	49,76	78,63	139	220	11	3,2

[a] Number of participating labs too low

[b] Analytical data too poor

[c] Obvious heterogeneity (low $s_{r,Anal}$, very high and/or equal $s_{r,Test}$ and s_R)

[d] Affected by biological activity

[e] The repeatability standard deviation of the eluate analysis as obtained in the validation of EN 12457 is consistent with the repeatability standard deviation obtained in the eluate analysis validation study .

[f] Number of pairs of results too low

[g] MESr – sample size reduced centrally by the sample dispatching laboratory as opposed to size reduction by participating laboratories, according to the standard.

NOTE For comparison, the performance of the Standard Eluate analysis is given in Table 3 for components measured with an analytical within laboratory variability of better than 10 %. This standard eluate was prepared especially for the validation of the leaching test.

Table 3 — Typical value and range of standard deviation for the analysis of a standard eluate

Analysis of the standard eluate	median	minimum	maximum
Repeatability standard deviation s_r %	2,9 %	2 %	9 %
Reproducibility standard deviation s_R %	7,7 %	6 %	23 %

8.4 Summary of the performance characteristics evaluation

The validation data have been evaluated. Table 4 gives the resulting typical values for repeatability and reproducibility limits as well as their observed ranges. The typical value is derived from the data in Table 2 by taking the median value and eliminating data as indicated in Table 2 and rounding the numbers.

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Table 4 — Typical values and observed ranges of the repeatability and reproducibility limits

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. Conversely, 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.

For instance if the typical value of 72 % is selected for the reproducibility limit of a test result of 5,4 mg / kg, the result is given as follows with its reproducibility limit at 95% statistical confidence: 5,4 mg / kg ± 3,9 mg / kg (i.e. ± 72 % of 5,4).

Results of the validation of the compliance leaching test EN 12457-2	Typical value	Observed range
Repeatability limit, r	24 %	7 % - 100 %
Reproducibility limit, R	72 %	20 % - 160 %

NOTE 1 The above results refer to the difference that may be found between two test results performed on two laboratory samples obtained under the same conditions. In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the above typical reproducibility values and observed reproducibility ranges should be divided by $\sqrt{2}$ to obtain the corresponding typical dispersion limit and its observed range (cf. the detailed note 2 of 8.3). In the example of As in COS, the result and its dispersion limit is 4,69 mg/kg ± 2,74 mg/kg (i.e. $2 \cdot s_R$ = 58,6% of 4,69). This means that with a 95 % statistical confidence, the values reasonably attributable to the measured parameter are larger than 4,69-2,74 mg/kg and lower than 4,69+2,74 mg/kg.

NOTE 2 The repeatability limit (r) and the reproducibility limit (R) as given in Table 2 and in this table are indicative values of the attainable precision if the compliance leaching test is performed in accordance with this EN 12457-2.

NOTE 3 For wastes with a leaching behaviour strongly influenced by the pH in the pH range that occurs in the compliance leaching test, a greater uncertainty is taken into account, since the material heterogeneity may induce pH differences between laboratory samples as well as between test portions. Also, the repeatability and the reproducibility limits may be larger than the values given in this chapter 8, since differences may occur in the particle size distribution for a given material, depending on the crushing procedure and the waste material being crushed.

NOTE 4 A limited number of wastes and parameters were tested. Consequently, for other wastes and parameters, performance characteristics can fall outside the limits as derived from the validation of the compliance leaching test EN 12457-2. The repeatability and reproducibility limits given for Part 1 cannot be compared with those of other parts due to the fact that dissimilar waste/component combinations were measured.

NOTE 5 In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits can be larger than the values given in this clause 8, since the extreme results were not taken into account in the statistical evaluation of the test data as described in 8.3.

