



SLOVENSKI STANDARD
SIST ENV 12506:2001
01-junij-2001

Characterization of waste - Analysis of eluates - Determination of pH, As, Cd, Cr VI, Cu, Ni, Pb, Zn, Cl, NO, SO

Characterization of waste - Analysis of eluates - Determination of pH, As, Cd, Cr VI, Cu, Ni, Pb, Zn, Cl, NO, SO

Charakterisierung von Abfällen - Analyse von Eluaten - Bestimmung von pH, As, Cd, Cr VI, Cu, Ni, Pb, Zn, Cl, NO, SO

Caractérisation des déchets - Analyse des éluats - Détermination du pH et dosage de As, Cd, Cr VI, Cu, Ni, Pb, Zn, Cl, NO, SO

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ICS:

13.030.20 Tekoči odpadki. Blato Liquid wastes. Sludge

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EUROPEAN PRESTANDARD
PRÉNORME EUROPÉENNE
EUROPÄISCHE VORNORM

ENV 12506

January 2000

ICS 13.030.40

English version

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

Caractérisation des déchets - Analyse des éluats -
Détermination du pH et dosage de As, Cd, Cr VI, Cu, Ni,
Pb, Zn, Cl, NO, SO

Charakterisierung von Abfällen - Analyse von Eluaten -
Bestimmung von pH, As, Cd, Cr VI, Cu, Ni, Pb, Zn, Cl, NO,
SO

This European Prestandard (ENV) was approved by CEN on 15 November 1999 as a prospective standard for provisional application.

The period of validity of this ENV 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 ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

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Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NNI.

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

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Introduction

SIST ENV 12506:2001

This 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.

It deals with the determination of pH and chemical constituents which have been extracted by leaching of waste samples for example using prEN 12457 „Characterization of waste - Leaching - Compliance test for leaching of granular waste materials - Determination of the leaching of constituents from granular waste materials and sludges“.

This European Standard is intended to define analytical methods for eluates. 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.

1 Scope

This European Standard specifies methods for the determination of parameters in aqueous eluates for the characterization of waste.

2 Normative references

This European Standard incorporates by dated or undated references, 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.

EN 26595:1992	Water quality - Determination of total arsenic - Silver diethyldithiocarbamate spectrophotometric method (ISO 6595:1982)
EN 26777:1993	Water quality - Determination of nitrite - Molecular absorption spectrometric method (ISO 6777:1984)
EN ISO 5667-3:1995	Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples (ISO 5667-3:1994)
EN ISO 10304-1:1995	Water quality - Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions - Part 1: Method for water with low contamination (ISO 10304-1:1992)
EN ISO 10304-2:1996	Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water (ISO 10304-2:1995)
EN ISO 11885:1997	Water quality - Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885:1996)
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 45001	General criteria for the operation of testing laboratories
ISO 8288:1986	Water quality - Determination of cobalt, nickel, copper, zinc, cadmium and lead - Flame atomic absorption spectrometric methods
ISO 9280:1990	Water quality - Determination of sulfate - Gravimetric method using barium chloride
ISO 9297:1989	Water quality - Determination of chloride - Silver nitrate titration with chromate indicator (Mohr's method)
ISO 10523:1994	Water quality - Determination of pH
ISO 11083:1994	Water quality - Determination of chromium (VI) - Spectrometric method using 1,5-diphenylcarbazide
ENV ISO 13530	Water quality - Guide to analytical quality control for water analysis (ISO TR 13530:1997)

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3 Definitions

For the purposes of this standard, the following 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.

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4 Sample pre-treatment

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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. No filtration or any kind of separation technique of the eluate is allowed. If the eluate comes from a procedure including 0,45 µm membrane filtration analytical results refer to the content dissolved by the leaching process.

Eluates are susceptible to being changed to differing 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 pre-treatment.

It is therefore essential to take the necessary precautions to minimize these reactions and in the case of many parameters to analyse the eluate sample with a minimum of delay. The maximum delay are given in EN ISO 5667-3:1995.

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 analysed, to avoid alteration of the test portion.

Split the eluate in an adequate number of test portions for different chemical analysis and preserve them according to the requirements in EN ISO 5667-3:1995. The following shall be noted:

- For trace metal analysis a test portion needs to be acidified to $\text{pH} \leq 2$ by addition of nitric acid.
- 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 diluting such samples 5 to 10 times prior to acidification. This additional dilution shall be taken into account in the calculations. A quantity of the undiluted test portion will have to be acidified and retained, in case the 5 to 10 dilution takes that sample below the detection limit for some determinants.

