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BSI Standards Publication

Characterization of waste — Compliance Leaching Test — One stage batch leaching test for monoliths at a fixed liquid to surface area ratio (L/A) for test portions with fixed minimum dimensions

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National foreword

This Published Document is the UK implementation of CEN/TS 15862:2012.

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

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Characterisation of waste - Compliance leaching test - One stage batch leaching test for monoliths at fixed liquid to surface area ratio (L/A) for test portions with fixed minimum dimensions

Caractérisation des déchets - Essai de lixiviation de conformité - Essai de lixiviation en bâchée unique pour des monolithes avec un rapport liquide/surface (L/A) fixe, pour des prises d'essai de dimensions minimales fixes

Charakterisierung von Abfällen - Auslaugung zur Übereinstimmungsuntersuchung - Einstufiges Auslaugungsverfahren für monolithische Abfälle bei festgelegtem Flüssigkeit/Oberfläche-Verhältnis (L/A) für Prüfmengen mit festgelegten Mindestabmessungen

This Technical Specification (CEN/TS) was approved by CEN on 6 December 2010 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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Foreword

This document (CEN/TS 15862:2012) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

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

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:

— NF X 31-211:2000;

— NEN 7345:1995;

— ÖNORM S 2116-4.

This document specifies compliance tests. For basic characterization a methodology for the determination of the leaching behaviour of waste has been developed and formulated in EN 12920.

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

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

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

Introduction

In the different European countries, tests have been developed to characterize 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 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.

Procedure to characterize the behaviour of waste materials can generally be divided into three steps, using different tests in relation to the objective. The following test hierarchy is taken from the Landfill Directive[1] and the Decision on Annex II of this Directive[2] for disposal of waste.

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

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

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

The procedure described in this document is a compliance leaching test and falls in category b).

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

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

1 Scope

This Technical Specification specifies a compliance test for routine testing providing information on the leaching of monolithic waste which can be obtained under the experimental conditions specified hereafter with a single batch leaching test at a specified liquid to surface area ratio (L/A) of 12 ($cm^3 \cdot cm^{-2}$). It applies to test portion of monolithic waste of regular shape, with a minimum dimension of 40 mm in all directions, obtained e.g. by cutting, coring or moulding.

This document is not applicable if the surface area of the test portion cannot be determined by simple geometrical means.

This document has been developed to determine the release of mainly inorganic constituents from wastes. It does not take into account the particular characteristics of organic constituents nor the consequences of microbiological processes in organic degradable wastes.

The test procedure specified in this document produces an eluate which subsequently need to be characterized physically and chemically, according to appropriate standard methods.

NOTE 1 If, in order to comply with the requirement of regular shape, the test portion is prepared by cutting or coring, then new surfaces are exposed which can lead to change(s) in leaching properties.

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

This leaching test does not provide information by itself on dynamic leaching behaviour, as specified in EN 12920. It does not give information on equilibrium conditions. For specific situations or basic characterization, other tests are available in the toolbox of CEN/TC 292 *"Characterization of waste"*.

This document does not address issues related to health and safety.

2 Normative references

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

EN 14346, *Characterisation of waste — Calculation of dry matter by determination of dry residue or water content*

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

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

EN ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples (ISO 5667-3)*

3 Terms and definitions

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

3.1
eluate
solution obtained from a leaching test

[SOURCE: EN 12457-1:2002, 3.1]

3.2
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

[SOURCE: IUPAC:1997, 3.2]

Note 1 to entry: 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 to entry: Several *laboratory samples* may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

3.3
leachant
liquid that is brought into contact with the test portion in the leaching procedure

Note 1 to entry: For the purpose of this document the leachant is water as specified in 5.1.

3.4
leaching test
test during which a material is put into contact with a leachant and some constituents of the material are extracted

[SOURCE: EN 12457-1:2002, 3.4]

3.5
liquid volume to surface area ratio
L/A
ratio between the amount of liquid (*L*), which, at any time of the test, is in contact with the waste, and the surface area of the *test portion* (*A*)

Note 1 to entry: *L*/*A* is expressed in $ml \cdot cm^{-2}$.

