

Lead and lead alloys — Analysis by flame atomic absorption spectrometry (FAAS) or inductively coupled plasma emission spectrometry (ICP-ES), without separation of the lead matrix

ICS 77.120.60

National foreword

This Draft for Development is the English language version of ENV 13800:2000.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature because, although the analytical procedures in the method are generally recognized, the evaluation of the method via 'Round-Robin' analyses, together with the resultant statistical validation, as required by CEN has not yet been carried out. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained.

Comments arising from the use of this Draft for Development are requested. The results of the evaluation of this Draft for Development and the comments arising from its application by CEN members will be reported to the European organization responsible for its conversion into a European Standard. A review of this publication will be initiated 2 years after its publication by the European organization so that a decision can be taken on its status at the end of its three-year life. The commencement of the review period will be notified by an announcement in *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the prestandard or to withdraw it. Comments should be sent in writing to the Secretary of BSI Technical Committee NFE/22, Lead and lead alloys, at 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

A list of organizations represented on this committee can be obtained on request from the secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled 'International Standards Correspondance Index', or by using the 'Find' facility of the British Standards Electronic Catalogue.

Summary of pages

This document comprises a front cover, an inside front cover, the ENV title page, pages 2 to 18, an inside back cover and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

This Draft for Development, having been prepared under the direction of the Engineering Sector Committee, was published under the authority of the Standards Committee and comes into effect on 15 September 2000

© BSI 09-2000

ISBN 0 580 36181 0

Amendments issued since publication

Amd. No.	Date	Comments

English version

Lead and lead alloys - Analysis by flame atomic absorption spectrometry (FAAS) or inductively coupled plasma emission spectrometry (ICP-ES), without separation of the lead matrix

Plomb et alliages de plomb - Analyse par spectrométrie d'absorption atomique dans la flamme (FAAS) ou spectrométrie d'émission à plasma inductif couplé (ICP-ES), sans séparation de la matrice plomb

Blei und Bleilegierungen - Analyse durch Flammen - Atomabsorptionsspektrometrie (FAAS) oder Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES), ohne Abtrennung der Bleimatrix

This European Prestandard (ENV) was approved by CEN on 25 June 2000 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.



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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Principle	5
3.1 Dissolution.....	5
3.2 Instrumental techniques	5
4 Apparatus	6
4.1 General.....	6
4.2 Volumetric glassware	6
4.3 Plastic ware	6
4.4 Instruments.....	6
5 Reagents	7
5.1 General.....	7
5.2 Nitric acid (HNO ₃)	7
5.3 Tartaric acid solution (C ₄ H ₆ O ₆) 300 g/l	7
5.4 Hydrochloric acid (HCl)	7
5.5 Fluoboric acid (HBF ₄).....	7
5.6 Lead for matrix matching	7
5.7 Standard solutions	8
6 Sampling and sample preparation	10
7 Procedure	10
7.1 Preparation of the test solution.....	10
7.2 Preparation of the blank test solution	11
7.3 Dilution of the test solutions and of the blank test solution	11
8 Determination of elements by FAAS.....	11
8.1 Preparation of calibration solutions	11
8.2 Calibration, measurements and results	13
9 Determination of elements by ICP-ES	15
9.1 Preparation of calibration solutions	15
9.2 Calibration, measurements and results	16
10 Test report	18

Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 306 "Lead and lead alloys", the secretariat of which is held by AFNOR.

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.

CAUTION FOR SAFETY AND TRAINING

The methods in this standard are recommended for the certification of reference materials and as umpire methods in cases of a dispute. The importance of either application, and the paramount issue of safety, requires that they should only be carried out by fully-trained analysts who are experienced in all relevant techniques and the precautions necessary in the inherently hazardous environs of a laboratory, especially those required when using particularly hazardous apparatus and reagents used in some of these methods.

Where a particular hazard exists, this is given as a **DANGER** adjacent to the point in the text where the apparatus or reagent is referenced.

1 Scope

This European Prestandard specifies methods of analysis using flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-ES) for the determination of element contents in lead and lead alloys for the ranges given in Table 1.

Table 1 — Ranges of application for the determination of element contents

Element	Ranges of application (% m/m)			
	FAAS		ICP-ES	
Ag	0,0005	- 1	0,0001	- 1
Al	0,005	- 0,04	0,0005	- 0,05
As	0,005	- 0,25	0,002	- 0,3
Ba	0,0025	- 0,025	0,0002	- 0,005
Bi	0,0025	- 0,2	0,002	- 0,2
Ca	0,001	- 0,2	0,0005	- 0,2
Cd	0,001	- 0,2	0,0001	- 0,2
Cu	0,0005	- 0,06	0,0001	- 0,06
Na	0,001	- 0,02	0,001	- 0,02
Ni	0,0005	- 0,005	0,0005	- 0,005
S		-	0,002	- 0,01
Sb	0,0025	- 10	0,002	- 10
Se	0,005	- 0,04	0,005	- 0,04
Sn	0,005	- 2	0,002	- 2
Te	0,0025	- 0,05	0,002	- 0,05
Tl	0,0025	- 0,05	0,0025	- 0,05
Zn	0,0005	- 0,01	0,0002	- 0,01

These methods are intended as the definitive methods in case of dispute for the determination of elements at low content in lead. They are also recommended for the analysis of Certified Reference Materials (CRM) and Reference Materials (RM) which are used in analysis according to ENV 12908.

2 Normative references

This European Prestandard 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 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 12402, *Lead and lead alloys - Methods of sampling for analysis.*

ENV 12908, *Lead and lead alloys - Analysis by Optical Emission Spectrometry (OES) with spark excitation.*

ISO 648:1977, *Laboratory glassware – One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware – One-mark volumetric flasks*.

ISO 3696, *Water for analytical laboratory use - Specification and test methods*.

3 Principle

3.1 Dissolution

Dissolution of a test portion in, either :

- nitric acid ; or
- mixture of nitric acid/tartaric acid ; or
- mixture of nitric acid/fluoboric acid.

Dilution of the test solution to a defined volume, and determination of the analyte concentration using one of the two techniques described in 3.2.

3.2 Instrumental techniques

3.2.1 Flame atomic absorption spectrometry (FAAS)

The analyte concentration in the test solution is obtained by :

- nebulization of the test solution into the flame of an atomic absorption spectrometer ;
- measurement of the absorption of the resonance line energy of the spectrum from the element at the relevant wavelength (absorbency) ;
- comparison with that of matrix-matched calibration solutions of the same element.

3.2.2 Inductively coupled plasma emission spectrometry (ICP-ES)

The analyte concentration in the test solution is obtained by :

- nebulization of the test solution into the plasma of an inductively coupled plasma optical emission spectrometer ;
- measurement of the intensity of the emission signal from the spectrum of the elements to be determined at the relevant wavelength ;
- comparison with that of matrix-matched calibration solutions of the same element.