SLOVENSKI STANDARD
SIST EN 12441-6:2004

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Cink in cinkove zlitine ï Kemiļ na analiza ï 6. del: Doloļ evanje aluminija in ģeleza ï Metoda plamenske atomske absorpcijske spektrometrije

Zinc and zinc alloys - Chemical analysis - Part 6: Determination of aluminium and iron- Flame atomic absorption spectrometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 6: Bestimmung von Aluminium und Eisen - FAAS-Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 6: Dosage de l'aluminium et du fer- Méthode par spectrométrie d'absorption atomique dans la flamme

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Zinc and zinc alloys - Chemical analysis - Part 6: Determination of aluminium and iron-Flame atomic absorption spectrometric method

Zinc et alliages de zinc - Analyse chimique - Partie 6: Dosage de l'aluminium et du fer-Méthode par spectrométrie d'absorption atomique dans la flamme

Zink und Zinklegierungen - Chemische Analyse - Teil 6: Bestimmung von Aluminium und Eisen - FAAS-Verfahren

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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.

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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 12441-6:2003) 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of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.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/*WG6 "Methods of analysis and testing*" with the preparation the following document:

EN12441-6, Zinc and zinc alloys - Chemical analysis - Part 6: Determination of aluminium and iron - Flame atomic absorption spectrometric 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 12441-4, *Zinc and zinc alloys – Chemical analysis – Part 4: Determination of iron in zinc alloys – Spectrophotometric method.*

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

Annex A is 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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a flame atomic absorption spectrometry method in zinc and zinc alloys for the determination of aluminium and iron. It is applicable to the products specified in EN 988, EN 1179, EN 1774, EN 12844 and EN 13283.

It is suitable for the determination of iron contents (mass fractions) between 0,001 % and 0,5 % and aluminium contents (mass fractions) between 0,000 5 % and 0,5 %.

NOTE EN 12441-1 specifies a method for the determination of aluminium contents (mass fractions) between 3 % and 30 %.

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 988, *Zinc and zinc alloys - Specifications for rolled flat products for building.*

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

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.*

EN 13283, *Zinc and zinc alloys - Secondary zinc.*

3 Terms and definitions

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

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

4 Principle

A sample of the metal or alloy is dissolved in hydrochloric acid and, after adequate dissolution and atomisation of the solution in an air-acetylene (or nitrous oxide-acetylene) flame, the content of the particular component is determined by atomic absorption spectrometry.

The wavelengths of the spectral lines are shown in annex A.

5 Reagents

5.1 General

During the test use only reagents of known 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 Hydrochloric acid (1 + 1)

Mix 1 volume of hydrochloric acid (5.2) with 1 volume of water.

5.5 Hydrogen peroxide, 30 % (mass fraction)

5.6 Iron, 1 g/l solution

Introduce 1,000 g of pure iron (purity 99,95 %) into a 250 ml beaker fitted with a watch-glass and dissolve by carefully adding 50 ml of hydrochloric acid (1 + 1) (5.4). After effervescence has stopped, add 1 ml of hydrogen peroxide (5.5) and boil to complete dissolution. Cool to room temperature and transfer to a 1 l volumetric flask. Add 50 ml of hydrochloric acid (5.2), dilute to the mark with water and mix. Store in a polyethylene bottle.

1 ml of this solution contains 1 mg of iron.

5.7 Standard iron, solution A

Transfer 10 ml of iron solution (5.6) into a 500 ml volumetric flask. Add 5 ml of hydrochloric acid (5.2), dilute to the mark with water and mix.

1 ml of this solution contains 0,02 mg of iron.

This solution shall be freshly prepared just before use.

5.8 Standard iron, solution B

Transfer 10 ml of iron solution (5.6) into a 200 ml volumetric flask. Add 2 ml of hydrochloric acid (5.2), dilute to the mark with water and mix.

1 ml of this solution contains 0,05 mg of iron.

This solution shall be freshly prepared just before use.

5.9 Aluminium, 1 g/l solution

Introduce 1,000 g of aluminium (purity 99,95 % min) into a 250 ml beaker fitted with a watch-glass and dissolve by carefully adding 50 ml of hydrochloric acid (1 + 1) (5.4). After effervescence has stopped, add 1 ml of hydrogen peroxide (5.5) and boil to complete dissolution. Cool and transfer to a 1 l volumetric flask. Add 50 ml of hydrochloric acid (5.2), dilute to the mark with water and mix. Store in a polyethylene bottle.

