BRITISH STANDARD

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13370:2003

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

The European Standard EN 13370:2003 has the status of a British Standard

ICS 13.030.99

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 13370:2003. It supersedes DD ENV 13370:2001 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee B/508, Waste management, to Subcommittee B/508/3, Waste characterisation, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

Caractérisation des déchets - Analyse chimique des éluats - Détermination de l'ammonium, des AOX, de la conductivité, du Hg, de l'indice phénol, du COT, des CN^- aisément libérables et des F^-

Charakterisierung von Abfällen - Chemische Analyse von Eluaten - Bestimmung von Ammonium, AOX, Leitfähigkeit, Hg, Phenolindex, TOC, leicht freisetzbarem CN^-, F^-

This European Standard was approved by CEN on 25 March 2003.

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 Management Centre or to any CEN member.

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

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Foreword

This document (EN 13370:2003) 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 December 2003, and conflicting national standards shall be withdrawn at the latest by December 2003.

This document supersedes ENV 13370:2001.

Annex A is informative.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

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 conductivity and chemical constituents which have been extracted by leaching of waste samples for example using EN 12457 Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges – Part 1 - 4.

This European Standard together with EN 12506 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.

For the analytical determinations ENV ISO 13530 and EN ISO/IEC 17025 should be considered.

1 Scope

This European Standard specifies methods for the determination of the parameters Ammonium, AOX, conductivity, Hg, phenol index, TOC, easily liberatable CN^-, F^- in aqueous eluates for the characterization of waste.

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).

EN 1483	Water quality - Determination of mercury
EN 1484	Water analysis - Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)
EN 1485	Water quality - Determination of adsorbable organically bound halogens (AOX)
EN 27888	Water quality - Determination of electrical conductivity (ISO 7888:1985)
EN ISO 5667-3	Water quality - Sampling - Part 3: Guidance on the preservation and handling of samples (ISO 5667-3:1994)
EN ISO 10304-1	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 14402	Water quality - Determination of the phenol index by flow analysis (FIA and CFA)(ISO 14402:1999)
EN ISO 14403:1998	Water quality – Determination of total cyanide and free cyanide by continuous flow analysis.(ISO 14403:2002)

EN ISO 11732	Water quality - Determination of ammonium nitrogen by flow analysis (CFA and FIA) and spectrometric detection
ISO 6439	Water quality - Determination of phenol index - 4-Aminoantipyrine spectrometic methods after distillation
ISO 6703-2	Water quality - Determination of cyanide - Part 2: Determination of easily liberatable cyanide
ISO 7150-1	Water quality - Determination of ammonium – Part 1: Manual spectrometric method
ISO 10359-1	Water quality - Determination of fluoride - Part 1: Electrochemical probe method for potable and lightly polluted water

3 Terms and definitions

For the purposes of this European Standard 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 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. redisolution, 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.

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. Conductivity 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 is given in EN ISO 5667-3 or in the 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 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 the analytical standards or EN ISO 5667-3.

One specific test portion may be an untreated aliquot of the laboratory sample for analysis of conductivity and fluoride.

For analysis of some parameters test portions need to be acidified.

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 analysing procedures shall be determined as described in the analytical standards and considered in the calculation of the results when appropriate.

6 Interference

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 interference is 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 interference 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 spectrometric determination.

Standard addition may be used to visualise and correct for interference effects (see ISO 8466-3).

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 interference.

It is pointed out that the standardized test methods listed in Table 1 have primarily been developed for the analysis of water samples. They were validated in a new interlaboratory trial for a limited number of waste eluate matrices (see annex A) performed by CEN/TC 292. Their suitability for other waste eluates has to be checked in the laboratory performing the analysis.

If the methods referred to in Table 1 are found to be inappropriate by reason of, for example, detection limits, repeatability or interference, other methods validated for water analysis such as AFS, ICP-MS can be used. Their suitability for waste eluates has to 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
Ammonium	EN ISO 11732 ISO 7150-1
AOX	EN 1485
conductivity	EN 27888
Hg	EN 1483
phenol index	ISO 6439 EN ISO 14402[a]
TOC	EN 1484
CN^- easily liberatable	ISO 6703-2 EN ISO 14403 [b]
F^-	EN ISO 10304-1 [c] ISO 10359-1
[a] after distillation [b] free cyanide is equivalent to easily liberatable cyanide [c] for eluates with low organic contamination	

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 except for conductivity 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;

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) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified;

i) a statement on measurement uncertainty (where needed);

j) a signature and title or an equivalent marking of person(s) accepting technical responsibility for the test report and date of issue;

k) a statement that the test results relate only to the laboratory sample;

l) a statement that the report shall not be reproduced except in full without the written approval of the testing laboratory.