NOTE 6 The above typical values and observed ranges also correspond to the usual critical range factor of 2 (and for dispersion calculation to the usual coverage factor k = 2, recommended in the Guide to the expression of Uncertainty in Measurement). However it can be necessary to use a larger value for f (or for k) in situations as described above in notes 4 and 5 as well as in 8.3. In such case the above typical values and observed ranges should be multiplied by a factor of f / 2 where f is the selected critical range factor for the considered case and 2 is the critical range factor used to determine the above typical values and observed ranges.

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Annex A
(informative)

Information on the influence on the test results of the parameters that affect leaching

In this annex information are provided on the possible sources of variability.

A.1 General aspects

The leaching of contaminants from waste is controlled by several parameters and external factors. These factors include the chemical nature of the waste especially in terms of pH, reducing properties and degradable organic matter content, the nature of the leachant, the contact time of the leachant with the waste and whether contaminant leaching is controlled by solubility or by diffusion. Furthermore, the chemical, physical and geotechnical nature of the environment, to which the waste is exposed, are important. The influence and importance of these factors should be examined in the basic characterisation tests in order that the leaching behaviour of the waste is better understood. In ENV 12920 the steps required to achieve such a determination are specified. This generally requires several tests to be performed, to use or establish a behavioural model and the validation of the model.

It is to be noted that in this compliance test the final conditions of the test are imposed by the waste itself. The key factors in this test are briefly addressed in A.2.

A.2 Factors influencing leaching

A.2.1 Influence of contact time

The compliance test is based on the assumption that equilibrium or semi-equilibrium is reached under the test conditions. The contact time required to reach this state is dependent on the particle size. In 24 h, this condition is considered to be sufficiently approached for many parameters from a variety of wastes.

A.2.2 Influence of the liquid to solid ratio (L/S)

In the four procedures described in these four European Standards, different L/S are specified : (10, 8 and 2) leading generally to different test results. This is caused on the one hand by different quantities of leachant being put into contact with the same quantity of material and on the other hand by different leaching conditions dictated by the waste itself (as a result of the compounds of the material dissolved into the leachate). It is to be noted that there is no relation available that could be applied to the results obtained with a given L/S to determine the results which would have been obtained if the test has been performed at another L/S.

At lower L/S some components are present in the leachate at higher concentration as a result of the lower quantity of the available leachant.

At L/S =2 the test is not applicable to different categories of waste which have a high water content before the test (such as dewatered sludges, filter cakes ….) or after the test (material retaining a too high proportion of the leachant). At L/S = 10 such limitations appear only in a few cases.

A.2.3 Influence of pH

In this compliance test the final conditions of the test are imposed by the waste itself. This is generally the case for pH. The sensitivity of leaching to relatively small changes in pH can be significant. Such sensitivity can induce varying results. Also exposure to atmospheric CO_2 or increased CO_2 levels in the laboratory during sample storage, handling, performance of the leaching test and analysis can affect the test results, as they can lead to pH changes in the leachate.

A.2.4 Influence of reducing properties

Materials to be tested can exhibit reducing properties, which is evident from a low redox potential in the leachate. For a proper evaluation of a material it is important to be aware of this aspect, as different degrees of oxidation in sample handling and storage may induce varying results.

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A.2.5 Influence of size reduction

Laboratory preparation, such as size reduction, can modify the properties of the waste and therefore its leaching behaviour. Size reduction can lead to a variation of the pH of the eluate and consequently to a different leachability of pH sensitive constituents. For this reason, this European Standard specifies (4.3.2) that the material shall not be finely ground and plans to limit the particle size reduction in order to maintain the physical state of the waste.

A.2.6 Influence of the leaching of organic contaminants

The leaching of organic contaminants from waste is an area that is still not well addressed. Traditionally leaching tests have been focused primarily on inorganic constituents and have been applied to leaching of organic contaminants without further evaluation of the suitability of the methods. The leachability of organic contaminants is governed by processes that differ considerably from that of inorganic contaminants. In addition, the properties of organic contaminants in relation to sorption on different materials with which they come in contact (e.g. bottles, filters) are different for organic contaminants than for inorganic contaminants.