1 ml of this solution contains 1 mg of aluminium.

5.10 Standard aluminium, solution A

Transfer 10 ml of aluminium solution (5.9) into a 100 ml volumetric flask. Add 5 ml of hydrochloric acid (5.2), dilute to the mark with water and mix.

1 ml of this solution contains 0,1 mg of aluminium.

This solution shall be freshly prepared just before use.

5.11 standard aluminium, solution B

Transfer 20 ml of aluminium solution (5.9) into a 100 ml volumetric flask. Add 5 ml of hydrochloric acid (5.2), dilute to the mark with water and mix.

1 ml of this solution contains 0,2 mg of aluminium.

This solution shall be freshly prepared just before use.

5.12 **Zinc**, 200 g/l solution

Introduce 200 g of zinc (purity 99,995 %, absent of iron and aluminium) into a 2 l beaker. Add 100 ml of water and 700 ml of hydrochloric acid (5.2) carefully in small amounts to control the rate of reaction. Add 1 ml of hydrogen peroxide (5.5) and evaporate carefully until a syrupy consistency is obtained. After cooling, dilute to about 800 ml with water and warm gently to dissolve any salts. Transfer into a 1 l volumetric flask, dilute to the mark with water and mix. Store in a polyethylene bottle.

1 ml of this solution contains 0,2 g of zinc.

5.13 **Sodium chloride**, 100 g/l solution

5.14 Aqua regia

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

5.15 Hydrochloric acid (1 + 19)

Mix 1 volume of hydrochloric acid (5.2) with 19 volumes of water.

5.16 **Ammonia solution**, ρ = 0,91 g/ml

5.17 Lanthanum solution

Introduce 5 g of lanthanum oxide (La_2O_3) in a 200 ml beaker. Add 5 ml of water and carefully dissolve with 20 ml of hydrochloric acid (5.2). After dissolution, cool to room temperature and transfer to a 1l volumetric flask. Dilute to the mark with water and mix.

5.18 **Phenolphthaleine**, ethanolic solution.

Dissolve 1 g of phenophtaleine in 100 ml of ethanol.

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 (5.14) prior to use.

6.2 Specific equipment

In addition to standard laboratory apparatus, the following shall be used :

a) **atomic absorption spectrometer,** equipped with a premix burner, with facilities for using the oxidiser–fuel combinations of air–acetylene or nitrous oxide–acetylene.

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 and 10 cm.

Spectral lines and oxidiser-fuel combinations are listed in annex A.

b) **membrane filtration apparatus**, with cellulose nitrate membrane filter having a diameter of 50 mm or 30 mm and a pore size of 0,45 µm or 0,6 µm.

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 0,001 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 and dissolve by carefully adding 50 ml of hydrochloric acid (5.2). Oxidise and complete the dissolution by adding a few drops of hydrogen peroxide (5.5).

8.2.2 Decompose the excess hydrogen peroxide by boiling.

8.2.3 Cool to room temperature and transfer to a 100 ml volumetric flask. In the particular case of aluminium in the range of contents (mass fractions) between 0,005 % to 0,025 %, add 5 ml of sodium chloride solution (5.13) before dilution to the mark (see Table 1). Dilute to the mark with water and mix.

8.2.4 Using Table 1 as a guide, transfer appropriate volumes of sample solution (8.2.3) sodium chloride solution (5.13), and hydrochloric acid (5.2) to a 100 ml volumetric flask. Dilute to the mark with water and mix.

Table 1 — Test solutions

Element	**Concentration range** % (mass fractions)	**Sample solution** ml/100 ml	**Hydrochloric acid** ml	**NaCl solution** ml
Aluminium	0,005 to 0,025	100[a]	-	5
	0,020 to 0,100	50	-	5
	0,100 to 0,500	10	10	5
Iron	0,001 to 0,005	100[a]	-	-
	0,005 to 0,025	50	-	-
	0,025 to 0,125	10	10	-
	0,100 to 0,500	1	10	-

[a] The test solution is the undiluted sample solution.