Annex A
(informative)

Validation of EN 13370

A .1 General

During 1999 - 2001 a project for validation of this standard has been organised and carried out.

The validation included an interlaboratory study for evaluation of performance characteristics of methods included in this standard (reproducibility and repeatability).

A.2 Interlaboratory study

The purpose of the validation trial was to check the suitability of the cited standards for analysis of waste eluates.

The study was not focused on performance characteristics of the analytical methods as they have been validated by ISO or CEN on different water matrices.

A.3 Selection of Laboratories

A questionnaire has been circulated by all CEN/TC292/WG3 and WG 2 members to collect a list of interested European laboratories. 41 laboratories gave their availability to participate to the inter-laboratory trial. All of them were asked to declare that they fulfil the minimum requirements to carry out the analyses according to the standards. According to ISO 5725 series no selection has been made in advance on the basis of the supposed "ability" of laboratories, their certifications, etc: it's therefore possible to assume that participating laboratories are a rather good "sample" of "normal" European laboratories.

A.4 Selection of samples

To test the analytical procedures on a proper number of eluates, four different materials were considered to produce bulk amounts of waste eluates.

— contaminated soil (COS):

— sewage sludge (SEW):

— sand blasting waste (SBW):

— fly ash filter cake from municipal solid waste incinerator (FFC).

Additionally, two synthetic solutions were prepared (SYN1, SYN2).

The analytical procedures mentioned in the cited standards require different conservation methods. Since it was impossible to produce waste eluates using all the different conservating agents and methods according to the corresponding standards, it was decided to use only two conservation methods:

— addition of HNO_3 to a pH of about 2:

— no conservation agents but storage of the eluates at 4 °C in the dark.

The waste eluates and synthetic solutions stabilised with HNO_3 were filled into polyethylene bottles and other samples into glass bottles. Table A.1 shows the different conservation procedures as mentioned in the standards and used in this trial.

A.5 Validation scope

In this trial not all parameter/method combinations were validated in the different samples. In Table A.1 an overview is given of the validation scope.

The parameter/method combinations marked with an **x** in Table A.1 were successfully validated. A remark is given in Table A.1 for those parameter/method combinations where the validation was not successful, because:

— The number of participants and/or the number of results was too low.

— Matrix dependent interference.

In the other cases no validation study was performed.

Complimentary some of these standards have been validated by ISO on waste water.

Table A.1 — Validated parameter/method/sample combinations

Parameter	Standard	Prescribed conservation in standard	Used Conservation in trial	COS	FFC	SBW	SEW	SYN1	SYN2	Validated by ISO on waste water
Ammonium-NH_4	ISO 7150-1	Immediate analysis or H_2SO_4 pH 2	None, 4 °C				x		x	
Ammonium-NH_4	EN ISO 11732	Immediate analysis or H_2SO_4 pH 2	None, 4 °C				x		x	x
AOX	EN 1485	HNO_3 pH 2	HNO_3 pH 2	x			x			x
Conductivity	EN 27888	None, 4 °C	None, 4 °C	x	x	x	x	x	x	x
Hg	EN 1483	$K_2Cr_2O_7$ pH 1	HNO_3 pH 2				[a]		x	x
Phenol index	ISO 6439	H_3PO_4 $CuSO_4$ pH 4	None, 4 °C			x			[a]	
Phenol index	EN ISO 14402	Immediatean alysis or H_2SO_4 pH 2	None, 4 °C			[a]			x	x
TOC	EN 1484	H_3PO_4 pH 2	HNO_3 pH 2	x			x			x
Cyanide easily liberatable	ISO 6703-2	NaOH pH 8	None, 4 °C				[a b]	[a]	x	
Cyanide easily liberatable	EN ISO 14403	NaOH pH 12	None, 4 °C					x	[a]	x
F	EN ISO 10304-1	None, 4 °C	None, 4 °C		x	x				
F	ISO 10359-1	None, 4 °C	None, 4 °C		x	x				

[a] Validation failed because less than 6 labs and/or 18 valid results were available.

