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Zinc and zinc alloys - Chemical analysis - Part 4: Determination of iron in zinc alloys -
 Spectrophotometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 4: Bestimmung von Eisen in
 Zinklegierungen - Spektrophotometrisches Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 4: Dosage du fer dans les alliages de
 zinc - Méthode spectrophotométrique

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Ta slovenski standard je istoveten z: EN 12441-4:2003

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

SIST EN 12441-4:2004

en

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 12441-4

March 2003

ICS 77.040.30; 77.120.60

English version

**Zinc and zinc alloys - Chemical analysis - Part 4: Determination
of iron in zinc alloys - Spectrophotometric method**

Zinc et alliages de zinc - Analyse chimique - Partie 4:
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spectrophotométrique

Zink und Zinklegierungen - Chemische Analyse - Teil 4:
Bestimmung von Eisen in Zinklegierungen -
Spektrophotometrisches Verfahren

This European Standard was approved by CEN on 21 November 2002.

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 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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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

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Foreword

This document (EN 12441-4:2003) 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 12441-4, *Zinc and zinc alloys – Chemical analysis – Part 4: Determination of iron in zinc alloys – Spectrophotometric method.*

This standard is part of a 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 12441-5, *Zinc and zinc alloys – Chemical analysis – Part 5: Determination of iron in primary zinc – 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 organisations 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.

EN 12441-4:2003 (E)

1 Scope

This European Standard specifies a spectrophotometric method for the determination of iron in zinc alloys. It is applicable to the products specified in EN 1774 and EN 12844.

It is suitable for the determination of iron contents (mass fractions) between 0,01 % 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 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 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, after separation of the copper. Spectrophotometric measurement of the yellow colour of the complex thus formed.

5 Reagents

5.1 General

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

5.2 Pure granulated cadmium, free from iron

5.3 Hydrochloric acid, $\rho = 1,19$ g/ml

5.4 Hydrochloric acid (1 + 1)

Mix 1 volume of hydrochloric acid (5.3) with 1 volume of water

5.5 Hydrogen peroxide, 30 % (mass fraction)

5.6 Sulfosalicylic acid, 800 g/l solution

5.7 Ammonia solution, $\rho = 0,91$ g/ml

5.8 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 a few drops of hydrogen peroxide (5.5). 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.

5.9 Standard iron solution B

Transfer exactly 20 ml of standard iron solution A (5.8) 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.

5.10 Nitric acid, $\rho = 1,4$ g/ml.

5.11 Aqua regia

Mix 3 volumes of hydrochloric acid (5.3) with 1 volume of nitric acid (5.10).

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 (5.11) 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 1 cm 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.

EN 12441-4:2003 (E)**8.3 Preparation of the test solution**

8.3.1 Introduce the test portion (8.1) into a 500 ml beaker fitted with a watch-glass and dissolve by carefully adding 100 ml of hydrochloric acid (1 + 1) (5.4). Oxidise and complete the dissolution by adding a few drops of hydrogen peroxide (5.5).

8.3.2 Decompose the excess hydrogen peroxide by boiling.

8.3.3 Add 2 g of cadmium (5.2) and heat gently for 3 min, shaking frequently in order to reduce the copper completely.

8.3.4 Cool to room temperature. Filter and wash through a rapid filter paper collecting the solution and washings quantitatively in a 250 ml volumetric flask. Dilute to the mark with water and mix.

8.3.5 Transfer a 25 ml aliquot to a 100 ml volumetric flask.

Add successively:

- a) 20 ml of sulfosalicylic acid solution (5.6) ;
- b) ammonia solution (5.7) until the solution has a yellow colour, then 20 ml in excess.

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

8.4 Calibration graph

NOTE The following is valid for 1 cm cells and for iron contents of 0,00 mg, 0,10 mg, 0,25 mg, 0,50 mg and 1,00 mg, corresponding to contents (mass fractions) in the test portion of 0 %, 0,010 %, 0,025 %, 0,050 % and 0,100 % of iron. It is necessary to apply appropriate modifications in the case of cells of different dimensions.

8.4.1 Introduce into a series of 100ml volumetric flasks 0,00 ml, 2,00 ml, 5,00 ml ; 10,00 ml and 20,00 ml respectively of standard iron solution B (5.9). Dilute to about 50 ml.

8.4.2 Add successively:

- a) 20 ml of sulfosalicylic acid solution (5.6);
- b) ammonia solution (5.7) until the solution has a yellow colour, 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.

8.5 Spectrophotometric measurement

Measure the absorbance of the test solution(s) against the blank solution (8.2) in 1 cm cells using the spectrophotometer (6.2) at a wavelength of 425 nm.