3.6
monolithic waste
waste which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered scenario (see EN 12457-1:2002)

3.7
release
emission of constituents from waste which pass through the external surface of a waste mass as specified in the considered scenario

[SOURCE: EN 12920:2006, 3.7]

3.8
single batch leaching test
leaching test in which a fixed amount of material is leached in one step with a fixed amount of *leachant*

[SOURCE: EN 12457-1:2002, 3.8]

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

[SOURCE: IUPAC, 1997, 3.9]

3.10
test portion of monolithic waste of regular shape
test portion of monolithic waste for which the surface area of the test portion can be calculated on the basis of simple geometric equations

3.11
test sample
sample, prepared from the *laboratory sample,* from which *test portions* are removed for testing or for analysis

[SOURCE: IUPAC:1997, 3.11]

4 Principle

This compliance leaching test for monolithic waste consists in a single batch leaching test with a L/A ratio of 12 $cm^3 \cdot cm^{-2}$. The test portion, obtained originally or after pre-treatment, has a minimum dimension of 40 mm in all directions. The test portion is brought into contact with water as specified in 5.1 under defined conditions for a duration of 24 h. After filtration, the properties of the eluate are measured using methods developed for water analysis adapted to meet criteria for analysis of eluate.

NOTE Different factors (see Annex C) can influence the results linked to the dimension-shape of the test portion (e.g. sharp edges, dimensions tolerance acceptance) or linked to the quality of the material (e.g. quality of the surface, density).

5 Reagents

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

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

5.2 Nitric acid, $c(HNO_3)$ = 0,1 $mol.l^{-1}$.

6 Equipment

6.1 General

Since low concentrations of leached components in the eluate can occur, the question of contamination of and adsorption on the equipment has to be addressed. Materials in contact with the eluate in 6.2.1, 6.2.6, 6.2.10, 6.2.11, shall be of such quality that contamination and adsorption are minimized. The blank test addresses the issue of contamination. The following materials are generally considered as suitable: glass, plastics (polyethylene (PE), polytetrafluoroethylene (PTFE), polyvinylchloride (PVC), polyethyleneterephtalate (PET)) according to EN ISO 5667-3.

6.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

6.2.1 Leaching vessel or tank, glass or plastic (e.g. polymethyl methacrylate (PMMA), polytetrafluoroethylene (PTFE), polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC), which can be closed and/or sealed, to avoid prolonged contact with the air and, if necessary, to apply vacuum.

Clean the vessel or tank before use by filling it with nitric acid (5.2), leaving it for at least 24 h and then flushing it out with water (5.1).

NOTE 1 The vessel or tank may have connections to allow to apply vacuum at the beginning of the leaching procedure.

NOTE 2 The contact with the air is kept limited to avoid uptake of CO_2 from the air in case leaching from alkaline materials.

The minimum distance between the test portion and the walls of the vessel or tank shall be 2 cm, all around the test portion. Examples are given in Annex B.

Supports made of inert material shall be used to allow direct contact with water on all sides of the specimen. Supports shall not affect significantly the surface area of the sample exposed to the leachant.

6.2.2 Diamond blade cutting device and/or core drilling device (dry process).

6.2.3 Analytical balance, with accuracy of at least of at least 1 g.

6.2.4 Device for measuring sample dimensions, with an accuracy of at least 1 mm.

6.2.5 Filtering device, 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 bars). Rinsing is compulsory.

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

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

6.2.6 Bottles made in material, that will not affect the analytical results or the assessment of the results in accordance with EN ISO 5667-3, with an appropriate volume (500 ml to 2 000 ml) to minimise headspace; glass bottles having caps of inert materials, for example PTFE.

NOTE For inorganic constituents high density polyethylene/polypropylene (HDPE/PP) bottles are preferred, except for samples analysed for the mercury.

