SLOVENSKI STANDARD
SIST EN 13615:2004

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Metode za analizo kositrovih ingotov ï Doloļ evanje neļ istoļ v kositru s ļ istostjo 99,90 % in 99,85 % z atomsko spektrometrijo

Methods for the analysis of ingot tin - Determination of impurity element contents in tin grades 99,90 % and 99,85 % by atomic spectrometry

Verfahren für die Analyse von Zinn in Masseln - Bestimmung des Gehaltes an Verunreinigungselementen in Zinn der Reinheitsgrade 99,90 % und 99,85 % durch Atomspektrometrie

Méthodes d'analyse des lingots d'étain - Détermination des teneurs en impuretés dans l'étain de qualité 99,90 % et 99,85 % par spectrométrie atomique

Ta slovenski standard je istoveten z: **EN 13615:2001**

ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.150.60	Svinļ eni, cinkovi in kositrovi izdelki	Lead, zinc and tin products

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This European Standard was approved by CEN on 5 October 2001.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 220 “Tin and tin alloys” the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by June 2002, and conflicting national standards shall be withdrawn at the latest by June 2002.

The annexes A and B are normative.

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.

1 Scope

This European Standard specifies atomic spectroscopic methods (Atomic Absorption Spectrometry (AAS) or inductively coupled plasma Atomic Emission Spectrometry (ICP-AES)) intended for the analysis of ingot tin. It is written for use by experienced analysts familiar with atomic spectrometric techniques.

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 (including amendments).

EN 610, *Tin and tin alloys — Ingot tin.*

3 Principle

The test sample is dissolved in hydrochloric acid plus nitric acid and tartaric or citric acid, and the concentration of the element sought is measured using atomic absorption spectrometry or inductively coupled plasma atomic emission spectrometry. Interference is minimised by matching sample and reference materials and by the choice of instrument parameters.

4 Reagents

4.1 General

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade and only distilled water or water of equivalent purity.

4.2 *Hydrochloric acid*, concentrated ($\rho \approx 1{,}18$ g/ml).

4.3 *Dilute hydrochloric acid (1 + 1)*. Dilute 100 ml hydrochloric acid (**4.2**) with 100 ml water.

WARNING The acid should added to the water for safety reasons.

4.4 *Dilute hydrochloric acid (1 + 19).* Dilute 10 ml hydrochloric acid (**4.2**) with 190 ml water.

4.5 *Nitric acid*, ($\rho \approx 1{,}42$ g/ml).

4.6 *Sulphuric acid*, ($\rho \approx 1{,}84$ g/ml).

4.7 *Tartaric acid or citric acid*.

4.8 *Acid mixture*. Add 250 ml hydrochloric acid (**4.2**) to 250 ml water. Cool. Add 250 ml nitric acid (**4.5**) and 50 g tartaric (or citric) acid (**4.7**). Dilute to 1 l with water.

4.9 *Dilute nitric acid (1 + 1)*. Add 100 ml nitric acid (**4.5**) to 100 ml water.

4.10 *Dilute nitric acid (1 + 4)*. Add 50 ml nitric acid (**4.5**) to 200 ml water.

4.11 *Dilute nitric acid (1 + 9)*. Add 25 ml nitric acid (**4.5**) to 225 ml water.

4.12 *Dilute nitric acid (1 %)*. Dilute 5 ml nitric acid (**4.5**) up to 500 ml.

4.13 *Standard solutions of metals*. Freshly purchased standard metal solutions may be used or standard metal solutions should be made up as follows (**4.13.1** to **4.13.25**).

4.13.1 *Standard antimony solution (1 ml contains 1 mg Sb)*. Place 0,10 g Sb in 5 ml sulphuric acid (**4.6**) and heat to complete dissolution. Cool. Carefully add approximately 10 ml water and cool again. Transfer to a 100 ml volumetric flask with dilute hydrochloric acid (1 + 1) (**4.3**).

4.13.2 *Standard antimony solution (1 ml contains 0,2 mg Sb)*. Transfer 10,00 ml of Sb solution **4.13.1** to a 50 ml volumetric flask and make up to volume with dilute hydrochloric acid (1 + 1) (**4.3**).

4.13.3 *Standard copper solution (1 ml contains 1 mg Cu)*. Dissolve 0,10 g Cu in 10 ml dilute nitric acid (1 + 4) (**4.9**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.4 *Standard copper solution (1 ml contains 0,2 mg Cu)*. Transfer 10,00 ml of Cu solution **4.13.3** to a 50 ml volumetric flask and make up to volume with water and mix.

