SLOVENSKI STANDARD
SIST ENV 12908:2000

01-november-2000

Svinec in svinļ eve zlitine - Analiza z optiļ no emisijsko spektrometrijo (OES), inducirano z iskrenjem

Lead and lead alloys - Analysis by Optical Emission Spectrometry (OES) with spark excitation

Blei und Bleilegierungen - Analyse durch Optische Emissionsspektrometrie (OES) mit Funkenanregung

Plomb et alliages de plomb - Analyse par Spectrométrie d'Emission Optique (OES) avec excitation par étincelles

Ta slovenski standard je istoveten z: **ENV 12908:1997**

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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Descriptors: lead, lead alloys, chemical analysis, analysis methods, optical emission spectrometry

English version

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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.

Within its programme of work, CEN/TC 306 requested CEN/TC 306/GT 3 "Lead and lead alloys - Sampling and analysis" to prepare the following prestandard :

ENV 12908 Lead and lead alloys - Analysis by optical emission spectrometry (OES) with spark excitation

WARNING : Any hazards involved in handling and use of lead and lead alloys are detailed in the supplier's health and safety data sheet. All relevant precautions shall be taken to protect personnel.

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 prestandard specifies the method for the analysis of lead and lead alloys by optical emission spectrometry (OES) with spark excitation. The method is applicable to the elements and ranges given in table 1 :

Table 1 : Elements - Ranges of application

Elements	Ranges of application % (m/m)
Ag	0,0002 to 0,3
Al	0,001 to 0,04
As	0,0005 to 0,3
Bi	0,0005 to 0,2
Ca	0,0005 to 0,2
Cd	0,0002 to 0,2
Cu	0,0002 to 0,1
Ni	0,0002 to 0,01
S	0,001 to 0,01
Sb	0,0005 to 15
Se	0,001 to 0,04
Sn	0,0005 to 15
Te	0,0005 to 0,05
Zn	0,0002 to 0,01

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.

prEN 12402	Lead and lead alloys - Methods of sampling for analysis

3 Definitions

For the purposes of this prestandard, the following definitions apply :

3.1 Certified Reference Material (CRM)

Material whose relevant analysis is known to a high degree of accuracy and element contents of which have been certified by numerous chemical analyses performed by several independant laboratories, followed by statistical analysis of all the results obtained.

NOTE : The method of chemical analysis used to analyse a CRM should conform to an International, European or National standard.

3.2 Reference Material (RM)

Material whose relevant analysis has been accurately determined by chemical methods, but which has only been analysed by one or more laboratories and which has not been certified.

NOTE : The method of chemical analysis used to analyse an RM should conform to an International, European or National standard.

3.3 Drift control samples

These samples are a series of homogeneous discs that contain all the elements which have been calibrated, at both near zero concentration and high concentrations near the top of the calibration ranges.

NOTE : No analyses are necessary on these discs. These discs are measured during the calibration. The intensities obtained are stored in the computer.

3.4 Sample

Portion of the lead or lead alloy representative of the chemical composition.

4 Principle

The sample is quantitatively analysed by measuring the intensity of the element specific radiation generated by the spark resulting from the application of a high voltage between the sample, as one electrode, and an inert counter-electrode.

The intensity measured is compared with the element calibration and converted to a percentage.

5 Apparatus

5.1 Optical emission spectrometer

Spectrometer with spark excitation that shall be able to measure the intensity of the optical radiation emitted at specific wavelenghts by the elements present in the material.

The wavelengths ***generally used*** are given in Annex A (informative).

5.2 Machine for sample surface preparation

The lathe or milling machine used for surface preparation shall be able to produce a surface that conforms to the requirements of 7.2.

6 Sampling

Sampling shall be carried out in accordance with prEN 12402

7 Procedure

7.1 Training of personnel

All personnel carrying out the analysis shall be fully trained in the steps detailed below.

7.2 Surface preparation

WARNING : The appropriate safety recommendations for the use of mechanical apparatus should be observed. These operations should be carried only by properly trained personnel wearing appropriate personal protective equipment.

7.2.1 The surface of the sample shall be prepared, by turning or milling, to a surface finish that is sufficiently plane and smooth so as not to affect the analytical results.

Once a method of preparation is chosen, whether turning or milling, the same method shall be used for all samples, reference materials and certified reference materials to avoid variations in the surface finish which may affect the analysis.

To avoid cross contamination between different materials, for example pure lead and lead alloys, all relevant components of the machine shall be thoroughly cleaned before use.

Once the surface has been prepared, it shall be kept clean to avoid any contamination, for example fingerprints.

NOTE : Measurements should be carried out as soon as possible after any surface preparation.

7.2.2 The minimum thickness of metal removed shall be 1 mm to eliminate any contamination.

7.2.3 The turning or milling shall be carried out at a suitable rate that avoids undue heating of the sample as this may cause changes in the sample and lead to variations in the analysis. Lubricant shall not be used.

7.3 Calibration

7.3.1 Method of calibration

The original calibration of the spectrometer is carried out by using a range of reference materials to prepare calibration graphs from which the analysis of unknown samples is obtained. The calibrations are usually stored within a computer integral to the spectrometer.

The calibration shall be in accordance with the spectrometer manufacturer's instruction manual, using the appropriate certified reference materials, if they exist. If no certified reference materials are available, reference materials with an accurate analysis shall be used.

7.3.2 Drift correction

Any drift from the original calibration is corrected periodically by the use of drift control samples in accordance with the spectrometer manufacturer's instruction manual.

7.3.3 Recalibration

To ensure the continuing validity of an element calibration, the zero, top and two others points in the calibration shall be checked periodically using CRMs, and/or the RMs. A drift correction operation shall be carried out immediately prior to these checks to ensure optimum setting of the instrument. If any significant error is found, the calibration (see 7.3.1) shall be repeated.

7.3.4 Range of calibration

The range of calibration for an element shall extend well above the element content of the sample with the highest percentage, unless unavoidable heterogeneity makes the preparation of reference materials for the higher percentage impossible.

7.3.5 Number of sparks on calibration materials

The number of sparks carried out on a reference material for calibration shall not be less than six. The sparks shall be evenly distributed over the prepared surface. All intensity measurements shall be examined ; if any intensity measurement is obviously aberrant, further sparks shall be carried out to obtain the minimum six acceptable measurements. The average of the six acceptable measurements is used for calibration.

7.4 Analysis

7.4.1 Analysis of samples

The method used shall be in accordance with the spectrometer manufacturer's instruction manual.