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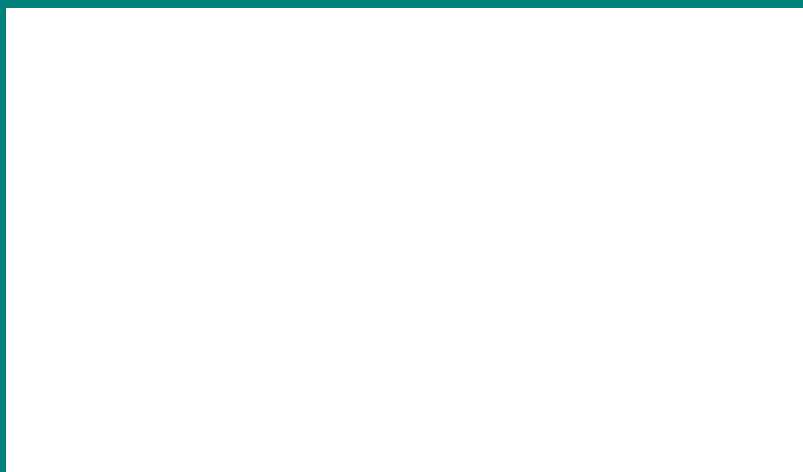
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Koppar och kopparlegeringar – Bestämning av blyhalt – Flamatomär absorptionspektrometrimetod (FAAS)

Copper and copper alloys – Determination of lead content – Flame atomic absorption spectrometric method (FAAS)



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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15622

March 2010

ICS 77.120.30

English Version

**Copper and copper alloys - Determination of lead content -
Flame atomic absorption spectrometric method (FAAS)**

Cuivre et alliages de cuivre - Dosage du plomb - Méthode
par spectrométrie d'absorption atomique dans la flamme
(SAAF)

Kupfer und Kupferlegierungen - Bestimmung des
Bleigehaltes - Flammenatomabsorptionsspektrometrisches
Verfahren (FAAS)

This European Standard was approved by CEN on 23 January 2010.

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 CEN Management Centre 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 CEN Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 15622:2010) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 10 "Methods of analysis" to prepare the following European Standard:

EN 15622, *Copper and copper alloys — Determination of lead content — Flame atomic absorption spectrometric method (FAAS)*.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This document specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the lead in copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having lead mass fractions between 0,01 % and 5,0 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1811-1, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 1: Sampling of cast unwrought products*

ISO 1811-2, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2: Sampling of wrought products and castings*

3 Principle

Dissolution of a test portion in fluoroboric-nitric acid mixture followed, after suitable dilution, by aspiration of the test solution into an air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 217,0 nm line emitted by a lead hollow-cathode lamp.

NOTE During the validation exercise of this standard method some laboratories have also carried out the measurements at 283 nm line. These measurements showed a satisfactory trueness. However, the number of results produced was too few and, therefore, insufficient for the estimation of the corresponding precision data.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Nitric acid, HNO_3 ($\rho = 1,40$ g/ml).

4.2 Nitric acid solution (1 + 1).

Add 100 ml of nitric acid (4.1) to 100 ml of water.

4.3 Hydrofluoric acid, HF, 40 % solution ($\rho = 1,13$ g/ml).

WARNING — Hydrofluoric acid is a hazardous substance. Care shall be taken and it shall be used under an efficient fume hood.

4.4 Boric acid, H_3BO_3 , 40 g/l solution.

4.5 Fluoroboric-nitric acid mixture.

Add 300 ml of boric acid (4.4), 30 ml of hydrofluoric acid (4.3) and 500 ml of nitric acid (4.1) to 150 ml of water and mix well.

4.6 Lead stock solution, 1,0 g/l Pb.

Weigh $(1 \pm 0,001)$ g of lead ($Pb \geq 99,9\%$) and transfer it into a 250 ml beaker. Add 20 ml of nitric acid solution (4.2), cover with a watch glass and heat gently until the lead is completely dissolved and then bring to the boiling point until the nitrous fumes have been expelled. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, add 100 ml of water and 9 ml of nitric acid solution (4.2). Dilute to the mark with water and mix well.

1 ml of this solution contains 0,001 g of Pb.

4.7 Lead standard solution, 0,1 g/l Pb.

Transfer 25,0 ml of the lead stock solution (4.6) into a 250 ml one-mark volumetric flask, add 100 ml of water and 9 ml of nitric acid solution (4.2). Dilute to the mark with water and mix well.

1 ml of this solution contains 0,1 mg of Pb.

4.8 Copper base solution, 20 g/l Cu.

Weigh $(10 \pm 0,01)$ g of pure, lead-free copper ($Pb < 0,0002\%$) and transfer it into a 600 ml polytetrafluorethylene, polyethylene or low-pressure polypropylene beaker. Add 400 ml of the fluoroboric-nitric acid mixture (4.5) and cover with a watch glass. Heat until the copper is completely dissolved, then boil until the nitrous fumes have been expelled. Allow to cool and transfer the solution into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

50 ml of the solution contain 1,0 g of copper and 40 ml of the fluoroboric-nitric acid mixture.

5 Apparatus

5.1 **Beakers**, polytetrafluorethylene, polyethylene or polypropylene.

