SLOVENSKI STANDARD
SIST EN 12441-9:2005

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Cink in cinkove zlitine ï Kemiļ na analiza ï 9. del: Doloļ evanje niklja v cinkovih zlitinah ï Metoda plamenske atomske absorpcijske spektrometrije

Zinc and zinc alloys - Chemical analysis - Part 9: Determination of nickel in zinc alloys - Flame atomic absorption spectrometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 9: Bestimmung von Nickel in Zinklegierungen - FAAS-Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 9 : Dosage du nickel dans les alliages de zinc - Méthode par spectrométrie d'absorption atomique dans la flamme

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ICS:

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

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Contents

Foreword

This document (EN 12441-9:2004) 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of an identical text or by endorsement, at the latest by June 2005, and conflicting national standards shall be withdrawn at the latest by June 2005.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/WG6 "Methods of analysis and testing" to prepare the following document:

EN 12441-9, *Zinc and zinc alloys – Chemical analysis – Part 9: Determination of nickel in zinc alloys – 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 8: Determination of tin in secondary zinc – 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*

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

1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of nickel in zinc alloys. It is applicable to the products specified in EN 1774 and EN 12844.

It is suitable for the determination of nickel contents (mass fractions) between 0,000 5 % and 0,020 %.

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 1774, *Zinc and zinc alloys – Alloys for foundry purposes – Ingot and liquid*

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

EN 12844, *Zinc and zinc alloys – Castings – Specifications*

3 Terms and definitions

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

3.1
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 alloy is dissolved in a nitric acid and, after adequate dissolution and atomisation of the solution in an air-acetylene flame, the content of nickel is determined by atomic absorption spectrometry at the wavelength of 232,0 nm.

5 Reagents

5.1 General

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

5.2 Nitric acid, ρ = 1,4 g/ml

5.3 Nickel, 1,0 g/l standard solution

Introduce 0,500 g of nickel [with a purity of no less than 99,99 % (mass fraction)] in the form of stripes, wire or powder, weighted to the nearest 0,001 g, into a 250 ml beaker fitted with a watch-glass. Add a few millilitres of water, 20 ml of nitric acid (5.2) and dissolve while gently heating. Dilute the solution with about 50 ml of water and boil to expel the nitrogen oxides. Cool to room temperature and transfer quantitatively to a 500 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of nickel.

5.4 Nickel, standard solution A

Transfer 25 ml of nickel solution (5.3) into a 1 000 ml volumetric flask and add 5 ml of nitric acid (5.2). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,025 mg of nickel.

5.5 Nickel, standard solution B

Transfer 25 ml of nickel solution (5.3) into a 250 ml volumetric flask and add 1 ml of nitric acid (5.2). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,100 mg of nickel.

5.6 Pure zinc, (nickel content below 0,2 µg/g)

5.7 Aluminium nitrate nonahydrate, (nickel content below 1 µg/g)

Test the nickel content before use, as this reagent may contain considerable amounts of nickel.

1,390 g corresponds to 0,100 g of aluminium.

5.8 Hydrochloric acid, ρ = 1,19 g/ml

5.9 Aqua regia

Mix 3 part volumes of hydrochloric acid (5.8) with 1 part volume nitric acid (5.2).

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 (5.9) 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 oxidiser-fuel combination of air-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 10 g of the test sample to the nearest 0,01 g.

8.2 Preparation of the test solution

8.2.1 Introduce the test portion (8.1) into a 400 ml beaker fitted with a watch-glass. Add 20 ml of water and dissolve by carefully adding 50 ml of nitric acid (5.2). Boil the solution to expel the nitrogen oxides.

8.2.2 Cool to room temperature and transfer quantitatively to a 100 ml volumetric flask. Dilute to the mark with water and mix.

8.3 Preparation of the calibration solutions

8.3.1 To each of a series of seven 400 ml beakers, add a g of the pure zinc (5.6), 20 ml of water, then carefully c ml of nitric acid (5.2) and, gently heat until complete dissolution. Allow to cool, add b g of aluminium nitrate nonahydrate (5.7) and, after dissolution, transfer quantitatively to a series of 100 ml volumetric flasks. For a, b and c, see Table 1.

Table 1 — Preparation of the calibration solutions

Aluminium content (mass fraction)	a	b	c
smaller than 0,05 %	10	0	50
between 3,7 % and 6,0 %	9,5	7	46
between 8,0 % and 11,0 %	9	14	42
between 25,0 % and 28 %	7,5	35	30

8.3.2 After cooling to room temperature, add 0,00 ml, 2,00 ml, 4,00 ml and 8,00 ml aliquots of the nickel standard solution A (5.4) and 5,0 ml, 10,0 ml and 20,0 ml aliquots of the nickel standard solution B (5.5). These aliquots correspond to nickel contents (mass fractions) in the test portion of 0,000 0 %, 0,000 5 %, 0,001 0 %, 0,002 0 %, 0,005 0 %, 0,010 % and 0,020 %. 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 232,0 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 nickel 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 1774 and EN 12844.

9.2 Precision

A planned trial of this method was carried out by 10 laboratories, using 4 samples with 4 levels of nickel contents, each laboratory making three determinations of nickel 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 nickel 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 2 — Repeatability limit and reproducibility limits

Nickel content	Repeatability limit	Reproducibility limits	
% (mass fraction)	r	R_w	R
0,000 5	0,000 04	0,000 15	0,000 18
0,001 0	0,000 06	0,000 18	0,000 28
0,002 0	0,000 10	0,000 22	0,000 44
0,005 0	0,000 19	0,000 30	0,000 78
0,010 0	0,000 31	0,000 37	0,001 19
0,020 0	0,000 50	0,000 46	0,001 84