SLOVENSKI STANDARD
SIST EN 12441-8:2005

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Cink in cinkove zlitine ï Kemiļ na analiza ï 8. del: Doloļ evanje kositra v sekundarnem cinku ï Metoda plamenske atomske absorpcijske spektrometrije

Zinc and zinc alloys - Chemical analysis - Part 8: Determination of tin in secondary zinc - Flame atomic absorption spectrometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 8: Bestimmung von Zinn in Sekundärzink - FAAS-Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 8: Dosage de l'étain dans le zinc - Méthode par spectrométrie d'absorption atomique dans la flamme

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77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.120.60	Svinec, cink, kositer in njihove zlitine	Lead, zinc, tin and their alloys

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Contents

Foreword

This document (EN 12441-8:2004) has been prepared by Technical Committee CEN/TC 209 “Zinc and zinc 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of an identical text or by endorsement, at the latest by June 2005, and conflicting national standards shall be withdrawn at the latest by June 2005.

Within its programme of work, Technical Committee CEN/TC 209 entrusted CEN/TC 209/WG 6 "*Methods of analysis and testing*" to prepare the following document:

EN 12441-8, *Zinc and zinc alloys – Chemical analysis – Part 8: Determination of tin in secondary zinc – Flame atomic absorption spectrometric method*

This document is a part of a series of eleven standards. The other documents are:

— *Part 1: Determination of aluminium in zinc alloys – Titrimetric method*

— *Part 2: Determination of magnesium in zinc alloys – Flame atomic absorption spectrometric method*

— *Part 3: Determination of lead, cadmium and copper – Flame atomic absorption spectrometric method*

— *Part 4: Determination of iron in zinc alloys – Spectrophotometric method*

— *Part 5: Determination of iron in primary zinc – Spectrophotometric method*

— *Part 6: Determination of aluminium and iron – Flame atomic absorption spectrometric method*

— *Part 7: Determination of tin – Flame atomic absorption spectrometric method after extraction*

— *Part 9: Determination of nickel in zinc alloys – Flame atomic absorption spectrometric method*

— *Part 10: Determination of chromium and titanium in zinc alloys – Spectrophotometric method*

— *Part 11: Determination of silicon in zinc alloys – Spectrophotometric method*

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1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of tin in secondary zinc. It is applicable to the products specified in EN 13283.

It is suitable for the determination of tin contents (mass fractions) between 0,1 % and 1,0 %.

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.

EN 12060:1997, *Zinc and zinc alloys – Method of sampling – Specifications*

EN 13283, *Zinc and zinc alloys – Secondary zinc*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12060:1997 and the following apply.

3.1
flame atomic absorption spectrometry
measurement of the absorption of an electromagnetic radiation, emitted by an element at a determined wavelength, by an absorbent medium (flame) formed by atoms of the same element that are in the ground state. Each element absorbs radiation of specific wavelengths and the intensity of the absorbed radiation is proportional to the concentration of said element

4 Principle

A sample of the metal is dissolved in a mixture of hydrochloric acid and hydrogen peroxide and, after adequate dilution and atomisation of the solution in a nitrous oxide-acetylene flame, the content of tin is determined by atomic absorption spectrometry at the wavelength of 235,5 nm.

5 Reagents

5.1 General

During the test, use only reagents of known or analytical grade and distilled or demineralised water.

5.2 Hydrochloric acid, ρ = 1,19 g/ml.

5.3 Nitric acid, ρ = 1,4 g/ml.

5.4 **Hydrogen peroxide**, 30 % (mass fraction)

Hydrogen peroxide solutions are liable to be stabilised with products containing tin. Therefore exactly the same volume of hydrogen peroxide shall be used both when dissolving samples and when preparing the calibration solutions.

5.5 **Pure zinc 99,995 %**, tin free

5.6 **Zinc**, 100 g/l solution

Dissolve 100 g of zinc (5.5) with 600 ml of hydrochloric acid (5.2). After dissolution, transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

5.7 **Tin**, 0,5 g/l standard solution

Weigh 0,5 g of tin [with a purity of no less than 99,9 % (mass fraction)] to the nearest 0,001 g. Carefully add 200 ml of hydrochloric acid (5.2). When dissolution is complete, cool to room temperature and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 0,500 mg of tin.

5.8 Aqua regia

Mix 3 part volumes of hydrochloric acid (5.2) with 1 part volume nitric acid (5.3).

6 Apparatus

6.1 General

All glassware used for the preparation of the solutions and for the implementation of the method shall be cleaned with boiling aqua regia (5.8) prior to use.