4.13.5 *Standard lead solution (1 ml contains 1 mg Pb)*. Dissolve 0,10 g Pb in 10 ml dilute nitric acid (1 + 4) (**4.10**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.6 *Standard lead solution (1 ml contains 0,1 mg Pb)*. Transfer 5,00 ml of Pb solution **4.13.5** to a 50 ml volumetric flask and make up to volume with water and mix.

4.13.7 *Standard bismuth solution (1 ml contains 1 mg Bi)*. Dissolve 0,10 g Bi in 10 ml dilute nitric acid (1 + 1) (**4.9**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.8 *Standard bismuth solution (1 ml contains 0,1 mg Bi).* Transfer 5,00 ml of bismuth solution **4.13.7** to a 50 ml volumetric flask and make up to volume with dilute hydrochloric acid (1 + 1) (**4.3**).

4.13.9 *Standard cadmium solution (1 ml contains 1 mg Cd).* Dissolve 0,10 g Cd in 10 ml dilute nitric acid (1 + 4) (**4.10**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.10 *Standard cadmium solution (1 ml contains 0,01 mg Cd).* Transfer 1,00 ml of cadmium solution **4.13.9** to a 100 ml volumetric flask and make up to volume with water and mix.

4.13.11 *Standard zinc solution (1 ml contains 1 mg Zn).* Dissolve 0,10 g Zn in 10 ml nitric acid (1 + 4) (**4.10**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.12 *Standard zinc solution (1 ml contains 0,01 mg Zn).* Transfer 1,00 ml of zinc solution **4.13.11** to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.13 *Standard iron solution (1 ml contains 1 mg Fe).* Dissolve 0,10 g Fe wire (free from rust) in 10 ml of nitric acid (1 + 4) (**4.10**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.14 *Standard iron solution (1 ml contains 0,02 mg Fe).* Transfer 1,00 ml of iron solution **4.13.13** to a 50 ml volumetric flask. Make up to volume with water and mix.

4.13.15 *Standard arsenic solution (1 ml contains 1 mg As).* Dissolve 0,1 320 g of As_2O_3 in a little sodium hydroxide solution (1 mol/l) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.16 *Standard arsenic solution (1 ml contains 0,1 mg As).* Transfer 5,00 ml arsenic solution **4.13.15** to a 50 ml volumetric flask. Make up to volume with water and mix.

4.13.17 *Standard aluminium solution (1 ml contains 1 mg Al).* Dissolve 0,100 g Al metal in 10 ml nitric acid (**4.10**) and transfer to a 100 ml volumetric flask. Make up to volume with water and mix.

4.13.18 *Standard aluminium solution (1 ml contains 0,05 mg Al).* Transfer 5,00 ml of aluminium solution **4.13.17** to a 100 ml volumetric flask and make up to volume with dilute nitric acid (1 %) (**4.12**).

4.13.19 *Standard aluminium solution (1 ml contains 0,005 mg Al).* Transfer 5,00 ml of aluminium solution **4.13.18** to a 50 ml volumetric flask and make up to volume with dilute nitric acid (1 %) (**4.12**).

4.13.20 *Standard silver solution (1 ml contains 1 mg Ag).* Dissolve 0,787 g silver nitrate in 50 ml water. Transfer to a 500 ml volumetric flask and make up to volume with nitric acid (1 %) (**4.12**).

4.13.21 *Standard silver solution (1 ml contains 0,1 mg Ag).* Transfer 5,00 ml of silver solution (**4.13.20**) to a 50 ml volumetric flask and make up to volume with water and mix.

4.13.22 *Standard nickel solution (1 ml contains 1,0 mg Ni).* Dissolve 0,100 g of nickel in 10 ml of nitric acid (1 + 4) (4.10) and transfer to a 100 ml volumetric flask and make up to volume with water and mix.

4.13.23 *Standard nickel solution (1 ml contains 0,1 mg Ni).* Transfer 5,00 ml of nickel solution **4.13.22** to a 50 ml volumetric flask and make up to volume with water and mix.

4.13.24 *Standard indium solution (1 ml contains 1,0 mg In).* Dissolve 0,100 g of indium in 10 ml of nitric acid (1 + 4) (**4.10**) and transfer to a 100 ml volumetric flask and make up to volume with water and mix.

