



SLOVENSKI STANDARD

SIST ENV 13370:2002

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Characterization of waste - Analysis of eluates - Determination of Ammonium - N, AOX, conductivity, Hg, phenol index, TOC, CN easily liberatable, F

Characterization of waste - Analysis of eluates - Determination of Ammonium - N, AOX, conductivity, Hg, phenol index, TOC, CN- easily liberatable, F-

Charakterisierung von Abfällen - Analyse von Eluaten - Bestimmung von Ammonium-N, AOX, Leitfähigkeit, Hg, Phenolindex, TOC, CN- leicht freisetzbar, F-

Caractérisation des déchets - Analyse chimique des éluats - Détermination de: N ammoniacal, AOX, conductivité, Hg, "indice phéno". COT, CN- aisément libérables, F-

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EUROPEAN PRESTANDARD
PRÉNORME EUROPÉENNE
EUROPÄISCHE VORNORM

ENV 13370

May 2001

ICS 13.030.40

English version

**Characterization of waste - Analysis of eluates - Determination
of Ammonium - N, AOX, conductivity, Hg, phenol index, TOC,
CN⁻ easily liberatable, F⁻**

Caractérisation des déchets - Analyse chimique des éluats
- Détermination de: N ammoniacal, AOX, conductivité, Hg,
"indice phénol", COT, CN⁻ aisément libérables, F⁻

Charakterisierung von Abfällen - Analyse von Eluaten -
Bestimmung von Ammonium-N, AOX, Leitfähigkeit, Hg,
Phenolindex, TOC, CN⁻ leicht freisetzbar, F⁻

This European Prestandard (ENV) was approved by CEN on 16 March 2001 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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Foreword

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

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 13370:2002

This European Prestandard 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. Characterization of waste is especially needed for the Hazardous Waste Directive (91/689/EEC) and the Landfill Directive (99/31/EC) and national regulations on waste management.

It deals with the determination of conductivity and the chemical determination of compounds which have been extracted by leaching of waste samples such as prEN 12457:1999 „Characterization of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Determination of the leaching of constituents from granular waste materials and sludges“.

This European Prestandard is intended to define analytical methods for eluates. A large number of compounds may interfere with the determination of the parameters concerned. These potential interferences are listed in the individual standards in question.

1 Scope

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

2 Normative references

This European Prestandard 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 Prestandard only when incorporated in it by amendment or

revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1483:1997	Water quality - Determination of mercury
EN 1484:1997	Water analysis - Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)
EN 1485:1996	Water quality - Determination of adsorbable organically bound halogens (AOX)
EN 27888:1993	Water quality - Determination of electrical conductivity (ISO 7888:1985)
EN 45001	General criteria for the operation of testing laboratories
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 11732:1997	Water quality - Determination of ammonium nitrogen by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732:1997)
ENV ISO 13530	Water quality - Guide to analytical quality control for water analysis (ISO/TR 13530:1997)
prEN 12457-1:1999	Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 1: One stage batch test at a liquid to solid ratio of 2 l/kg with particle size below 4mm (without or with size reduction)
prEN 12457-2:1999	Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 2: One stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 4mm (without or with size reduction)
prEN 12457-3:1999	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 and 8 l/kg with particle size below 4 mm (without or with size reduction)
prEN 12457-4:1999	Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 4: One stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 10 mm (without or with size reduction)
EN ISO 5667-3:1995	Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples (ISO 5667-3:1994)
ENV 12506:2000	Characterization of waste - Analysis of eluates - Determination of pH, As, Cd, Cr VI, Cu, Ni, Pb, Zn, Cl ⁻ , NO ₂ ⁻ , SO ₄ ²⁻
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 10359-1:1992	Water quality - Determination of fluoride - Part 1: Electrochemical probe method for potable and lightly polluted water
EN ISO 14402:1999	Water quality - Determination of phenol index by flow analysis (FIA and CFA) (ISO 14402:1999)
prEN ISO14403:1998	Water quality - Determination of total cyanide and free cyanide by continuous flow analysis (ISO/DIS 14403:1998).

3 Terms and definitions

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

3.1

leaching test

laboratory test for the determination of the release of matter from a waste into water or an aqueous solution [ENV 12506:2000]

3.2

leachant

aqueous solution used in a leaching test [ENV 12506:2000]

3.3

eluate

solution obtained by a leaching test [ENV 12506:2000]

3.4

sample

portion of material selected from a larger quantity of material [ENV 12506:2000]

3.5

laboratory sample

sample or subsample(s) sent to or received by the laboratory [ENV 12506:2000]

3.6

test sample; analytical sample

sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis [ENV 12506:2000]

3.7

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 [ENV 12506:2000]

NOTE 1 The test portion may 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, may be a test portion.

4 Sample pre-treatment

The eluate shall be analysed for its total content. 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. 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. 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. Conductivity shall be determined immediately after preparation of the eluates and prior to sample pre-treatment.

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:

- In cases where high contents of soluble solids are leached, acidification of the eluates may lead

to precipitation of salts. This can be avoided by diluting such samples in an adequate way prior to acidification. This additional dilution shall be taken into account in the calculations. A quantity of the undiluted test sample will have to be acidified and retained, in case the dilution takes that sample below the detection limit for some determinants.

- Aliquots of the laboratory sample may have to be retained for analysis of anions, due to interferences from the counter-ions added by the acidification.

NOTE It is recommended that the addition of chemicals to the test portions is carried out under a hood, as volatile toxic substances may 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 (physical, chemical and biological) may contribute to inaccuracies in the determination of the various parameters, especially trace elements. These potential interferences are listed in the individual standards (see Normative references) and the interferences shall be considered separately for each analytical technique.

Chemical interferences are characterized by molecular compound formation, ionization effects and 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 when analysing eluate samples containing high concentrations of acids and/or dissolved components.

The colour or turbidity of eluates may cause interferences in spectrometric determinations.

Other common interferences that may 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 the standards ENV ISO 13530 and EN 45001 should be considered.

NOTE The analytical methods for determination of additional parameters needed for the characterization of eluates but not dealt with ENV 12506:2000 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	Standard
1	Ammonium - N	EN ISO 11732:1997 ISO 7150-1:1984
2	AOX	EN 1485:1996
3	conductivity	EN 27888:1993
4	Hg	EN 1483:1997
5	phenol index ¹⁾	ISO 6439:1990 EN ISO 14402:1999
6	TOC	EN 1484:1997
7	CN ⁻ easily liberatable	ISO 6703-2:1984 prEN ISO 14403:1998
8	F ⁻	EN ISO 10304-1:1995 ISO 10359-1:1992

¹⁾ after distillation

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 may 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.

Except for conductivity the results of the tests shall be expressed as a concentration of the constituents in the eluate, expressed in mg/l. The mass 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.