6.2 Specific equipment

In addition to standard laboratory apparatus an atomic absorption spectrometer, equipped with a premix burner, with facilities for using the oxidizer-fuel combination of nitrous oxide-acetylene, shall be used.

NOTE Excitation sources should be operated in accordance with the manufacturer's recommendations. The optical path length within the flame should be between 5 cm to 10 cm.

7 Sampling

The test sample shall be selected and prepared in accordance with the procedure given in EN 12060.

8 Procedure

8.1 Test portion

Weigh 5 g of the test sample to the nearest 0,001 g.

8.2 Preparation of the test portion

8.2.1 Introduce the test portion (8.1) into a 250 ml beaker fitted with a watch-glass and dissolve by carefully adding 40 ml of hydrochloric acid (5.2). Oxidize and complete the dissolution by adding 100 µl of hydrogen peroxide (5.4). Do not heat in order to avoid the loss of $SnCl_4$.

8.2.2 After dissolution, cool to room temperature and transfer quantitatively to a 100 ml volumetric flask. Dilute to the mark with water and mix.

8.2.3 Transfer exactly 10,00 ml of this solution, (8.2.2) to a 100 ml volumetric flask, add 10 ml of hydrochloric acid (5.2), dilute to the mark with water and mix.

8.3 Preparation of the calibration solutions

To each of a series of eight 100 ml volumetric flasks, transfer 10 ml of the hydrochloric acid (5.2) and 100 µl of hydrogen peroxide (5.4).

Add 5 ml of the zinc solution (5.6) to each flask. Then add 0,00 ml, 1,00 ml, 2,00 ml, 3,00 ml, 5,00 ml, 7,00 ml, 8,00 ml and 10,0 ml aliquots of the tin standard solution (5.7). These aliquots correspond to tin contents (mass fractions) in the test portion of 0,0 %, 0,1 %, 0,2 %, 0,3 %, 0,5 %, 0,7 %, 0,8 % and 1,0 %. Dilute to the mark with water and mix.

8.4 Spectrometric measurements

Measure the absorbances of the calibration solutions and the test solution(s) by taking alternate readings to ensure that the settings of the burner and of the apparatus do not change during the readings.

The wavelength of the line used shall be 235,5 nm.

To comply with the concentration ranges recommended by the manufacturer of the apparatus, the same dilutions for the calibration solutions and the test solution(s) shall be made if necessary.

9 Calculation and expression of results

9.1 Method of calculation

Establish a calibration graph by plotting the measured absorbances of the calibration solutions against their respective contents (mass fractions).

Determine from the measured absorbances of the test solutions, the associated amount of tin from the calibration graph. If a number of determinations are carried out then a mean of all results shall be determined by adding the individual results together and by dividing by the number of individual results.

The results shall be expressed as specified in EN 13283.

9.2 Precision

A planned trail of this method was carried out by 9 laboratories, using 2 samples with 2 levels of tin contents, each laboratory making three determination of tin content in each sample (see Notes 1 and 2).

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1; i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day) by the same operator as in Note 1, using the same apparatus and a different calibration.

The details of the samples used and the mean results obtained are given in the Tables A.1 and A.2.

The results obtained were treated statistically in accordance with ISO 5725.

The data obtained showed a logarithmic relationship between the tin content and the repeatability limit (r) and reproducibility limits (R_w and R) of the test results (see Note 3), as summarised in Table 1. The graphical representation of the data is shown in Figure B.1.

NOTE 3 From the two values obtained in Day 1, the repeatability limit (r) and the reproducibility limit (R) were calculated using the procedure specified in ISO 5725. From the first value obtained in Day 1 and the value obtained in Day 2, the within-laboratory reproducibility limit (R_w) was calculated using the procedure specified in ISO 5725.

Table 1 — Repeatability limit and reproducibility limits

Tin content	Repeatability limit	Reproducibility limits	
% (mass fraction)	r	R_w	R
0,2	0,008 9	0,007 0	0,027 1
0,5	0,016 1	0,012 5	0,035 1
1,0	0,025 1	0,019 1	0,042 7

10 Test report

The test report shall include the following details:

a) identification of the sample;

b) test method used (i.e. reference to this document);

c) tin content, expressed as a percentage by mass, giving where possible the results for the individual and mean values;

d) any unusual occurrence during the determination;

e) any steps in the procedure beyond those specified in this document, and any circumstances that may have affected the results;

f) date of the test report;

g) name of the laboratory or testing organisation;

h) signature of the laboratory manager or other responsible person.