6.2.7 Conductivity meter, with an accuracy of at least 0,1 $mS.m^{-1}$.

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

6.2.9 Thermometer.

6.2.10 Stirring device, magnetic bar, propeller or other (made of or coated by inert material).

6.2.11 Pre-rinsed or similarly membrane filters for filtration of the eluates, with a pore size of 0,45 µm. (e.g. rinsed with 0,1 $mol.l^{-1}$ HNO_3 (5.2) and water (5.1)).

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.

6.2.12 Vacuum pump or a water suction device, reaching a residual pressure of 50 mm Hg (6 700 Pa) or less.

6.2.13 Redox potential meter (optional).

7 Sample preparation

7.1 Laboratory sample

For the performance of this procedure a test portion is required, consisting of at least one monolithic specimen or test piece, the structure, homogeneity and composition of which shall be representative for the waste material that is to be investigated. The specimen(s) or test piece(s) shall have a minimum dimension in all directions of 40 mm.

NOTE In order to increase the representativeness of the test portion, it can be useful to test more than one specimen or test piece, together. The surface area of the test portion is, in that case, the total surface area of the individual specimens or test pieces.

Minimum dimensions are relevant, in order to ensure that, even for easily leachable constituents, no depletion occurs during the test. Minimum dimensions of 40 mm in nearly all cases suffice. However, in case of high release rates, it is recommended to justify afterwards that no depletion has taken place (see Annex C).

The laboratory sample consists of one or more specimens or test pieces.

The age of waste materials/specimens is an important factor, which can influence on leaching properties. In case the waste material results from a stabilization process (performed in the laboratory or in the practical situation) the waste material should be cured sufficiently long to avoid major variations in leaching due to ongoing changes in pore structure and in formation of release controlling mineral phases. The production date and/or curing time of the laboratory sample (at least if prepared in the laboratory) should be reported in the test report.

7.2 Test portion

7.2.1 Preparation of the test portion:

A test portion is obtained from the laboratory sample, by applying EN 15002.

NOTE 1 If, in order to comply with the requirement of regular shape, the test portion is prepared by cutting or coring, new surfaces are exposed, which can lead to change(s) in leaching properties, compared with aged surfaces.

Store the test portion in accordance with EN 15002 to minimize changes due to the exposure to atmosphere (drying, carbonation, etc.).

The choice of the procedure used shall be justified and described in the test report.

The preparation of the test portion shall prevent leaching (e.g. use of cooling fluid shall be excluded).

When the waste material results from a stabilisation process, the waste material should be cured sufficiently long to avoid major variations in leaching due to ongoing changes in pore structure and in formation of release controlling mineral phases.

NOTE 2 The test portion could be a laboratory prepared sample, which is prepared under conditions similar to the field situation. It could also be a part of a waste material, unless cutting results in significant differences in surface properties influencing the leaching behaviour.

7.2.2 Influence of curing time and ageing of the test portion

The date of preparation of the test portion (if prepared in the laboratory) and the date of the performing of the test shall be reported in the test report.

NOTE The age of product is an important factor that can influence the leaching properties.

7.2.3 Storage conditions of the test portion

For thermally prepared monolithic waste, monolithic materials with organic binders, cored and cut test portions are ready for testing immediately after their preparation, unless otherwise specified. For any hydraulic binder based stabilized waste materials the curing conditions shall be reported.

NOTE The minimum curing time depends on the particular stabilization process under consideration.

The test portion shall be stored at room temperature (20 ± 5) °C in closed flexible vessels as to minimize changes due to the exposure to atmosphere (drying, carbonation, etc.).

8 Procedure

8.1 Testing conditions

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

8.2 Description of the single batch leaching test

8.2.1 Preparation

Remove dust and loose particles of the test portion by blowing gently using compressed air.

Determine the surface area A (cm^2) of the test portion by simple geometrical means.

Determine the mass M_0 (g) of the test portion.

Determine the volume of leachant L (cm^3) to add for the leaching procedure:

$$L / A = 12 \text{ cm}^3 \cdot \text{cm}^{-2}$$

NOTE If required, for example to express the results in a different way (e.g. mg/kg), the dry is determined matter according to EN 14346 on a separate test portion.

