



SLOVENSKI STANDARD

SIST ENV 13800:2002

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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

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

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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

Ta slovenski standard je istoveten z: ENV 13800:2000

ICS:

77.120.60	Svinec, cink, kositer in njihove zlitine	Lead, zinc, tin and their alloys
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EUROPEAN PRESTANDARD
PRÉNORME EUROPÉENNE
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ENV 13800

July 2000

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), 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

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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.

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

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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.

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.

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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.

4 Apparatus

4.1 General

Use ordinary apparatus as available in a chemical laboratory.

All glassware and plastic ware to be used shall be cleaned with diluted nitric acid (5.2.2) and thoroughly rinsed with water.

4.2 Volumetric glassware

4.2.1 One-mark volumetric flasks of capacities 100 ml, 250 ml, 500 ml, and 1 000 ml in accordance with ISO 1042:1983 class A.

4.2.2 One-mark pipettes of capacities 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml and 100 ml in accordance with ISO 648:1977 class A.

4.3 Plastic ware

The following are required :

- Polytetrafluoroethylene (PTFE) beakers of 250 ml capacity ;
- Suitable PTFE covers ;
- One-mark volumetric flasks of 100 ml capacity.

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4.4 Instruments

4.4.1 Flame atomic absorption spectrometer

Flame atomic absorption spectrometer (FAAS) equipped with laminar flow burners suitable for acetylene-air, hydrogen-air or acetylene-nitrous oxide flames, and with radiation sources such as hollow cathode lamps (HCL) or electrode-less discharge lamps (EDL) as appropriate to the element to be determined.

The instrument shall be used in accordance with the manufacturer's instructions and the performance checked (see the ISO documents in preparation, numbers 00023056 and 00023057).

DANGER To avoid any risk to personnel due to emission of acid and lead fumes, the off-gas shall be exhausted externally.

4.4.2 Inductively coupled plasma emission spectrometer

Inductively coupled plasma emission spectrometer (ICP-ES), either a simultaneous instrument with the relevant wavelengths installed or a sequential instrument where a monochromator system allows the selection of wavelengths (see the ISO document in preparation, number 00021288).

The instrument shall be used in accordance with the manufacturer's instructions and the performances checked (see the ISO document in preparation, number 00023058).

DANGER To avoid any risk to personnel due to emission of acid and lead fumes, the off-gas shall be exhausted externally.

5 Reagents

5.1 General

For all stages of analysis, unless otherwise stated, use only reagents of recognised analytical grade, preferably with an actual analysis, and only water of at least grade 2, as specified in ISO 3696.

Prepare all solutions using the same container of each reagent.

5.2 Nitric acid (HNO₃)

5.2.1 Concentrated nitric acid

Nitric acid which $\rho_{20} = 1,41$ g/ml.

5.2.2 Nitric acid 1:1 (v/v)

Add to one volume of water, the same volume of nitric acid (5.2.1) in a suitable container and mix thoroughly. This diluted acid is also used to clean the laboratory ware.

5.2.3 Nitric acid 1:2 (v/v)

Add 200 ml nitric acid (5.2.1) to 400 ml water into a 1 000 ml beaker and mix thoroughly.

5.3 Tartaric acid solution (C₄H₆O₆) 300 g/l

Weigh 150 g of tartaric acid (C₄H₆O₆) into a 500 ml volumetric flask, add about 350 ml of water and shake until dissolution is complete. Make up to the mark with water and mix thoroughly.

5.4 Hydrochloric acid (HCl)

Hydrochloric acid which $\rho_{20} = 1,18$ g/ml.

5.5 Fluoboric acid (HBF₄)

Fluoboric acid which $\rho_{20} = 1,22$ g/ml.

DANGER This reagent has severe risks both by ingestion and particularly by skin contact. Before using this reagent carefully read the safety data sheet to ensure that all the necessary precautions are taken, including the personal protective equipment required.

5.6 Lead for matrix matching

5.6.1 Pure lead

For the determination of most of the element contents detailed in clauses 8 and 9, pure lead (99,99 % m/m) may be used providing that no relevant elements are present in an amount that could be significant to the determination required. If any relevant elements are present, lead of higher purity (99,999 % or more) shall be used.

5.6.2 Lead solution (100g/l)

Weigh 100g of lead (5.6.1) into a 600 ml beaker, add 300ml of water, and then, in several steps to control a possibly vigorous reaction, add 100 ml of nitric acid (5.2.1); cover with a watch-glass and heat gently until dissolution is complete. Boil gently until nitrous fumes are expelled and then, allow to cool.

Transfer into a 1 000 ml volumetric flask (4.2.1), make up to the mark with water and mix thoroughly.

5.7 Standard solutions

5.7.1 Standard solution (1g/l)

5.7.1.1 General

Either use commercially available certified standard solutions or prepare standard solutions as described in 5.7.1.2 to 5.7.1.12.

5.7.1.2 Solutions for silver, bismuth, cadmium, copper, nickel, thallium and zinc

Weigh $(1,000 \pm 0,001)$ g of the relevant metal of 99,9% (m/m) minimum purity and transfer into a 250 ml beaker, add 75 ml of nitric acid (5.2.3), cover with a watch glass, and heat gently until dissolution is complete. Allow to cool, then transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

Store the silver solution in the dark.

5.7.1.3 Solution for aluminium

The aluminium used for this solution shall be in a form of clean wire, foil or pieces cut from an ingot. Do not use aluminium powder because of possible oxidation of its very large specific surface involved.