4.13.25 *Standard indium solution (1 ml contains 0,1 mg In).* Transfer 5,00 ml of indium solution **4.13.24** to a 50 ml volumetric flask and make up to volume with dilute nitric acid (1 %) (**4.12**).

4.14 *Tin*, min. purity 99,99 %.

5 Apparatus

5.1 *Ordinary laboratory apparatus*. Use grade A glassware.

5.2 *Burette*, of capacity 5 ml, graduated in 0,02 ml.

5.3 *Atomic absorption spectrometer or inductively coupled plasma atomic emission spectrometer*, conforming to the performance requirements in normative annexes A and B.

5.4 *Hollow cathode lamps or electrodeless discharge tubes for antimony, bismuth, cadmium, copper, lead, silver, iron, zinc, arsenic, nickel, aluminium, indium, gold and cobalt*.

NOTE The presence of other elements may also need to be ascertained.

6 Sampling

The sample for analysis shall be obtained as described in EN 610.

7 Procedure

7.1 Preparation of the solution of the sample under test

7.1.1 Weigh 1,0 g ± 0,1 g of the sample and transfer to a 250 ml beaker. Add 20 ml of the acid mixture (**4.8**), heat to complete dissolution and cool. Transfer to a 100 ml volumetric flask and make up to the volume with dilute hydrochloric acid (1 + 19) (**4.4**) and mix.

7.1.2 Prepare a blank test solution following the procedure described in **7.1.1**, but using 1,0 g of high purity tin (**4.14**) instead of the sample.

7.2 Preparation of calibration solutions

For the determination of the impurities, weigh 1,0 g ± 0,1 g high purity tin (**4.14**) into each of 7 250 ml beakers. Add 20 ml of the acid mixture (**4.8**) and warm to dissolve. Cool and transfer to a 100 ml volumetric flask and add the amounts of the standard metal solutions shown in Table 1.

Finally make up to the mark with dilute hydrochloric acid (**4.4**).

Table 1 — Volume of standard metal solutions used in the preparation of the calibration solutions

Flask No.	Standard metal solution ml					
	Sb 4.13.2	**Cu** 4.13.4	**Pb** 4.13.6	**Bi** 4.13.8	**Cd** 4.13.10	**Zn** 4.13.12
1	0,0	0,0	0,0	0,0	0,0	0,0
2	0,5	0,5	0,5	0,5	0,5	0,5
3	1,0	1,0	1,0	1,0	1,0	1,0
4	2,0	2,0	2,0	2,0	2,0	2,0
5	3,0	3,0	3,0	4,0	3,0	3,0
6	4,0	4,0	4,0	6,0	4,0	4,0
7	5,0	5,0	5,0	8,0	5,0	5,0

Flask No.	Standard metal solution ml					
	Fe 4.13.14	**As** 4.13.16	**Al** 4.13.18	**Ag** 4.13.21	**Ni** 4.13.23	**In** 4.13.25
1	0,0	0,0	0,0	0,0	0,0	0,0
2	1,0	1,0	1,0	1,0	1,0	1,0
3	2,0	2,0	2,0	2,0	2,0	2,0
4	4,0	3,0	3,0	3,0	3,0	3,0
5	6,0	4,0	4,0	4,0	4,0	4,0
6	8,0	5,0	5,0	5,0	5,0	5,0
7	10,0	6,0	6,0	6,0	6,0	6,0

For the determination of antimony in all samples and for copper in sample numbers 1,2,3 and 6 transfer 10,0 ml of the solutions to a 100 ml flask and dilute to the mark with dilute hydrochloric acid (**4.4**).

7.3 Certified reference materials (CRM)

Where CRM of similar matrix to the sample under test is available, treat an appropriate sample of the CRM in exactly the same way as the sample under test (**7.1**).

7.4 Spectrometric measurements

Set up the spectrometer (AAS, ICP-AES) using the wavelengths given in **A.3.5** or **B.3.3**, as appropriate. A minimum of two runs of the sample under analysis shall be made; first the calibration solutions, then the samples under analysis, and the cycle repeated without altering the instrument parameters. For the purposes of calculating the element content in the sample, the average of readings from a minimum of two separate runs shall be used.

8 Expression of results

8.1 Determining the metal content

By means of the calibration curves, determine the quantities of each metal corresponding to the spectrometer measurements of the test and the blank test solutions.

NOTE Calibration curves are usually prepared automatically by the instruments.