



SLOVENSKI STANDARD
SIST EN 12402:2000
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Lead and lead alloys - Methods of sampling for analysis

Blei und Bleilegerungen - Probenahme für die Analyse

Plomb et alliages de plomb - Méthodes d'échantillonnage pour analyse

Ta slovenski standard je istoveten z: **EN 12402:1999**

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ICS:

77.120.60	Svinec, cink, kositer in njihove zlitine	Lead, zinc, tin and their alloys
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 12402

August 1999

ICS 71.040.40; 77.120.60

English version

Lead and lead alloys - Methods of sampling for analysis

Plomb et alliages de plomb - Méthodes d'échantillonnage
pour analyse

Blei und Bleilegerungen - Probenahme für die Analyse

This European Standard was approved by CEN on 2 July 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 306 "Lead and lead alloys", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2000, and conflicting national standards shall be withdrawn at the latest by February 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European Standard specifies the methods of sampling of lead and lead alloys for the purpose of chemical analysis or for the purpose of spectrometric analysis by Optical Emission Spectrometry (OES), with spark excitation.

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.

ENV 12908 1997 Lead and lead alloys - Analysis by Optical Emission Spectrometry (OES), with spark excitation.

3 Definitions

For the purposes of this European Standard, the following definitions apply :

3.1

cast

product of one furnace or crucible melt.

NOTE All the ingots from a single cast have the same identifying mark.

3.2

ingot

cast product intended for remelting and/or processing

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3.3

jumbo or block

very large ingot with a mass generally much more than 50 kg.

3.4

bundle

collection of ingots taken from a single cast and secured if necessary, for the purposes of handling, shipment and storage.

3.5

sample

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

3.6

laboratory sample

final form of the sample submitted for analysis.

4 Principle

Since lead and lead alloys can have different shapes and masses (ingots, jumbos) which are not easily handled, sampling from the molten metal during the casting process is recommended. Nevertheless, sampling from a consignment shall be feasible, if required, in any case of dispute.

5 Sampling of molten lead and lead alloys

5.1 Conditions for sampling

All samples representing a cast shall be taken from the molten metal under the appropriate conditions of temperature, mixing and cleanliness to ensure that the molten metal is homogeneous. These conditions will depend on the characteristics of the metal to be sampled, but are particularly important for lead alloys, many of which are subject to segregation if conditions are incorrect.

5.2 Apparatus

All apparatus used shall be clean and be inert to the molten metal.

5.2.1 Ladle

The ladle shall be large enough to allow the sample mould to be filled in one operation.

5.2.2 Sample mould

The sample mould shall be designed to cool the molten metal as rapidly as possible to avoid segregation. An example is proposed in annex A (informative).

Samples shall be of suitable dimensions to be analysed on any usual spectrometer (OES). The minimum thickness of the disc shall be 5 mm. The maximum thickness and the diameter depend on the dimensions of the sample platform of the spectrometer, but shall not be so large that segregation can occur within the sample due to an extended cooling time.

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5.3 Procedure

5.3.1 Collection of samples

Preheat the ladle by holding it in the molten metal of an ingot mould during filling. When heated, fill the ladle from the molten metal and, without delay, fill the sample mould in one pouring operation. When the metal has completely solidified, remove the sample from the mould, cool and mark with its sample identity. If it is necessary to pour samples in quick succession, several sample moulds shall be used to avoid the possibility of a slower rate of cooling allowing segregation to occur.

WARNING : Due to the inherent risks associated with high temperatures, molten metal and moving machinery, only properly trained personnel wearing appropriate personal protective equipment may carry out the sampling.

NOTE For process control, the following method should be used for sampling of metal in the kettle :

- preheat the ladle by holding it in the molten metal ;
- thoroughly mix the lead bath using an appropriate mixer ;
- fill the preheated ladle while the lead remains well stirred, and without delay ;
- fill the sample mould in one processing operation ;

Continue the operations as described above.

5.3.2 Frequency of sampling

5.3.2.1 Lead

Samples shall be taken at the beginning and end of a cast as a minimum.

NOTE Additional samples can be agreed between the supplier and the purchaser.

5.3.2.2 Lead alloys

Samples shall be taken at the beginning, middle and end of a cast as a minimum.

NOTE Additional samples can be agreed between the supplier and the purchaser.

