SLOVENSKI STANDARD
SIST EN 12457-3:2004

01-januar-2004

Karakterizacija odpadkov - Izluģevanje - Preskus skladnosti za izluģevanje granuliranih odpadkov in blata - 3. del: Dvostopenjski ģargģni preskus pri razmerju tekoļ e/trdno 2 l/kg in 8 l/kg za materiale z visoko vsebnostjo suhe snovi in z velikostjo delcev pod 4 mm (brez drobljenja ali z njim)

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

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Charakterisierung von Abfällen - Auslaugung - Übereinstimmungsuntersuchung für die Auslaugung von körnigen Abfällen und Schlämmen - Teil 3: Zweistufiges Schüttelverfahren mit einem Flüssigkeits-/Feststoffverhältnis von 2 l/kg und 8 l/kg für Materialen mit hohem Feststoffgehalt und mit einer Korngröße unter 4 mm (ohne oder mit Korngrößereduzierung)

Caractérisation des déchets - Lixiviation - Essai de conformité pour lixiviation des déchets fragmentés et des boues - Partie 3: Essai en bâchée double avec un rapport liquide-solide de 2 l/kg et de 8 l/kg pour des matériaux a forte teneur en solides et une granularité inférieure a 4 mm (sans ou avec réduction de la granularité)

Ta slovenski standard je istoveten z: **EN 12457-3:2002**

ICS:

13.030.10	Trdni odpadki	Solid wastes
13.030.20	Tekoļ i odpadki. Blato	Liquid wastes. Sludge

SIST EN 12457-3:2004 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

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EN 12457-3

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ICS 13.030.10; 13.030.20

English version

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

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

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Foreword

This document (EN 12457-3:2002) 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.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative annex ZA, which is an integral part of this document.

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 :

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DIN 38414-S4:1984

AFNOR X-31 210:1992

NEN 7343:1992

ÖNORM S 2072:1990

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

Annexes A, B, C, D, E and ZA 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.

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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:

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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 2 l/kg dry matter in a first step and subsequently of 8 l/kg dry matter in a second step. 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 eluates which shall subsequently be characterised physically and chemically according to appropriate standard methods.

This procedure is only applicable to waste material and sludges having a high solid content : the dry matter content ratio shall be at least higher than 33%. In addition, the necessary quantity of eluate in each step shall be obtained to perform the physical and chemical characterisation of the eluate. Furthermore, the minimum dry matter content ratio shall be high enough to allow a sufficient mixing of the leachant and the test portion.

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.

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NOTE 4 In relation with the minimum dry matter content ratio required for obtaining enough eluate in the first step, the test portion specified in this European Standard of 175 gram dry matter results in a maximum volume of eluate of 175 ml if its dry matter content ratio is 50 %.

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When the first step of this part 3 is not applicable, the leaching test at L/S = 10 specified in part 2 can be performed.

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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, Cl-, Co, Cr(VI), Cu, Mo, Ni, NO2-, Pb, total S, SO_4^{2-}, V and Zn*

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.*

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

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

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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

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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)

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3.15
test portion
amount or volume of the *test sample* taken for analysis, usually of known weight or volume (IUPAC definition)

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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 test is a serial batch leaching test consisting of two steps. 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 eluates 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 500 ml for the first step and a nominal volume of 2 litre for the second step, 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 500 ml and 2 litre respectively are selected in combination with the mass M_D of 175 gram as specified in 4.3.4 in order to minimise headspace. 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.

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

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.

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

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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.

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

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.

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

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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 :

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$$DR = 100 \times M_D/M_W \quad (1)$$

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 :

$$MC = 100 \times (M_W - M_D)/M_D \quad (2)$$

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 0,175 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.

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$$M_W = 100 \times M_D/DR \quad (3)$$

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 First leaching step

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- Place the test portion with the total mass M_w corresponding to 0,175 kg ± 0,005 kg of dry mass M_D in the bottle (4.2.1) of 500 ml.

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- Add an amount of leachant (L_2) establishing a liquid to solid ratio (L/S) = 2 l/kg ± 2 % during the first extraction. Care should be taken to obtain good mixing of solid and liquid.

$$L_2 = (2 - MC/100) \times M_D \quad (4)$$

Where

L_2 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 6 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 First liquid-solid separation step

- Allow the suspended solids to settle for 15 min ± 5 min.