8.2.2 Leaching procedure

Place the support of the test portion in the bottle.

Place the test portion of surface area A on the support, in the leaching vessel or tank (6.2.1).

Add a volume of leachant (L), as specified in 5.1, establishing a liquid to surface area ratio (L/A) = 12 $cm^3 \cdot cm^{-2}$ ± 2 % during the extraction such that the top of the test portion is at least 2 cm submerged.

Agitate for (24 ± 0,5) h with the stirring device (6.2.10). The stirring action shall be efficient to expose the entire surface area of the test portion to the same conditions, also taking into account the elements previously transferred in the solution and therefore the concentration obtained in the solution (i.e. homogeneous concentrations in the tank). The criterion should be set, considering that the increase of the stirring intensity does not increase anymore the selected parameter (pH or constituents' concentration).

NOTE For a cylindrical test portion of 8 cm high and 4 cm in diameter, a magnetic stirrer with a 120 rpm speed is enough.

Filter the eluate, over a 0,45 µm membrane filter using a vacuum or pressure device (6.2.5, 6.2.11, 6.2.12). Rinsing of the filter with water or another solvent is not allowed after filtration.

When filtration as specified above is not possible, it shall be reported in detail in the test report.

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

It is recommended to try first to filtrate and then to centrifugate.

Measure the volume of filtered eluate VE (in ml).

Measure immediately conductivity (in mS.m-1), temperature and pH (and optionally redox potential Eh in mV) of the eluate.

8.3 Further preparation of the eluate for analysis

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.

Determine the concentrations of constituents of interest using the methods of eluate analysis (EN 16192 and others).

In cases where high contents of dissolved solids (conductivity $> 7{,}5$ S.m^{-1}) are leached, acidification of the eluates can lead to precipitation of salts. This can be avoided by diluting such samples five to ten 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 five to ten dilution takes the sample below the detection limit for some determinands. Such procedure should be documented in the test report.

8.4 Blank test for the verification of the leaching procedure

Blank tests shall be carried out. A volume of leachant equivalent to *L* (in liters) is submitted to the whole procedure (except steps concerning the test portion preparation, but including the eluate analysis step).

The eluate of this blank test shall fulfil 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.

9 Calculations and expression of results

The concentrations of the constituents in the eluates are expressed in mg·l^{-1}. The final results are expressed as the amount of constituent leached relative to the surface area of the sample, in mg·m^{-2}.

Calculate the quantity of a constituent leached from the material, from formula:

$$R = \frac{10 \times C \times L}{A} \qquad (2)$$

where

L is the volume of leachant in ml according to the L/A ratio (3.5);

A is the surface area in cm^2 of the test portion;

R is the release of the constituent in mg·m^{-2};

C is the concentration of a particular constituent in the eluate in mg.l^{-1}.

In view of the surface area related nature of the test, the results are expressed in mg.m^{-2}.

If required, for example for the purpose of comparison with historic data sets, information necessary for the user to also express test results in different units (e.g. mg.kg^{-1} of dry mass) shall be provided in the test report.

10 Documentation and test report

10.1 General

In order to comply with the present document, the following information shall be documented in such a way that they are immediately available on request. 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 document shall also be documented in the test report.

10.2 General data

— reference to this document*;

— nature of the waste (solidified waste, monolithic material)*;

— identification of the laboratory sample*;

— date of receipt of the laboratory sample*;

— address of the laboratory* and name (s) of the responsible person (s) in charge of the performance of the leaching test.

10.3 Sample preparation

— mass of laboratory sample in kg (refer to sampling report)*;

— conditions and date of preparation of the test portion(s) (e.g. method of cutting, coring)* if carried out by the laboratory;

— storage conditions between reception of laboratory sample and preparation of the test portion(s).