Within the category of organic contaminants a significant difference in behaviour exists between the more polar, relatively water soluble compounds and apolar, hydrophobic organic contaminants. In the latter case mechanisms of release (e.g. particle-bound or dissolved organic carbon-bound) can be more crucial. For these reasons, this European Standard specifies a scope which excludes organic contaminants.

A.2.7 Influence of the mixing of the waste and the leachant

In experimental studies, various agitation devices have been compared showing significant differences in results. Such differences are induced by the different characteristics of the agitation devices to approach near equilibrium conditions during the leaching test. Insufficient mixing can lead to underestimated results. Agitation devices providing adequate mixing are an end-over-end tumbler and a roller table (see 4.2.2). An horizontal shaker table is an example of an unsuitable device.

A.2.8 Influence of temperature

The solubility of constituents can be affected by temperature. Wastes containing organic degradable matter can be affected by biological processes, which are also temperature dependent. The validation tests have shown that a temperature range of 20 °C ± 5 °C provides sufficient control of such effects.

A.3 Analytical versus leaching test uncertainties

From the validation tests that were carried out prior to the adoption of this European Standard, it can be concluded that the contribution of the analytical variability in analysis of eluates in the overall variability of the leaching test specified in this European Standard is generally small, provided that the constituents of interest using the selected analytical methods can be measured with sufficient precision, i.e. well above the detection limit. It is to be noted that this analytical contribution may be less than 10 % of the overall variability for heterogeneous materials and may reach 50 % for relatively homogeneous materials

A.4 Evaluation of test results

The test results obtained with the compliance tests specified in this European Standard only allow a direct comparison with regulatory limits on a pass/fail basis. A comprehensive evaluation of the leaching behaviour requires a basis or framework of reference such as that provided by ENV 12920.

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Annex B
(informative)

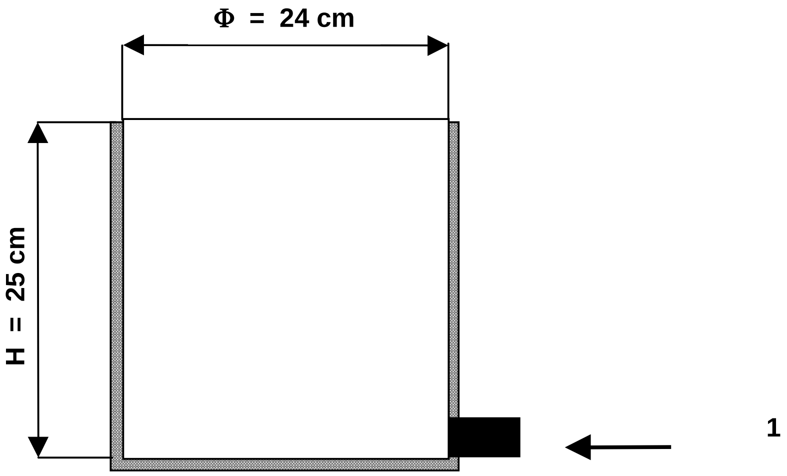
Test for determining whether waste is in the liquid state

B.1 Principle

This test consists in determining the possibility for the waste to flow through a calibrated orifice upstream of which the material is under a determined head corresponding to the weight of the column which it forms. By convention, the waste is said to be liquid if it completely flows up to the level of the upper part of the orifice within a period of a limited duration. It is to be noted that CEN / TC 308 is preparing a test for sludges.

B.2 Procedure

- The test is to be performed at room temperature (20 °C ± 5 °C)
- Seal off the cylinder orifice with a rubber stopper.
- Fill up the cylinder completely with the waste.
- Remove the stopper.
- The waste will be said to be liquid if it flows completely up to the level of the upper part of the orifice within a period not exceeding 8 h, without vibration or shaking.