5.2 **One-mark volumetric flasks**, polytetrafluorethylene, polyethylene or polypropylene.

5.3 **Atomic absorption spectrometer**, fitted with an air/acetylene burner.

5.4 **Lead hollow-cathode lamp**.

6 Sampling

Sampling shall be carried out in accordance with ISO 1811-1 or ISO 1811-2, as appropriate.

Test samples shall be in the form of fine drillings, chips or millings with a maximum thickness of 0,5 mm.

7 Procedure

7.1 Preparation of the test portion solution

7.1.1 Test portion

Weigh $(1 \pm 0,001)$ g of the test sample.

7.1.2 Test portion solution

Transfer the test portion (7.1.1) into a 250 ml polytetrafluorethylene, polyethylene or polypropylene beaker (5.1). Add 40 ml of the fluoroboric-nitric acid mixture (4.5). Cover with a watch glass and heat gently

until the test portion is completely dissolved, then heat at a temperature of approximately 90 °C until brown fumes have been expelled. If polyethylene or polypropylene beakers are used, heating shall be carried out in water bath. Wash the cover and the sides of the beaker with water and allow to cool. Transfer the dissolved test portion quantitatively into a 100 ml one-mark volumetric flask, dilute the solution to the mark with water and mix well.

7.1.3 Lead mass fractions between 0,01 % and 0,15 %

Proceed as indicated in 7.5.

7.1.4 Lead mass fractions between 0,1 % and 0,75 %

Take a 20 ml aliquot of the test portion solution (7.1.2) and introduce into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Proceed as indicated in 7.5.

NOTE The validation exercise of this standard method showed that for lead mass fractions between 0,5 % and 0,75 % results are better when the determination is carried out following 7.1.4.

7.1.5 Lead mass fractions between 0,5 % and 5,0 %

Take a 20 ml aliquot of the test portion solution (7.1.2) and introduce into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well. Proceed as indicated in 7.5.

7.2 Blank test

Carry out a blank test simultaneously with the determination, following the same procedure and using the same quantities of all reagents, as used for the determination, but omitting the test portion.

7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of a reference material or a synthetic sample containing a known amount of lead and of composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

7.4 Establishment of the calibration curve

7.4.1 Preparation of the calibration solutions

7.4.1.1 General

In all cases, copper, salt-concentrations and acidity in the calibration solutions shall be similar to those of the test portion solutions.

The presence of copper in the calibration solutions compensates for chemical interaction effects of copper in the test solution. Normally no similar additions are required to compensate for the effect of alloying elements. If an alloying element is present in the material to be analysed in mass fraction > 10 %, an appropriate mass of this element shall be added to the calibration solutions. The volumes of copper base solution added (4.8) have been calculated to compensate for chemical interaction effects of copper in test solutions of copper or high-copper alloys. Overcompensation may occur if the same volumes are added when the test samples are copper-based alloys where the percentage of copper is lower. In these cases, the volumes of copper base solution shall be decreased to match the copper content of the test sample in solution.

The lead concentration of the calibration solutions shall be adjusted to suit the sensitivity of the spectrometer used, so that the curve of absorbance as a function of concentration is a straight line.

7.4.1.2 Lead mass fractions between 0,01 % and 0,15 %

Into each of a series of six 100 ml one-mark volumetric flasks, introduce the volumes of lead standard solution (4.7) and of copper base solution (4.8) shown in Table 1. Dilute to the mark with water and mix well.

Table 1 — Calibration for lead mass fractions between 0,01 % and 0,15 %

Lead standard solution volume (4.7)	Corresponding lead mass	Corresponding lead concentration after final dilution	Copper base solution volume (4.8)	Corresponding copper mass	Corresponding lead mass fraction of sample
ml	mg	mg/ml	ml	g	%
0 ^a	0	0	50	1,000	0
1	0,1	0,001	50	1,000	0,01
2	0,2	0,002	50	1,000	0,02
5	0,5	0,005	50	1,000	0,05
10	1,0	0,010	50	1,000	0,10
15	1,5	0,015	50	1,000	0,15

^a Blank test on reagents for calibration curve.

7.4.1.3 Lead mass fractions between 0,1 % and 0,75 %

Into each of a series of five 100 ml one-mark volumetric flask, introduce the volumes of lead standard solution (4.7) and of copper base solution (4.8) as shown in Table 2. Dilute to the mark with water and mix well.

Table 2 — Calibration for lead mass fractions between 0,1 % and 0,75 %

Lead standard solution volume (4.7)	Corresponding lead mass	Corresponding lead concentration after final dilution	Copper base solution volume (4.8)	Corresponding copper mass	Corresponding lead mass fraction of sample
ml	mg	mg/ml	ml	g	%
0 ^a	0	0	10	0,200	0
2	0,2	0,002	10	0,200	0,10
5	0,5	0,005	10	0,200	0,25
10	1,0	0,010	10	0,200	0,50
15	1,5	0,015	10	0,200	0,75

^a Blank test on reagents for calibration curve.

7.4.1.4 Lead mass fractions between 0,5 % and 5,0 %

Into each of a series of six 100 ml one-mark volumetric flask, introduce the volumes of lead standard solution (4.7) and of copper base solution (4.8) as shown in Table 3. Dilute to the mark with water and mix well.