



SLOVENSKI STANDARD

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Nadomešča:

SIST EN 12506:2004

SIST EN 13370:2004

Karakterizacija odpadkov - Analiza izlužkov

Characterization of waste - Analysis of eluates

Charakterisierung von Abfällen - Analyse von Eluaten

Caractérisation des déchets - Analyse des éluats

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ICS:

13.030.20 Tekoči odpadki. Blato Liquid wastes. Sludge

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EUROPEAN STANDARD

EN 16192

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Characterization of waste - Analysis of eluates

Caractérisation des déchets - Analyse des éluats

Charakterisierung von Abfällen - Analyse von Eluaten

This European Standard was approved by CEN on 15 October 2011.

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

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

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Foreword

This document (EN 16192:2011) has been prepared by Technical Committee CEN/TC 292 "Characterization 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of an identical text or by endorsement, at the latest by May 2012, and conflicting national standards shall be withdrawn at the latest by May 2012.

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 supersedes EN 12506:2003 and EN 13370:2003.

Details of significant technical changes between this European Standard and the previous edition are:

- This European Standard, EN 16192, is now a single document (instead of two) for the analysis of eluates specifying methods for the determination of the parameters pH, ammonium, AOX, As, Ba, Cd, Cl⁻, easily liberatable CN⁻, Co, Cr, Cr(VI), Cu, DOC/TOC, electrical conductivity, F⁻, Hg, Mo, Ni, NO₂⁻, Pb, phenol index, total S, Sb, Se, SO₄²⁻, TDS, V and Zn in aqueous eluates for the characterization of waste.
- In Clause 7 the parameters, previously described in two documents, are now all integrated in Table 1.
- In Table 1 for all parameters EN and ISO standards are updated, removed if withdrawn and new relevant standards are added, i.e.:
 - addition of the parameters Sb and Se together with the related analytical methods;
 - revision of the standards EN ISO 11885 (ICP-OES) and EN ISO 10304-1 (IC);
 - addition of the ICP-MS method (EN ISO 17294-1:2006 and EN ISO 17294-2:2004);
 - addition of the AAS with graphite furnace technique (EN ISO 15586:2003);
 - addition of the flow analysis techniques for Cl⁻ (EN ISO 15682:2001), Cr(VI) (EN ISO 23913:2009) and SO₄²⁻ (ISO 22743:2006);
 - addition of the parameter TDS (total dissolved solids) together with the related analytical method;
 - addition of the parameter DOC (dissolved organic carbon) to the parameter TOC (total organic carbon);
 - revision of the standards EN ISO 11732 (ammonium by flow analyser);
 - replacement of EN 1485 (AOX) by EN ISO 9562:2004;
 - revision and addition of new standards for Hg determination – EN 1483 and EN ISO 17852.
- In Annex B (informative) additional validation data are added obtained from a round robin test for the determination of Ba, Cd, Cr, Mo, Sb and Se in eluates and from round robin tests in the framework of acceptability of waste at landfills, both organized in Belgium.

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

Introduction

This European standard is intended to be used for the characterization of waste as defined in the Council Directive 75/442/EEC on waste, as amended by Council Directive 91/156/EEC of 18th March 1991, and national regulations, whose final destination for disposal is landfill. In the Council Decision of 19 December 2002 establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 of and Annex II to Directive 1999/31/EC, the test methods are described for determining the acceptability of waste at landfills. In section 3 of the Annex of this Decision the European standards EN 12506 and EN 13370 are included which are replaced by this European Standard.

This European Standard deals with the determination of chemical constituents, electrical conductivity, pH and total dissolved solids (TDS) in eluates which have been obtained by leaching of waste samples for example using EN 12457 "Characterization of waste - Leaching - Compliance test for leaching of granular waste materials and sludges" (Part 1 to Part 4). In principle, it may be used for the analysis of every kind of eluate as long as the performance characteristics of the applied analytical method fulfill the specific requirements.

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1 Scope

This European Standard specifies methods for the determination of the parameters pH, ammonium, AOX, As, Ba, Cd, Cl⁻, easily liberatable CN⁻, Co, Cr, Cr(VI), Cu, DOC/TOC, electrical conductivity, F⁻, Hg, Mo, Ni, NO₂⁻, Pb, phenol index, total S, Sb, Se, SO₄²⁻, TDS, V and Zn in aqueous eluates for the characterization of waste.