Key

1 Rubber stopper Φ = 2,5 cm

Figure B.1

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Annex C
(informative)

Example of datasheet for EN 12457-2

Sample code :
Nature of the waste :
Sample description :

Sample weight (kg) :
Date of reception :

Method of size reduction :
Fraction of non-crushable (% *w*/*w*) :
Fraction of oversize (% *w*/*w* > 4 mm) :
Mass of test portion M (in kg) :
Method of liquid-solid separation :

Moisture content (in % *w*/*w*) :
Volume of leachant L (in l) :

Date of blank determination :

Date of the leaching test :

Parameters	
PH Temperature (°C) Conductivity (mS/m) Redox potential (mV)	

Constituents	Limit of determination (mg/l)	Blank (mg/l)	Concentration in the eluate (mg/l)	Amount leached (mg/kg)

Deviation from EN 12457-2 and justification :

Laboratory :
Address :
City :

Responsible official :
Signature :
Tel. : Fax :

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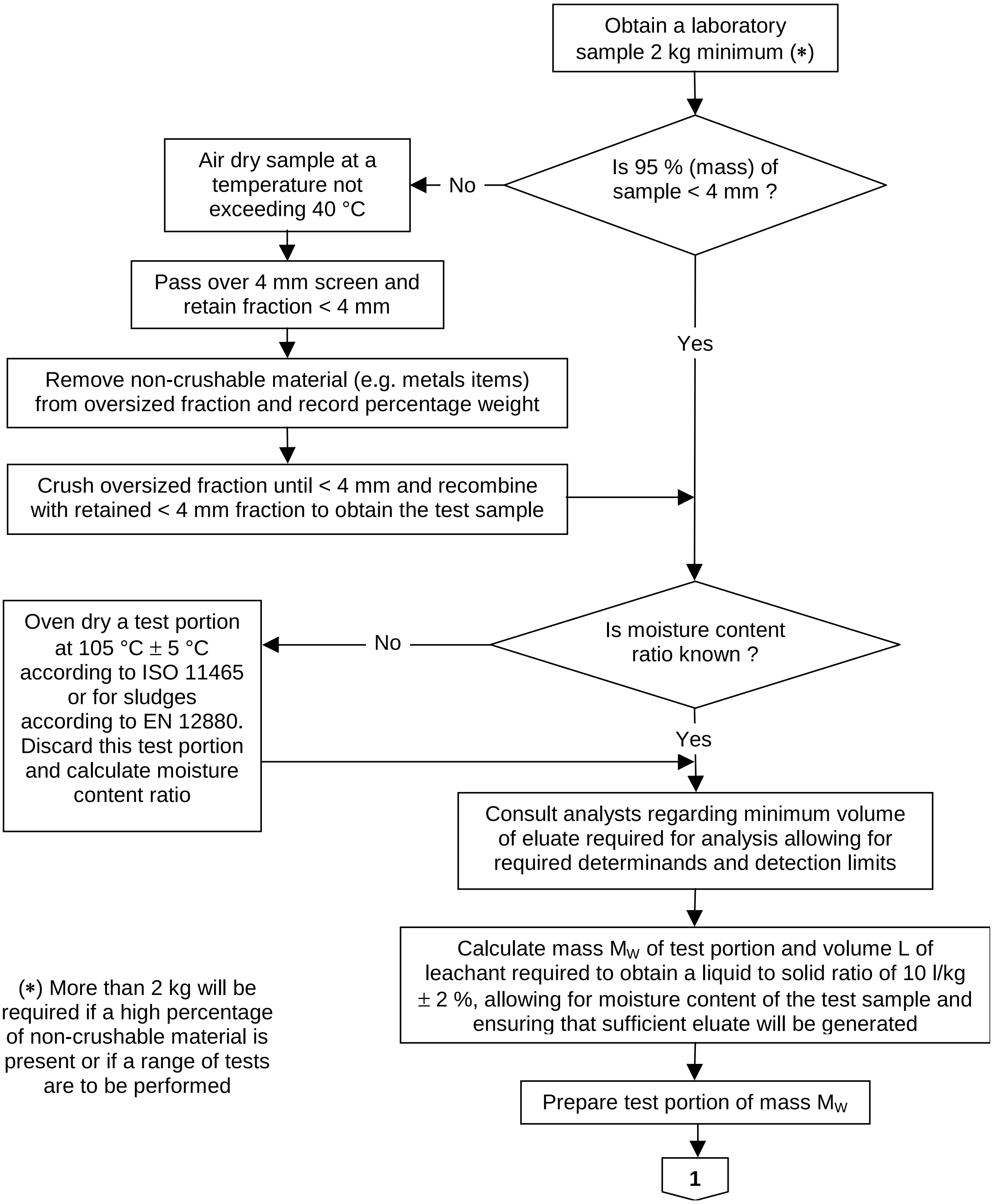
Annex D
(informative)