10.4 Production of the eluate

— dimensions in cm, geometrical shape, and surface area in cm^2 of the test portion*;

— mass M_0 of the test portion, in g*;

— dry matter w_{dm} (%) according to EN 14346;

— date of performing the leaching test*;

— description of the leaching test, especially the type, size and material of the leaching vessel, stirring device applied, and all relevant information on equipment and appliances used*;

— temperature at which the test was performed;

— volume L of leachant (ml) added*;

— duration of leaching procedure expressed in h;

— volume of eluate V_E (ml) after the leaching step;

— results of immediate measurements (temperature*, pH value*, electrical conductivity* and, optionally, redox potential) on the eluate;

— conditioning and storage conditions for the different subsamples of the eluate;

— observations in carrying out the test (e.g. gas evolution, precipitation, degradation aspects such as cracking)*;

— all deviations from this document*.

10.5 Analytical procedure

Since the analysis step is not included in the scope of this document, the analytical reports specified in the document 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 methods being used, the address of the laboratory.

10.6 Blank test information

— date of the last blank test performed;

— results of the last blank test, including the elements considered for the test material and the levels above which the results can be considered as valid.

10.7 Results of the leaching test

— the measured concentrations C in $mg \cdot l^{-1}$*;

— the calculated release of constituents R in $mg \cdot m^{-2}$ of geometric surface area*;

— the calculated release in other unit if required.

11 Test performance

The test specified in this Technical Specification is not validated and no data are available on robustness, repeatability and reproducibility.

Annex A
(informative)

Example of data sheet showing the reporting of key elements mentioned in CEN/TS 15862

A.1 Laboratory sample

Date of reception: Sample reference:

Nature of sample (solidified, monolithic, etc.):

Sample mass (g): Sample size (cm):

Assessment of monolithic character:

A.2 Test portion

Date of preparation:

Method of preparation:

Surface area (cm^2): Geometrical shape:

Mass of test portion (g): Dry matter (%) w_{dm} :

A.3 Leaching test

Date of performance of leaching test: Temperature during leaching test (°C):

Volume of leachant L ml	**Duration of leaching step** h

Observation:

A.4 Blank test

Date of last blank test:

pH	
Temperature (°C)	
Conductivity ($mS.m^{-1}$)	
Redox – potential (mV)	

Constituent	Limit of quantification $mg.l^{-1}$	Concentration $mg.l^{-1}$

A.5 Results of the leaching test

pH	Conductivity $mS.m^{-1}$	Redox potential mV

Constituent	Limit of quantification $mg.l^{-1}$	Concentration in eluate $mg.l^{-1}$	Release [a] of the constituent $mg.m^{-2}$
X_1			
X_2			
X_3			
$X_{...}$			
[a] Calculation in $mg \cdot m^{-2}$ and possibly conversion to $mg.kg^{-1}$.			

Annex B
(informative)

Examples of data about the L/A ratio

In the table below for some specimen types (cube, rectangular and cylinder) and the most common dimensions minimum and maximum vessel diameters are calculated, in the case cylindrical vessels are used. The minimum diameter is calculated in order to fulfil the criterion, mentioned in 6.2.1, stating that the minimum distance between the test portion and the walls shall be 2 cm, all around the test portion. The maximum diameter is calculated in order to fulfil the criterion, mentioned in 8.2.2, stating that the top of the test portion shall be at least 2 cm submerged.

Table B.1 — Vessel dimensions (in cm), in relation with specimens of different sizes and / or dimensions (with L/A = 12 $cm^3 \cdot cm^{-2}$)

Specimen										
Type	**Height**	**Diameter**	**Length**	**Width**	**Volume**	**Surface**	**V_{total} [a]**	**Minimum distance between vessel and specimen [b]**	**Distance between specimen and water level [b]**	**Vessel diameter range [c]**
	cm	cm	cm	cm	*V* cm^3	*A* cm^2	cm^3	cm	cm	min. to max. cm
cube	10	-	10	10	1 000	600	8 200	2,0	19,7	18,1 to 27,3
								6,6	2,0	
cube	5	-	5	5	125	150	1 925	2,0	13,0	11,1 to 16,5
								4,7	2,0	
cube	4	-	4	4	64	96	1 216	2,0	10,6	9,7 to 13,9
								4,1	2,0	
cylinder	10	10	-	-	785	471	6 437	2,0	29,8	14,0 to 24,2
								7,1	2,0	
cylinder	8	4	-	-	100	126	1 608	2,0	22,0	8,0 to 13,1
								4,5	2,0	
rectangular	5	-	20	10	1 000	700	9 400	2,0	10,2	26,4 to 36,5
								7,1	2,0	
rectangular	16	-	4	4	256	288	3 712	2,0	32,7	9,7 to 15,4
								4,9	2,0	

[a] V_{total} is the sum of the volume of the specimen (V) and volume of leachant (L) as calculated according to L = 12 x A.

