SLOVENSKI STANDARD
SIST EN 12441-5:2004

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Cink in cinkove zlitine ï Kemiļ na analiza ï 5. del: Doloļ evanje ģeleza v primarnem cinku ï Spektrofotometrijska metoda

Zinc and zinc alloys - Chemical analysis - Part 5: Determination of iron in primary zinc - Spectrophotometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 5: Bestimmung von Eisen in Primärzink - Spektrophotometrisches Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 5: Dosage du fer dans le zinc primaire - Méthode spectrophotométrique

Ta slovenski standard je istoveten z: **EN 12441-5:2003**

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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English version

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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 12441-5:2003) 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of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.

Within its programme of work, Technical Committee CEN/TC 209 entrusted CEN/TC 209/WG 6, “Methods of analysis and testing” with the preparation of the following document:

EN 12441-5, *Zinc and zinc alloys - Chemical analysis – Part 5: Determination of iron in primary zinc – Spectrophotometric method*.

This standard is part of series of eleven standards. The other standards are:

— EN 12441-1, *Zinc and zinc alloys – Chemical analysis – Part 1: Determination of aluminium in zinc alloys – Titrimetric method*;

— EN 12441-2, *Zinc and zinc alloys – Chemical analysis – Part 2: Determination of magnesium in zinc alloys – Flame atomic absorption spectrometric method*;

— EN 12441-3, *Zinc and zinc alloys – Chemical analysis – Part 3: Determination of lead, cadmium and copper – Flame atomic absorption spectrometric method*;

— EN 12441-4, *Zinc and zinc alloys – Chemical analysis – Part 4: Determination of iron in zinc alloys – Spectrophotometric method.*.

— EN 12441-6, *Zinc and zinc alloys – Chemical analysis – Part 6: Determination of aluminium and iron – Flame atomic absorption spectrometric method*;

— prEN 12441-7, *Zinc and zinc alloys – Chemical analysis – Part 7: Determination of tin – Flame atomic absorption spectrometric method after extraction*;

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

— prEN 12441-9, *Zinc and zinc alloys – Chemical analysis – Part 9: Determination of nickel in zinc alloys – Flame atomic absorption spectrometric method*;

— prEN 12441-10, *Zinc and zinc alloys – Chemical analysis – Part 10: Determination of chromium and titanium in zinc alloys – Spectrophotometric method*;

— prEN 12441-11, *Zinc and zinc alloys – Chemical analysis – 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, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a spectrophotometric method for the determination of iron in primary zinc. It is applicable to the products specified in EN 1179.

It is suitable for the determination of iron contents (mass fractions) between 0,001 % and 0,1 %.

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 1179, *Zinc and zinc alloys - Primary zinc.*

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

3 Terms and definitions

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

4 Principle

Formation of a sulfosalicylic acid ferric complex in an ammoniacal medium. Spectrophotometric measurement of the yellow colour of the thus formed complex.

5 Reagents

5.1 General

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

5.2 Ammonia solution, ρ = 0,91 g/ml.

5.3 Hydrochloric acid, ρ = 1,19 g/ml.

5.4 Hydrogen peroxide, 30 % (mass fraction).

5.5 Sulfosalicylic acid, 400 g/l solution.

5.6 Nickel chloride solution

Dissolve 2 g of nickel chloride hexahydrate ($NiCl_2.6H_2O$) in 500 ml of water. Make up to 1 l and mix.

5.7 Standard iron solution A

Weigh 0,250 g of pure iron to the nearest 0,001 g. Add by small fractions approximately 10 ml of hydrochloric acid (5.3). Oxidise by adding a few drops of hydrogen peroxide (5.4). Decompose the excess hydrogen peroxide by boiling. Cool to room temperature. Transfer quantitatively to a 1 l volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 0,250 mg of iron.

5.8 Standard iron solution B

Transfer exactly 20 ml of standard iron solution A (5.7) to a 100 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 0,050 mg of iron.

5.9 Nitric acid, $\rho = 1{,}4$ g/ml.

5.10 Aqua regia

Mix 3 volumes of hydrochloric acid (5.3) with 1 volume of nitric acid (5.9).

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 (5.10) prior to use.

6.2 Specific equipment

In addition to standard laboratory apparatus, a spectrophotometer, set at a wavelength of 425 nm and using 1 cm optical cells shall be used.

NOTE The dilution and aliquot parts defined in this standard only apply if 1cm cells are used. It is necessary to apply the appropriate modifications in the case of cells with other dimensions.

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 Blank test

Simultaneously with each determination, carry out a blank test using the same quantities of each reagent and following the same procedure.

8.3 Preparation of the test solution

8.3.1 Introduce the test portion (8.1) in a 500 ml beaker fitted with a watch-glass and dissolve by carefully adding 50 ml of hydrochloric acid (5.3). Oxidise and complete the dissolution by adding a few drops of hydrogen peroxide (5.4).

If dissolution is very difficult, 2 ml of nickel chloride solution (5.6) should be added to expedite the attack.

8.3.2 Decompose the excess hydrogen peroxide by boiling.

8.3.3 For iron contents (mass fractions) equal to or greater than 0,01 % proceed as follows (if less than 0,01 % see 8.3.4):

a) cool to room temperature. Transfer quantitatively to a 250 ml volumetric flask. Dilute to the mark with water and mix;

b) transfer a 25 ml aliquot to a 100 ml volumetric flask;

c) add successively:

1) 25 ml of water;

2) 5 ml of sulfosalicylic acid (5.5);

3) ammonia solution (5.2) until the solution becomes yellow, then 20 ml in excess;

d) cool to room temperature. Dilute to the mark with water and mix.

8.3.4 For iron contents (mass fractions) less than 0,01 % proceed as follows:

a) evaporate the test portion until a syrupy consistency is obtained;

b) cool to room temperature;

c) take up with a minimum of water (so as not to exceed 30 ml) and transfer quantitatively to a 100 ml volumetric flask;

d) add successively:

1) 5 ml of sulfosalicylic acid solution (5.5);

2) ammonia solution (5.2) until the solution has a yellow colour, then 50 ml in excess;

e) cool to room temperature. Dilute to the mark and mix. Measure the absorbance of the solutions (see 8.5) after first measuring the calibration graph as in 8.4.

8.4 Calibration graph

NOTE The following is valid for 1 cm cells and for iron contents (mass fractions) of 0 mg, 0,10 mg, 0,25 mg, 0,5 mg and 1,00 mg of iron corresponding to mass fractions in the test portion of 0 %, 0,01 %, 0,025 % 0,05 % and 0,1 % when using the procedure specified in 8.3.3 and 0 %, 0,001 % 0,0025 %, 0,005 % and 0,01 % when using the procedure specified in 8.3.4. It is necessary to apply the appropriate modifications in the case of cells of different dimensions.

8.4.1 Introduce into a series of 100 ml volumetric flasks, 0,00 ml, 2,00 ml, 5,00 ml, 10,00 ml and 20,00 ml of standard iron solution B (5.8).

8.4.2 Add successively:

a) 5 ml of sulfosalicylic acid solution (5.5);

b) ammonia solution (5.2) until the solution becomes yellow, then 20 ml in excess.

8.4.3 Cool to room temperature. Dilute to the mark with water and mix.

8.4.4 Measure the absorbance of each solution against the solution with 0,00 ml standard iron solution B, in 1 cm cells, using the spectrophotometer (6.2) at a wavelength of 425 nm.

8.4.5 Produce a calibration graph by plotting the iron content versus the absorbance readings obtained for each calibration standard.