[b] Validation failed because coloured solutions cannot be analysed by this method (see Chapter 6).

A.6 Results and statistics

In Table A.2 – Table A.7 an overview is given of the results of the interlaboratory study.

The data from the validation have been assessed according to ISO 5725-2. Further the results have been filtered by acceptance criteria.

This report shows the tested parameter; the accepted combination of method, parameter and sample; and the results and statistics.

Results from rejected combinations of methods, parameter and samples are not shown in the result tables.

The acceptance criterion was:

— Minimum number of laboratories: 6.

— Minimum number of results (outliers excluded): 18.

For a given parameter and where available F-test on the sR and t-test for the means have been run for comparing alternative methods at the 99 % confidence interval. Where one of the tests failed a remark is given in the tables.

Table A.2 — Results of the interlaboratory study on validation of methods for eluate analysis (EN 13370) – Contaminated Soil Eluate (COS)

Parameter	Standard	Units	Number of					Mean	s_r	s_R	S_r	S_R	Remarks
			Labs total	Labs accepted	Values total	Values accepted	Outliers				%	%	
AOX	EN 1485	µg/l	14	13	42	38	4	27,8	2,43	7,73	8,75	27,8	
Conductivity	EN 27888	µS/cm	12	11	36	30	6	2450	7,35	207,8	0,3	8,48	
TOC	EN 1484	mg/l	15	13	45	39	6	16,3	0,30	1,54	1,82	9,42	

S_r is the relative repeatability
S_R is the relative reproducibility

Table A.3 — Results of the interlaboratory study on validation of methods for eluate analysis (EN 13370) – Sewage Sludge Eluate (SEW)

Parameter	Standard	Units	Number of					Mean	s_r	s_R	S_r	S_R	Remarks
			Labs total	Labs accepted	Values total	Values accepted	Outliers				%	%	
AOX	EN 1485	mg/l	13	11	39	32	7	0,153	0,011	0,061	7,16	39,95	
Conductivity	EN 27888	µS/cm	13	10	37	28	9	3530	11,6	469	0,33	13,3	
NH_4 – FIA/CFA	EN ISO 11732	mg/l	11	11	34	34	-	401	5,77	81,3	1,44	20,28	F-test failed
NH_4 - Photometry	ISO 7150-1	mg/l	11	10	33	30	3	360	8,03	152	2,23	42,11	F-test failed
TOC	EN 1484	mg/l	15	13	47	41	6	1560	40,3	99,7	2,58	6,39	

Table A.4 — Results of the interlaboratory study on validation of methods for eluate analysis (EN 13370) – Sandblasting Waste Eluate (SBW)

Parameter	Standard	Units	Number of					Mean	s_r	s_R	S_r	S_R	Remarks
			Labs total	Labs accepted	Values total	Values accepted	Outliers				%	%	
Conductivity	EN 27888	µS/cm	12	11	33	31	2	530	2,12	23,5	0,4	4,43	
F - Electrode	ISO 10359-1	mg/l	10	10	30	30	-	7,66	0,060	0,99	0,78	12,91	
F - IC	EN ISO 10304-1	mg/l	11	11	33	33	-	7,42	0,153	1,23	2,06	16,57	
Phenol index - Photometry	ISO 6439	mg/l	8	8	24	24	-	0,246	0,0187	0,095	7,62	38,6	

Table A.5 — Results of the interlaboratory study on validation of methods for eluate analysis (EN 13370) – Flyash-Filtercake Eluate (FFC)

Parameter	Standard	Units	Number of					Mean	s_r	s_R	S_r	S_R	Remarks
			Labs total	Labs accepted	Values total	Values accepted	Outliers				%	%	
Conduct. electrode	EN 27888	µS/cm	12	12	33	33	-	2880	13,5	240	0,47	8,34	
F - Electrode	ISO 10359-1	mg/l	9	9	27	21	6	0,709	0,023	0,086	3,25	12,15	
F – IC	EN ISO 10304-1	mg/l	8	7	24	20	4	0,629	0,0085	0,123	1,35	19,52	