[b] The distance of 2,0 cm is fixed, the non-fixed distance is calculated based on size and/or shape of the specimen type.

[c] The minimum diameter is calculated in order to fulfil the criterion, mentioned in 6.2.1, stating that the minimum distance between the test portion and the walls shall be 2 cm. The maximum diameter is calculated in order to fulfil the criterion, mentioned in 8.2.2, stating that the top of the test portion shall be at least 2 cm submerged.

Annex C
(informative)

Examples of factors influencing the leaching release of monolithic waste

There are several factors influencing release from monolithic specimen, which obviously are not equally relevant for any one given specimen. Below the different factors are briefly presented.

Two types of factors can be distinguished. On the one hand, those which cannot be influenced by laboratory people performing this document. Those factors are linked to the material itself (e.g. intrinsic characteristics), including the possible curing stage:

Nature of the material – the nature of the material is already in part addressed in view of tortuosity, porosity, dimensions, pH, etc. The nature of a product will be of relevance for the substances that are likely to be of relevance. Cement-based materials, sintered bricks, light weight concrete, bituminous materials each have their specific characteristics and most relevant substances.

Porosity – the porosity of a specimen is of relevance as together with tortuosity, it controls the rate of transport by diffusion within the matrix. This relates to connected porosity, as closed porosity (as in light weight concrete) does not contribute to release from the specimen.

Tortuosity – the tortuosity is the extended path length for diffusion due to the non-linear nature of pores. This parameter is a property, that is unique for any one specimen as it depends on curing time, water to cement ratio (relevant for products based on hydraulic or pozzolanic properties), production conditions, degree of compaction, constituents, sintering temperature, etc.

pH – the pH of a monolithic specimen is important as solubility is strongly controlled by pH. In particular, cement based materials undergo carbonation with time depending on the scenario of use. In general, a pH front will develop, where in the modified zone leaching will be different from the leaching of the bulk matrix. In case of materials with a neutral pH (e.g. bitumen, sintered products) the release in a neutral pH environment is not as sensitive to pH. On the other hand materials with a very low ANC are sensitive to field imposed pH conditions (e.g. from acid rain or acidic groundwater).

Redox – in case of stabilised waste with reducing properties (e.g. sulphide addition), a redox front can develop, when the product is exposed to the atmosphere. This strongly affects the release behaviour of specific constituents (R.E.C.O.R.D. study on reduced metallurgical slag)[1].

Availability – the availability of constituents is a fraction of the total content that is relevant for modelling release and can be seen as relevant to assess the maximum released amount that might occur under the most extreme field exposed conditions. This quantity can be derived from the pH dependence leaching test (CEN/TS 14429 or CEN/TS 14997).

On the other hand, factors which can be changed and/or need to be controlled by laboratory people performing this document. Those factors are linked to the test itself (excluding the possible curing stage).

