

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-ENV 14029:2010 sisaldab Euroopa standardi ENV 14029:2001 ingliskeelset teksti.

Standard on kinnitatud Eesti Standardikeskuse 30.09.2010 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.

Euroopa standardimisorganisatsioonide poolt rahvuslikele liikmetele Euroopa standardi teksti kättesaadavaks tegemise kuupäev on 18.07.2001.

Standard on kättesaadav Eesti standardiorganisatsioonist.

This Estonian standard EVS-ENV 14029:2010 consists of the English text of the European standard ENV 14029:2001.

This standard is ratified with the order of Estonian Centre for Standardisation dated 30.09.2010 and is endorsed with the notification published in the official bulletin of the Estonian national standardisation organisation.

Date of Availability of the European standard text 18.07.2001.

The standard is available from Estonian standardisation organisation.

ICS 77.040.30, 77.120.60

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English version

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

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

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

This European Prestandard (ENV) was approved by CEN on 9 June 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.

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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.

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.

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.

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		
Ag	0,00001	-	0,001	0,00001	-	0,001
Al	0,0001	-	0,005	0,00005	-	0,005
As	0,0002	-	0,005	0,00005	-	0,005
Bi	0,00005	-	0,0025	0,00005	-	0,0025
Ca	0,00005	-	0,001	0,00001	-	0,001
Cd	0,00005	-	0,001	0,00001	-	0,001
Co	0,00005	-	0,001	0,00005	-	0,001
Cr	0,00005	-	0,001	0,00005	-	0,001
Cu	0,00001	-	0,001	0,00001	-	0,001
Fe	0,00005	-	0,001	0,00005	-	0,001
Mg	0,00005	-	0,001	0,00005	-	0,001
Mn	0,00005	-	0,001	0,00005	-	0,001
Na	0,00005	-	0,001	0,00001	-	0,001
Ni	0,00005	-	0,001	0,00001	-	0,001
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
Tl	0,00002	-	0,0025	0,00002	-	0,0025
Zn	0,00001	-	0,001	0,00001	-	0,001

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

ISO 1042:1998, *Laboratory glassware – One-mark volumetric flasks.*

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

3 Principle

3.1 Preparation of the test solution

Dissolution of a test portion in nitric acid.

Separation of lead by crystallisation as lead nitrate.

Reduction 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 (absorbance);
- 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 element to be determined at the relevant wavelength;
- comparison with that of matrix-matched calibration solutions of the same element.