- Aliquot of the laboratory sample may have to be retained for analysis of anions such as chloride, sulfate, nitrite, chromium VI.

NOTE: Acidification of test portion under a hood, as volatile toxic substances can be generated.

5 Blank determination

The blank contribution of the leachant shall be determined and data obtained for the eluates corrected accordingly.

6 Interferences

Several types of interference effects can contribute to inaccuracies in the determination of the various parameters, especially trace elements. These potential interferences are listed in the individual standards in 2 normative references and the interferences shall be considered separately for each analytical technique.

Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization and precipitation effects. Such effects can be overcome by buffering the test portion and by standard addition procedures.

Physical interferences are generally considered to be effects caused by change 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 interferences in spectrometric determinations.

Other common interferences that can occur in aqueous solutions such as eluates are caused by decomposition of organic matter.

7 List of parameters and standardized test methods

The parameters given in table 1 have been selected following a consideration of the frequency at which the determinants are requested and the availability of standardized test methods developed by CEN/TC 230 or ISO/TC 147 Standards.

For the analytical determinations ENV ISO 13530 and EN 45001 should be considered.

NOTE: The analytical methods for determination of additional parameters will be issued later in a further European Standard.

It is pointed out that the standardized test methods listed in table 1 have primarily been developed for the analysis of water samples. Their suitability for the quantitative analysis of eluates for the characterization of wastes will have to be validated in the laboratory performing the analysis.

Table 1: Parameters and test methods

No	Parameter	Test method
1	pH ¹⁾	ISO 10523:1994
2	As	EN 26595:1992 EN ISO 11885:1997 EN ISO 11969:1996
3	Cd	ISO 8288:1986 EN ISO 11885:1997
4	Cr VI	ISO 11083:1994
5	Cu	ISO 8288:1986 EN ISO 11885:1997
6	Ni	ISO 8288:1986 EN ISO 11885:1997
7	Pb	ISO 8288:1986 EN ISO 11885:1997
8	Zn	ISO 8288:1986 EN ISO 11885:1997
9	Cl ⁻	ISO 9297:1989 EN ISO 10304-1:1995 EN ISO 10304-2:1996
10	NO ₂ ⁻	EN 26777:1993 EN ISO 10304-1:1995 EN ISO 10304-2:1996 EN ISO 13395:1996
11	SO ₄ ²⁻	ISO 9280:1990 EN ISO 10304-1:1995 EN ISO 10304-2:1996
¹⁾ At pH < 3 or pH > 10 use the same method but be aware that inaccuracy will increase.		

If the methods referred to in table 1 are found to be inappropriate by reason of for example detection limits, repeatability or interferences, other validated methods such as GF-AAS, ICP-MS can be used. The reason for the deviation shall be stated in the test report.

8 Expression of results

Specific instructions for the calculation of the results given in the individual analytical standards shall be strictly observed.

The results of the tests shall be expressed as a concentration of the constituents in the eluate, expressed in mg/l. The amount of constituent leached relative to the total mass of the sample, in mg/kg of dry matter, can be calculated using the liquid to solid ratio of the leaching test.

9 Test report

The work carried out by the testing laboratory shall be covered by a report which accurately, clearly and unambiguously presents the test results and all other relevant information.

Each test report shall include at least the following information:

- a) name and address of testing laboratory and location where the test was carried out when different from the address of the testing laboratory;
- b) unique identification of report (such as serial number) and of each page and total number of pages of the report;
- c) description and identification of the laboratory sample;
- d) date of receipt of laboratory sample and date(s) of performance of test;
- e) identification of the test specification or description of the method or procedure;
- f) description of eluate sampling and treatment, where relevant;
- g) any deviations, additions to or exclusions from the test specification, and any other information relevant to a specific test;
- h) identification of any non-standard test method or procedure utilized;
- i) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified;
- j) a statement on measurement uncertainty (where relevant);
- k) a signature and title or an equivalent marking of person(s) accepting technical responsibility for the test report and date of issue;
- l) a statement that the test results relate only to the laboratory sample;
- m) a statement that the report shall not be reproduced except in full without the written approval of the testing laboratory.

Corrections or additions to a test report after issue shall be made only by a further document suitably marked, e. g. "Amendment/Addendum to test report serial number (or as otherwise identified)", and shall meet the relevant requirements of the preceding paragraphs.

A test report shall not include any advice or recommendation arising from the test results.