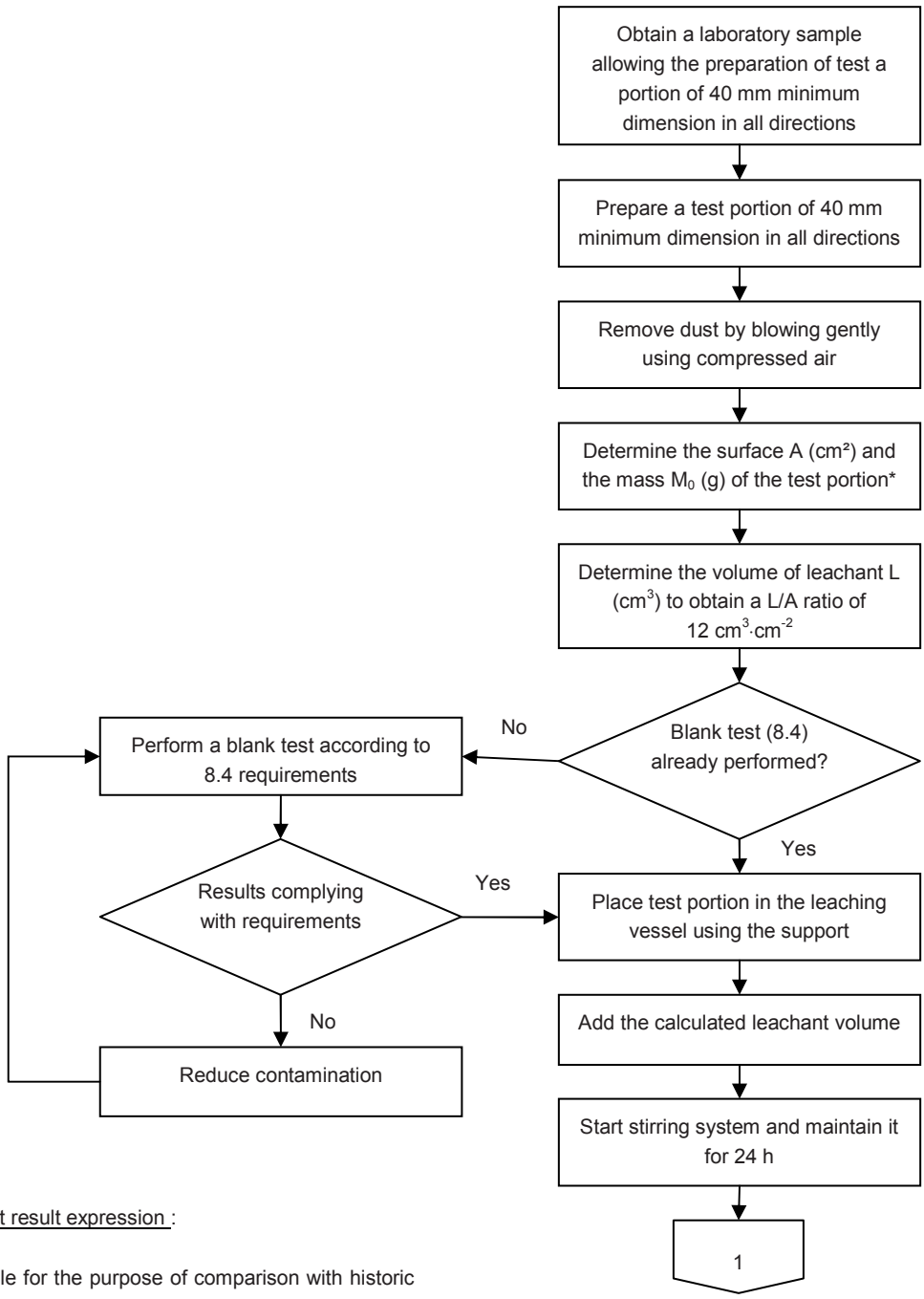
Shape – specimen can be tested in different shapes, e.g. block, cube, cylinder, block with open spaces. Important for expressing the results and interpreting release is the need to be able to quantify the surface area as results are expressed per unit surface area. Monolithic materials with irregular surfaces are therefore more difficult to handle. In that case the geometric surface area can be estimated, or a specimen sampled from an irregular form by cutting or coring can be used as test portion.

Dimensions – the dimension of specimen to be tested is important from the viewpoint of depletion. I.e. in case of small dimensions mobile species can become depleted. This depletion is a function of the porosity, tortuosity and the interaction of the constituent of interest with the solid matrix in a given period of time. For concrete in general 4 cm cubes will be adequate, for stabilised waste specimen larger specimen (10 cm diameter) are generally provide better representative results.