2 Normative references

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

EN 1483:2007, *Water quality — Determination of mercury — Method using atomic absorption spectrometry*

EN 1484:1997, *Water analysis — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

EN 15216:2007, *Characterization of waste — Determination of total dissolved solids (TDS) in water and eluates*

EN 26777:1993, *Water quality — Determination of nitrite — Molecular absorption spectrometric method (ISO 6777:1984)*

EN 27888:1993, *Water quality — Determination of electrical conductivity (ISO 7888:1985)*

prEN ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples (ISO/DIS 5667-3:2010)*

EN ISO 9562:2004, *Water quality — Determination of adsorbable organically bound halogens (AOX) (ISO 9562:2004)*

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EN ISO 10304-1:2009, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007)*

EN ISO 10304-3:1997, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate (ISO 10304-3:1997)*

EN ISO 11732:2005, *Water quality — Determination of ammonium nitrogen — Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732:2005)*

EN ISO 11885:2009, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885:2007)*

EN ISO 11969:1996, *Water quality — Determination of arsenic — Atomic absorption spectrometric method (hydride technique) (ISO 11969:1996)*

EN ISO 13395:1996, *Water quality — Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection (ISO 13395:1996)*

EN ISO 14402:1999, *Water quality — Determination of the phenol index by flow analysis (FIA and CFA) (ISO 14402:1999)*

EN ISO 14403:2002, *Water quality — Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403:2002)*

EN ISO 14911:1999, *Water quality — Determination of dissolved Li⁺, Na⁺, NH₄⁺, K⁺, Mn²⁺, Ca²⁺, Mg²⁺, Sr²⁺ and Ba²⁺ using ion chromatography — Method for water and waste water (ISO 14911:1998)*

EN ISO 15586:2003, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace (ISO 15586:2003)*

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EN ISO 15682:2001, *Water quality — Determination of chloride by flow analysis (CFA and FIA) and photometric and potentiometric detection (ISO 15682:2000)*

EN ISO 17294-1:2006, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines (ISO 17294-1:2004)*

EN ISO 17294-2:2004, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2:2003)*

EN ISO 17852:2008, *Water quality — Determination of mercury — Method using atomic fluorescence spectrometry (ISO 17852:2006)*

EN ISO 23913:2009, *Water quality — Determination of chromium(VI) — Method using flow analysis (FIA and CFA) and spectrometric detection (ISO 23913:2006)*

ISO 6439:1990, *Water quality — Determination of phenol index — 4-Aminoantipyrine spectrometric methods after distillation*

ISO 6703-2:1984, *Water quality — Determination of cyanide — Part 2: Determination of easily liberatable cyanide*

ISO 7150-1:1984, *Water quality — Determination of ammonium — Part 1: Manual spectrometric method*

ISO 8288:1986, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

ISO 9297:1989, *Water quality — Determination of chloride — Silver nitrate titration with chromate indicator (Mohr's method)*

ISO 9965:1993, *Water quality — Determination of selenium — Atomic absorption spectrometric method (hydride technique)*

ISO 10359-1:1992, *Water quality — Determination of fluoride — Part 1: Electrochemical probe method for potable and lightly polluted water*

ISO 10523:2008, *Water quality — Determination of pH*

ISO 11083:1994, *Water quality — Determination of chromium (VI) — Spectrometric method using 1,5-diphenylcarbazide*

ISO 22743:2006, *Water quality — Determination of sulfates — Method by continuous flow analysis (CFA)*

3 Terms and definitions

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

3.1

sample

portion of material selected from a larger quantity of material

3.2

eluate

solution obtained by a leaching test

3.3

laboratory sample

sample or subsample(s) sent to or received by the laboratory

3.4

test sample; analytical sample

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

3.5

test portion; analytical portion

quantity of material of proper size for measurement of the concentration or other properties of interest, removed from the test sample

NOTE 1 The test portion can be taken from the laboratory sample directly if no preparation of sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

NOTE 2 A unit or increment of proper homogeneity, size and fineness, needing no further preparation, can be a test portion.

3.6

leachant

aqueous solution used in a leaching test

3.7

leaching test

laboratory test for the determination of the release of matter from a waste into water or an aqueous solution

4 Sample pretreatment

The eluate shall be analyzed for the total content of its constituents. If precipitation occurs between the preparation of the eluate and the analysis it is necessary to ensure by appropriate methods (e.g. redissolution, separate analysis of solution and precipitate) that the total content of the parameters of interest is determined. If the eluate results from a procedure including 0,45 µm membrane filtration analytical results refer to the content dissolved by the leaching process.

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Eluates are susceptible to be changed to different extents as a result of physical, chemical or biological reactions which may take place between the time of leaching and the analysis. pH shall be determined immediately after preparation of the eluates and prior to sample pretreatment.

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It is therefore essential to take the necessary precautions to minimize these reactions and in the case of many parameters to analyze the eluate sample with a minimum of delay. The maximum delay is given in prEN ISO 5667-3 or in the respective analytical standards.

Precautions should be taken before and during transport as well as during the time in which the samples are preserved in the laboratory before being analyzed, to avoid alteration of the test portion.

