SLOVENSKI STANDARD
SIST ENV 14138:2004

01-januar-2004

Svinec in svinļ eve zlitine ï Analiza s plamensko atomsko absorpcijsko spektrometrijo (FAAS) ali emisijsko spektrometrijo z induktivno sklopljeno plazmo (IC-ES) po loļ enju elementov s soobarjanjem

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

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

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

Ta slovenski standard je istoveten z: ENV 14138:2001

ICS:

77.120.60	Svinec, cink, kositer in njihove zlitine	Lead, zinc, tin and their alloys

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EUROPEAN PRESTANDARD

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English version

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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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Contents

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 1042:1998).*

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.

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 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 calibration solutions of the same element.

4 Apparatus

4.1 General

Use ordinary apparatus as available in a chemical laboratory.

All glassware to be used shall be cleaned with diluted hydrochloric acid (5.3.2) and thoroughly rinsed with water.

4.2 Volumetric glassware

4.2.1 One-mark volumetric flasks of capacities 25 ml, 100 ml, 500 ml and 1 000 ml in accordance with EN ISO 1042 class A.

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

4.3 Filtration system

Vacuum filtration system with a filter membrane of PTFE, or other material inert to nitric acid, of about 5 µm porosity (filtering diameter 20 mm to 50 mm).

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 also ISO/DIS 13204-2 and ISO/DIS 13204-3).

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 also ISO/DIS 12235-1).

The instrument shall be used in accordance with the manufacturer's instructions and the performances checked (see also ISO/DIS 12235-2).

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, suitable for trace analysis and only water of at least grade 2, as specified in EN ISO 3696.

Prepare all solutions using the same container of each reagent.

5.2 Nitric acid (HNO_3)

5.2.1 Concentrated nitric acid

Nitric acid of high purity, which ρ_{20} = 1,41 g/ml.

5.2.2 Nitric acid 1:1 (*V/V*)

Add to one volume of water, in a suitable container, the same volume of concentrated nitric acid (5.2.1) and mix thoroughly.

5.2.3 Nitric acid 1:2 (*V/V*)

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

5.3 Hydrochloric acid (HCl)

5.3.1 Concentrated hydrochloric acid

Hydrochloric acid of high purity, which ρ_{20} = 1,18 g/ml.

5.3.2 Hydrochloric acid 1:1 (*V/V*)

Add 400 ml of concentrated hydrochloric acid (5.3.1) to 400 ml water in a 1 000 ml flask. This acid is used to clean all glassware which should be soaked for at least 1 h prior to use, then rinsed thoroughly with water.

5.4 Ammonia solution

Ammonia solution of high purity which ρ_{20} = 0,88 g/ml.

5.5 Pure lead

For the determination of the recovery rate of the analyte (see 7.2), very pure lead (99,9999 % *m/m*) should be used. However, lead of lower purity may be used provided that the analyte is not present in an amount that could be significant to the determination required.