Temperature – the temperature plays a role as diffusion is a temperature controlled parameter. The Arrhenius equation applies, which in the case of testing at 10 °C and 25 °C amounts to about a factor of 2 difference in release. The temperature has also a strong effect on the solubility of some key substances. For translating effects from lab to field this factor can not be ignored.

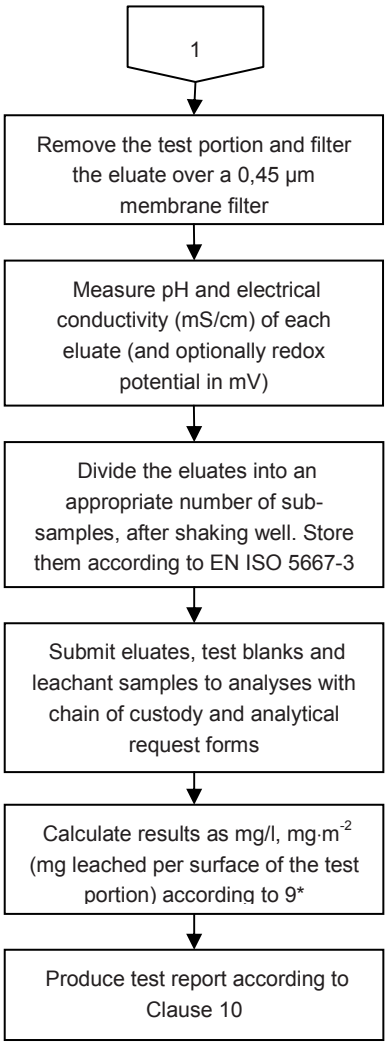
Annex D
(informative)

Process map for CEN/TS 15862



* Other test result expression :

For example for the purpose of comparison with historic data sets, the user can also express test results in different units (e.g. $mg \cdot kg^{-1}$ of dry mass). In that case some other information can be needed (e.g. determination of dry matter)


1
Remove the test portion and filter the eluate over a 0,45 µm membrane filter
Measure pH and electrical conductivity (mS/cm) of each eluate (and optionally redox potential in mV)
Divide the eluates into an appropriate number of sub-samples, after shaking well. Store them according to EN ISO 5667-3
Submit eluates, test blanks and leachant samples to analyses with chain of custody and analytical request forms
Calculate results as mg/l, mg·m-2 (mg leached per surface of the test portion) according to 9*
Produce test report according to Clause 10

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EN 12920:2006, *Characterisation of waste — Methodology for the determination of the leaching behaviour of waste under specified conditions*

CEN/TS 14405, *Characterisation of waste — Leaching behaviour tests — Up-flow percolation test (under specified conditions)*

CEN/TS 14429, *Characterisation of waste — Leaching behaviour tests — Influence of pH on leaching with initial acid/base addition*

CEN/TS 14997, *Characterisation of waste — Leaching behaviour tests — Influence of pH on leaching with continuous pH-control*

CEN/TS 15863, *Characterisation of waste — Leaching behaviour test for basic characterisation — Dynamic monolithic leaching test with periodic leachant renewal, under fixed test conditions*

CEN/TS 15864, *Characterisation of waste — Leaching behaviour test for basic characterisation — Dynamic monolithic leaching test with continuous leachant renewal under conditions relevant for specified scenario(s)*

EN 16192, *Characterisation of waste — Analysis of eluates*

AFNOR XP X 31-211:2000, *Waste — Test for the determination of the leachability of a solid waste material initally massive or generated by a solidification process*

NEN 7345:1995, *Leaching characteristics of solid earthy and stony building and waste materials — Leaching tests — Determination of the leaching of inorganic components from buildings and monolitic waste materials with the diffusion test*

ÖNORM S 2116-4, *Untersuchung verfestigter Abfälle — Elutionstests über 24 Stunden, 64 Tage, 2 Tage*

[1] POLDEN INSAVALOR, ECN (1992). Study of couples waste matrix / solidication reagents in relation to leaching. Theoretical and experimental aspects. Association RECORD, Contract no. 91-0305/1A (in French).

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