Split the eluate in an adequate number of test portions for different chemical analyses and preserve them according to the requirements in the analytical standards or prEN ISO 5667-3.

One specific test portion may be an untreated aliquot of the laboratory sample for the analysis of chromates such as chloride, fluoride, sulfate, nitrite and chromium(VI) as well as for the determination of electrical conductivity.

For trace metal analysis test portions usually need to be acidified to $\text{pH} \leq 2$.

NOTE 1 For safety reasons it is recommended to acidify the test portion under a hood as volatile toxic substances can be generated.

NOTE 2 In cases where high contents of soluble solids are leached, acidification of the eluates can lead to precipitation of salts. This can be avoided by dilution prior to acidification.

5 Blank determination

The blank contribution of the applied analytical procedures shall be determined as described in the analytical standards and considered in the calculation of the results when appropriate.

6 Interference

A large number of compounds can interfere with the determination of the parameters concerned. These potential interferences are listed in the individual standards in question.

Several types of interference effects can contribute to inaccuracies in the determination of the various parameters, especially at low concentrations. These potential interference effects are listed in the individual standards and shall be considered separately for each analytical technique.

Chemical interferences are characterized by molecular compound formation, ionization effects, solute vaporization, precipitation and effects of decomposition of organic matter. Addition of buffer and/or preservation methods may reduce these effects.

Physical interferences can be caused by changes of viscosity and surface tension. They can cause significant inaccuracies especially in eluate samples containing high concentrations of acids and/or dissolved components. The colour or turbidity of eluates can cause interference in spectrophotometric determination.

7 Selection of the suitable test method

Select the appropriate standardized test method listed in Table 1 according to the type of waste eluate, the concentration range of the parameter of interest and the expected interferences.

For analytical quality control purposes ENV ISO 13530 and EN ISO/IEC 17025 should be considered.

It is pointed out that the standardized test methods listed in Table 1 have primarily been developed for the analysis of water samples. Most of them were validated in an interlaboratory trial for a limited number of waste eluate matrices (see Annex A). Their suitability for other waste eluates shall be checked in the laboratory performing the analysis. Additional validation data obtained in the evaluation of the analytical performance of laboratories are given in Annex B.

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Those standards cited in Table 1 that have not been validated in the CEN/TC 292 interlaboratory trial in 1999-2001, have the matrix waste water and/or leachates included in their scope, and they proved to be applicable for the analysis of eluates in routine analyses.

If the methods referred to in Table 1 are found to be inappropriate by reason of, for example, detection limits, repeatability or interferences, other methods validated for water analysis e.g. discrete analyzer, can be used. Their suitability for waste eluates shall be checked in the laboratory performing the analysis. The reason for the deviation shall be stated in the test report.

Table 1 — Parameters and test methods

Parameter	Test method
pH	ISO 10523:2008
Ammonium	EN ISO 11732:2005 EN ISO 14911:1999 ISO 7150-1:1984
AOX	EN ISO 9562:2004
As	EN ISO 11885:2009 EN ISO 11969:1996 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Ba	EN ISO 11885:2009 EN ISO 17294-1:2006 EN ISO 17294-2:2004

Table 1 (continued)

Parameter	Test method
Cd	ISO 8288:1986 EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Cl ⁻	ISO 9297:1989 EN ISO 10304-1:2009 EN ISO 15682:2001
CN ⁻ _{easily liberatable}	EN ISO 14403:2002 ^a ISO 6703-2:1984
Co	EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Cr	EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Cr(VI)	ISO 11083:1994 EN ISO 10304-3:1997 EN ISO 23913:2009
Cu	ISO 8288:1986 EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
DOC/TOC	EN 1484:1997
Electrical conductivity	EN 27888:1993
F ⁻	EN ISO 10304-1:2009 ^b ISO 10359-1:1992
Hg	EN 1483:2007 EN ISO 17852:2008
Mo	EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004

Table 1 (continued)

Parameter	Test method
Ni	ISO 8288:1986 EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
NO ₂ ⁻	EN 26777:1993 EN ISO 10304-1:2009 EN ISO 13395:1996
Pb	ISO 8288:1986 EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Phenol index	EN ISO 14402:1999 ^c ISO 6439:1990
Total S	EN ISO 11885:2009
Sb	EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Se	EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004 ISO 9965:1993
SO ₄ ²⁻	EN ISO 10304-1:2009 ISO 22743:2006
TDS	EN 15216:2007
V	EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
Zn	ISO 8288:1986 EN ISO 11885:2009 EN ISO 15586:2003 EN ISO 17294-1:2006 EN ISO 17294-2:2004
^a free cyanide is equivalent to easily liberatable cyanide for eluates with low organic content after distillation. ^b for eluates with low organic content. ^c after distillation.	