Process map for EN 12457-2



(∗) More than 2 kg will be required if a high percentage of non-crushable material is present or if a range of tests are to be performed

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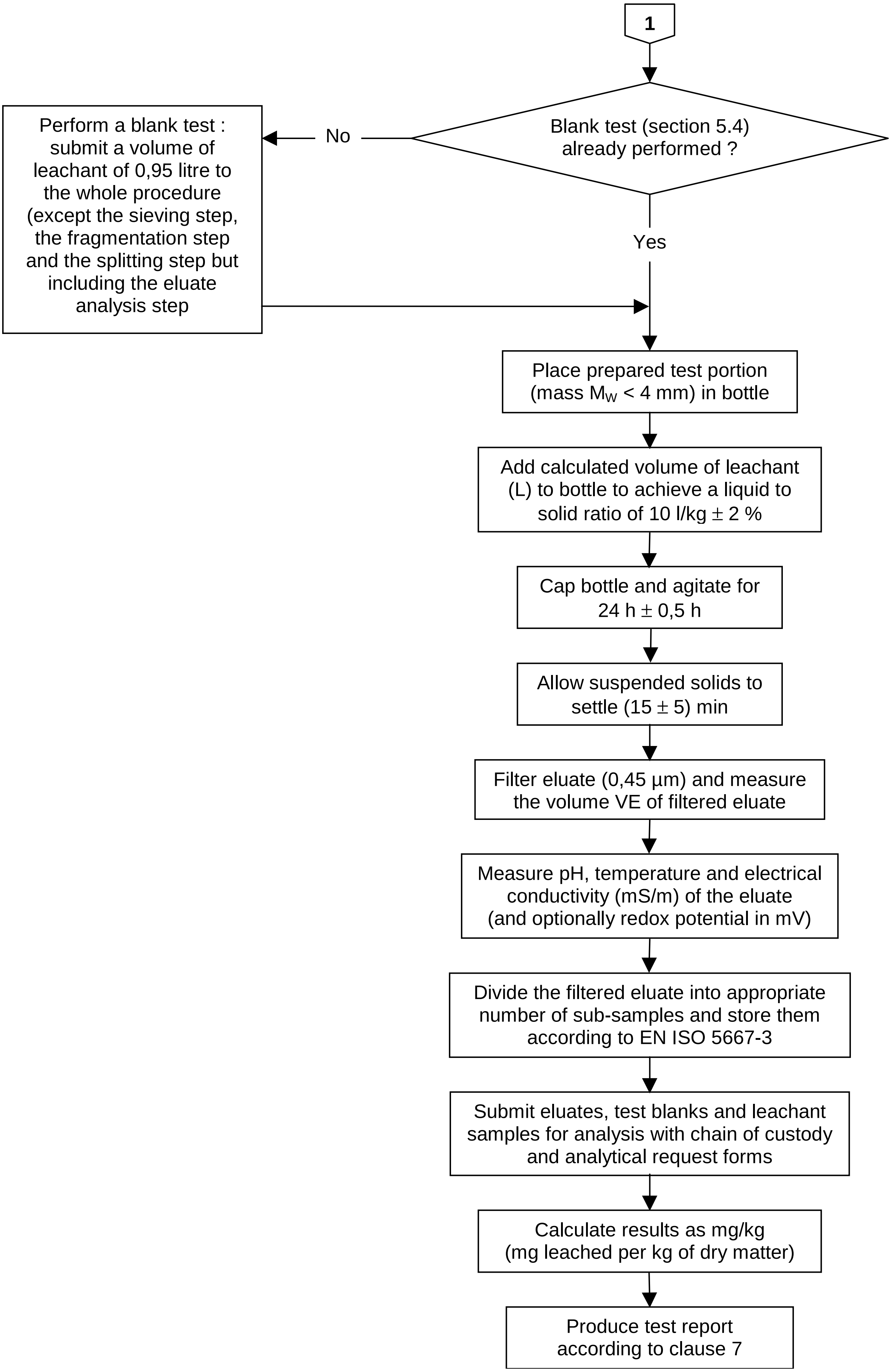

