

ICS 77.120.60

English version

Lead and lead alloys - Analysis by flame atomic absorption spectrometry (FAAS) or inductively coupled plasma emission spectrometry (ICP-ES), after separation by co-precipitation

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

Blei und Bleilegierungen - Analyse durch Flammen-Atomabsorptionsspektrometrie (FAAS) oder Emission-Spektrometrie mit induktiv gekoppeltem Plasma (ICP-ES), nach Abtrennung durch Mitfällung

This European Prestandard (ENV) was approved by CEN on 18 November 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.



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

	page
Foreword.....	4
1 Scope	5
2 Normative references	5
3 Principle	5
3.1 Preparation of the test solution.....	5
3.2 Instrumental techniques	6
3.2.1 Flame atomic absorption spectrometry (FAAS)	6
3.2.2 Inductively coupled plasma emission spectrometry (ICP-ES).....	6
4 Apparatus	6
4.1 General.....	6
4.2 Volumetric glassware	6
4.3 Filtration system	6
4.4 Instruments.....	6
4.4.1 Flame atomic absorption spectrometer	6
4.4.2 Inductively coupled plasma emission spectrometer	7
5 Reagents	7
5.1 General.....	7
5.2 Nitric acid (HNO ₃)	7
5.2.1 Concentrated nitric acid.....	7
5.2.2 Nitric acid 1:1 (V/V)	7
5.2.3 Nitric acid 1:2 (V/V)	7
5.3 Hydrochloric acid (HCl)	7
5.3.1 Concentrated hydrochloric acid.....	7
5.3.2 Hydrochloric acid 1:1 (V/V)	7
5.4 Ammonia solution.....	7
5.5 Pure lead	7
5.6 Manganese nitrate solution	8
5.7 Potassium permanganate solution	8
5.8 Standard solutions	8
5.8.1 Standard solutions (1g/l).....	8
5.8.2 Standard solutions (100 mg/l).....	8
5.8.3 Standard solutions (10 mg/l).....	9
5.8.4 Multi-element solutions (either 10 mg/l or 100 mg/l).....	9
6 Sampling and sample preparation	9
7 Procedure	9
7.1 Preparation of the test solution.....	9
7.1.1 Dissolution.....	9
7.1.2 Co-precipitation with manganese dioxide.....	9
7.1.3 Test solution.....	9
7.2 Preparation of the synthetic test solution.....	9
7.3 Preparation of the blank test solution	10
7.4 Preparation of calibration solutions	10
8 Determination of elements by FAAS.....	10
8.1 General.....	10
8.2 Calibration and measurement of the test solution	11
8.3 Expression of results	11
8.3.1 Analyte recovery rate	11
8.3.2 Analyte content in the sample	11
9 Determination of elements by ICP-ES	12
9.1 General.....	12
9.2 Calibration and measurement of the test solution	12

9.3 Expression of results13

9.3.1 Analyte recovery rate13

9.3.2 Analyte content in the sample.....13

10 Test report13

Bibliography14

This document is a preview generated by EVS

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 Prestandard 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 using flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-ES) for the determination of elements at low content in lead for the ranges given in Table 1.

Higher contents than those listed in Table 1 should be determined according to ENV 13800.

Table 1 — Ranges of application for the determination of elements

Element	Ranges of applications (% m/m)			
	FAAS		ICP-ES	
As	0,0002	- 0,005	0,00005	- 0,005
Sb	0,0002	- 0,0025	0,0002	- 0,0025
Se	0,0002	- 0,005	0,0002	- 0,005
Sn	0,0005	- 0,005	0,0002	- 0,005
Te	0,00002	- 0,0025	0,00002	- 0,0025

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*.

ENV 13800, *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*.

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

EN ISO 1042, *Laboratory glassware – One-mark volumetric flasks (ISO 1042:1993)*.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*.

3 Principle

3.1 Preparation of the test solution

Dissolution of a test portion in nitric acid.

Separation of analyte by co-precipitation with manganese dioxide.

Dissolution of the precipitate, made up to a defined volume.

Determination of the analyte concentration using one of the two techniques described in 3.2.