5.3.3 Identification

Each sample shall be permanently identified with a unique identity and, preferably, the cast identity.

5.3.4 Sample preparation

5.3.4.1 Samples for spectrometric analysis

The final surface of the sample disc shall be prepared immediately prior to the analysis. Surface preparation is detailed in ENV 12908:1997.

5.3.4.2 Samples for chemical analysis

Half of each sample shall be prepared by milling or filing through its depth. All the millings or filings from each separate sample shall be collected. Any iron particles introduced during the milling or filing operations shall be removed using a strong magnet. Thoroughly mix the sample to homogenise it.

All the apparatus used shall be thoroughly cleaned prior to use. The speed of milling or filing shall be chosen in order to avoid heating and oxidizing, without the use of a lubricant

For those lead alloy samples which are subject to segregation according to particle size, the millings or filings shall be screened through a sieve with an aperture size of 0,16 mm to provide two fractions, ensuring that all the sample is retained.

Each fraction shall be weighed to determine the proportion in percent.

If any doubt exists concerning segregation of a lead alloy sample, the millings or filings shall be sieved.

NOTE 1 Millings or filings should be not greater than 0,5 mm for their largest dimension.

NOTE 2 The sample discs used for optical emission spectrometric analysis (OES) can be used as samples if a subsequent chemical analysis is required. In this case, turnings obtained from a newly prepared surface (without any traces of sparks) can be used instead of millings or filings.

5.3.5 Laboratory sample

For an unsieved sample, place the millings or filings into a suitable container, close and label ; it constitutes the laboratory sample.

For a sieved sample, place each of the two fractions into a suitable container, close and label the container specifying the size fraction and its percentage. Place the two containers into a bag, seal if required and label the bag specifying the contents. The contents of the bag constitute the laboratory sample.

6 Sampling of lead and lead alloy ingot consignment for chemical analysis

This procedure shall compulsorily be used in any case of dispute, and due to the nature of the sample produced, the analysis shall be carried out by chemical methods.

NOTE Because of difficulties of sampling large ingots (jumbos), the sampling procedure should be agreed between the supplier and the purchaser.

6.1 Selection of ingots

6.1.1 If the consignment consists of bundles from more than one cast, each cast shall be sampled separately.

6.1.2 From each cast delivered that is to be sampled, take at random from different bundles, a number of ingots, as given in table 1.

Table 1 - Number of ingots for sampling

Number of ingots	Cast delivered	
	Lead	Lead alloys
10	up to 100 t	up to 50 t
20	over 100 t	over 50 t

NOTE When the consignment consists of less than 10 ingots, the sampling procedure should be agreed between the supplier and the purchaser. If sampling according to this standard is required, all the ingots should be individually sampled according to clause 7.

6.1.3 Carefully clean the surface of each ingot selected to remove dirt, oil, grease and any other contaminants.

6.2 Sample preparation

For lead, obtain the sample by drilling or by sawing, in accordance with the procedures given in 6.2.2 or 6.2.3.

NOTE The use of sawing is recommended. [SIST EN 12402:2000](https://standards.itech.ai/catalog/standards/sist/8f64ca91-aedb-46f0-9369-4e1d26e69402/sist-en-12402-2000)
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For lead alloys, only sawing shall be used due the possibility of segregation.

WARNING : Observe the appropriate safety recommendations for the use of mechanical equipment. Only properly trained personnel wearing appropriate personal protective equipment may carry out these operations.

6.2.1 Arrangement of ingots

Arrange the selected ingots flat, side by side, in groups of ten ingots ; ensure that the manufacturer's identify marks (usually cast into the base of the ingots) are arranged in the same way for each one of the ingots :

- for drilling (6.2.2), turn alternate ingots upside down as indicated in figure 1 a) ;
- for sawing (6.2.3), arrange the ingots as indicated in figure 1 b).

In each group, draw a diagonal across the rectangle thus formed [see figure 1a) or 1 b)].

6.2.2 Drilling

Using a clean drill of appropriate dimensions, drill each ingot to half of its depth at the points indicated in figure 1 a).

NOTE The use of a high speed steel drill of about 15 mm diameter is recommended.

Carry out the drilling without undue heating which may cause oxidation, at a suitable drilling rate to obtain drillings of a thickness between 0,2 mm and 0,5 mm, without the use of lubricant.