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Annex E
(informative)

Example of a specific liquid-solid separation procedure for soil sample

E.1 Introduction

The original scope of leaching tests covered in particular solid substances containing large amounts of dissolved salts. The general feasability of these methods has limitations, when the solubility of substances has to be determined in soil sample in particular when, e.g. oxidised, adsorbed or organically bound heavy metals have to be rather insoluble in those materials. Despite their low solubility, heavy metals are important from an environmental point of view. The lower “pure” solubility of heavy metals in a contaminated soil sample, the bigger is the relative influence of colloidal particle portions in eluate on the end result.

Especially in case of fine-textured soil samples being rich in humus but poor in electrolytes, the filter cake produced during filtration exhibits very fine pores and less colloids pass through the membrane filter. Thus the production of filter cake largely affects the “solubility” of heavy metals, which is identified by this method. To obtain comparable results it is necessary to stipulate the factors determining the height of the filter cake. In addition to sample-specific properties, the thickness of the filter cake is determined predominantly by the filter diameter and the volume of the eluate to be filtered.

E.2 Procedure

E.2.1 Equipment

- Pressure filtration unit for membrane filter (142 mm diameter) ;
- Membrane filter of pore size 0,45 µm.

When using another filter size the volume to be filtered shall be modified according to the filter surface. The relationship between the volume to be filtered and the filter surface shall be maintained (relationship : about 1 l volume to 158 cm^2 filter surface (diameter 142 mm)).

E.2.2 Material in contact with extracts

- Media-guiding apparatus in PTFE.

E.2.3 Procedural steps

- For sedimentation of the larger particles, the suspension shall stand for 15 minutes after shaking ;
- Transfer almost completely the supernatant liquid into a centrifuge tube or bottle device ;
- Apply centrifugation (30 min at 2 000g) ;
- After this, transfer almost completely the supernatant liquid into the membrane pressure filter apparatus ;
- After 5 min of such filtration without pressure, apply a pressure of 1 bar to accelerate filtration. If after 15 min less than two thirds of the eluate have passed through the filter, the pressure is increased to 2 bar. If necessary, the pressure is increased to a maximum of 3,5 bar after 30 min. Filtration proceeds until all the supernatant of centrifugation has passed through the filter. If the filtration is still incomplete after 120 min, it is stopped and worked on with incomplete filtrate.

NOTE By using this procedure, a significant reduction in the possible errors resulting from the proportions of dissolved or under colloidal form heavy metals in the filtrate can be obtained. Subsequently it is not allowed to decant the first part of the filtrate and to put it again on the filter – a method that is quite common in several laboratories.

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