Table A.6 — Results of the interlaboratory study on validation of methods for eluate analysis (EN 13370) – Synthetic Eluate 1 (SYN1)

Parameter	Standard	Units	Number of					Mean	s_r	s_R	S_r	S_R	Remarks
			Labs total	Labs accepted	Values total	Values accepted	Outliers				%	%	
CN – CFA	EN ISO 14403	mg/l	7	7	22	22	-	0,175	0,0071	0,087	4,08	49,62	
Conductivity	EN 27888	µS/cm	13	11	37	31	6	353	5,26	17,4	1,49	4,92	

Table A.7 — Results of the interlaboratory study on validation of methods for eluate analysis (EN 13370) – Synthetic Eluate 2 (SYN2)

Parameter	Standard	Units	Number of					Mean	s_r	s_R	S_r	S_R	Remarks
			Labs total	Labs accepted	Values total	Values accepted	Outliers				%	%	
CN - Photometry	ISO 6703-2	mg/l	6	6	18	18	-	0,041	0,003	0,006	6,7	13,7	
Conductivity	EN 27888	µS/cm	13	13	36	36	-	478	2,92	21,6	0,61	4,51	
Hg - CV-AAS	EN 1483	µg/l	12	11	36	33	3	6,25	0,13	1,36	2,1	21,8	
NH_4 – FIA/CFA	EN ISO 11732	mg/l	10	8	32	26	6	0,248	0,013	0,067	5,22	27,17	F-test failed
NH_4 - Photometry	ISO 7150-1	mg/l	11	9	33	27	6	0,223	0,007	0,019	3,26	8,31	F-test failed
Phenol index – FIA/CFA	EN ISO 14402	mg/l	6	6	18	18	-	0,067	0,0028	0,016	4,16	24,5	

A.7 Conclusion

The validation of this standard was performed on a selection of waste and synthetic eluates. For some parameters different analytical methods were validated.

For some methods validation data are available for at least two eluates per parameter. In the case of Hg (EN 1483), CN (ISO 6703-2, EN ISO 14403), phenol index (ISO 6439, EN ISO 14402) only one matrix was validated.

For the analyses of a given parameter within a specific matrix, it is the responsibility of the laboratory to choose the appropriate analytical method depending on the expected interference and concentration range as mentioned in the according standards.

There is no international standard on equivalence testing between alternative physical or chemical methods available. However based on the F-test and the t-test for means (used are the s_R values) there is a realistic chance to prove equivalence between the following method/matrix combinations:

— for SBW F-electrode (ISO 10359-1), F-IC (EN ISO 10304-1),

— for FFC F-electrode (ISO 10359-1), F-IC (EN ISO 10304-1).

Bibliography

EN 12457-1:2002	Characterization 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 4 mm (without or with size reduction)
EN 12457-2:2002	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 4 mm (without or with size reduction)
EN 12457-3:2002	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 with particle size below 4 mm (without or with size reduction)
EN 12457-4:2002	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 12506:2003	Characterization of waste - Analysis of eluates - Determination of pH, As, Ba, Cd, Cl-, Co, Cr Vl, Cu, Mo, Ni, NO2-, Pb, total S, SO42-, V and Zn
ENV ISO 13530:1998	Water quality - Guide to analytical quality control for water analysis (ISO TR 13530:1997)
ISO 5725-1 + Cor 1	Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions
ISO 5725-2	Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement met
ISO 5725-3 + Cor 1	Accuracy (trueness and precision) of measurement methods and results - Part 3: Intermediate measures of the precision of a standard measurement method
ISO 5725-4	Accuracy (trueness and precision) of measurement methods and results - Part 4: Basic methods for the determination of the trueness of a standard measurement method
ISO 5725-5	Accuracy (trueness and precision) of measurement methods and results - Part 5: Alternative methods for the determination of the precision of a standard measurement method
ISO 5725-6 + Cor 1	Accuracy (trueness and precision) of measurement methods and results - Part 6: Use in practice of accuracy values
EN ISO/IEC 17025:2000	General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)
75/442/EEC	Council Directive of 15 July 1975 on waste
91/156/EEC	Council Directive of 18 March 1991 amending Directive 75/442/EEC on waste (91/156/EEC)

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