Weigh $(1,000 \pm 0,001)$ g of aluminium of 99,9 % (m/m) minimum purity and transfer into a 250 ml beaker, add 10 ml hydrochloric acid (5.4), cover with a watch glass, and heat gently until dissolution is complete. Allow to cool, then transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

NOTE If aluminium of high purity is used, it is possible to increase the dissolution rate by adding 1 ml of nickel chloride solution (10g/l). If nickel and aluminium are to be determined together, use 1 ml of mercury (II) chloride solution (10g/l) instead of the nickel chloride solution.

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5.7.1.4 Solution for arsenic

Dry approximately 5 g of arsenic oxide (As_2O_3 , 99,9 % (m/m) minimum purity) in an oven at $(105 \pm 2)^\circ\text{C}$ for two hours and allow to cool completely in a desiccator. Weigh $(1,320 \pm 0,001)$ g of dried arsenic dioxide and transfer into a 250 ml beaker, add 75 ml of nitric acid (5.2.3), cover with a watch glass, and swirl until dissolution is complete. Transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.5 Solution for barium

Dry approximately 5 g of barium carbonate (BaCO_3 , 99,9 % (m/m) minimum purity) in an oven at $(105 \pm 2)^\circ\text{C}$ for two hours and allow to cool completely in a desiccator. Weigh $(1,437 \pm 0,001)$ g of dried barium carbonate and transfer into a 250 ml beaker, add 75 ml of nitric acid (5.2.3), cover with a watch glass, and swirl until dissolution is complete. Transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.6 Solution for calcium

Dry approximately 5 g of calcium carbonate (CaCO_3 , 99,9 % (m/m) minimum purity) in an oven at $(105 \pm 2)^\circ\text{C}$ for two hours and allow to cool completely in a desiccator. Weigh $(2,497 \pm 0,001)$ g of dried calcium carbonate and transfer into a 250 ml beaker, add 25 ml of water, then 75 ml of nitric acid (5.2.3), cover with a watch glass and swirl until dissolution is complete. Transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.7 Solution for sodium

Dry approximately 5 g of sodium chloride (NaCl , 99,9 % (m/m) minimum purity) in an oven at $(105 \pm 2)^\circ\text{C}$ for two hours and allow to cool completely in a desiccator. Weigh $(2,542 \pm 0,001)$ g of dried sodium chloride and transfer into a 250 ml beaker, add 75 ml of nitric acid (5.2.3), cover with a watch glass and swirl until dissolution is

complete. Transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.8 Solution for sulphur

Dry approximately 5 g of sodium sulphate (Na_2SO_4 , 99,9 % (m/m) minimum purity) in an oven at $(105 \pm 2)^\circ\text{C}$ for two hours and allow to cool completely in a desiccator. Weigh $(4,430 \pm 0,001)\text{g}$ into a 250 ml beaker, add 75 ml of nitric acid (5.2.3), cover with a watch glass and swirl until dissolution is complete. Transfer into a 1000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.9 Solution for antimony

Dry approximately 5 g of antimony oxide (Sb_2O_3 , 99,9% (m/m) minimum purity) in an oven at $(105 \pm 2)^\circ\text{C}$ for two hours and allow to cool completely in a desiccator. Weigh $(1,197 \pm 0,001)\text{g}$ into a 250 ml beaker, add 25 ml water and 50 ml of hydrochloric acid (5.4), cover with a watch glass and swirl until dissolution is complete. Transfer into a 1 000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.10 Solution for selenium

Weigh $(1,000 \pm 0,001)\text{g}$ of selenium of 99,9 % (m/m) minimum purity into a 250 ml beaker, add 100 ml of nitric acid (5.2.2), cover with a watch glass, and heat gently to initiate the reaction of dissolution. Cool if the reaction proceeds too vigorously. Allow to cool, then transfer into a 1000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.11 Solution for tin

Weigh $(1,000 \pm 0,001)\text{g}$ of tin of 99,9% (m/m) minimum purity into a 250 ml beaker, add 100 ml of hydrochloric acid (5.4), cover with a watch glass and heat gently until dissolution is complete. Allow to cool, then transfer into a 1000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.1.12 Solution for tellurium

Weigh $(1,000 \pm 0,001)\text{g}$ of tellurium of 99,9 % (m/m) minimum purity into a 250 ml beaker, add 100 ml of nitric acid (5.2.2), cover with a watch glass, and swirl until dissolution is complete. Transfer into a 1000 ml volumetric flask (4.2.1), then make up to the mark with water and mix thoroughly.

5.7.2 Working standard solutions

5.7.2.1 Solutions of silver, arsenic, barium, bismuth, calcium, cadmium, copper, sodium, nickel, sulphur, selenium, tellurium, thallium and zinc (10 mg/l)

Using a 10 ml pipette (4.2.2), transfer 10 ml of the relevant standard solution (5.7.1) into a 1 000 ml volumetric flask (4.2.1). Add 150 ml of nitric acid (5.2.3), then make up to the mark with water and mix thoroughly.

Each solution shall be prepared on the day of use.

5.7.2.2 Solutions of silver, arsenic, barium, bismuth, calcium, cadmium, copper, sodium, nickel, sulphur, selenium, tellurium, thallium and zinc (100 mg/l)

Using a 50 ml pipette (4.2.2), transfer 50 ml of the relevant standard solution (5.7.1) into a 500 ml volumetric flask (4.2.1). Add 75 ml of nitric acid (5.2.3), then make up to the mark with water and mix thoroughly.

5.7.2.3 Solutions of aluminium, antimony and tin (10 mg/l)

Using a 10 ml pipette (4.2.2), transfer 10 ml of the relevant standard solution (5.7.1) into a 1 000 ml volumetric flask (4.2.1). Add 20 ml of tartaric acid solution (5.3), 150 ml of nitric acid (5.2.3), then make up to the mark with water and mix thoroughly.

Each solution shall be prepared on the day of use.