SLOVENSKI STANDARD
SIST EN 12441-10:2005

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Cink in cinkove zlitine ï Kemiļ na analiza ï 10. del: Doloļ evanje kroma in titana v cinkovih zlitinah ï Metoda spektrofotometrije

Zinc and zinc alloys - Chemical analysis - Part 10: Determination of chromium and titanium in zinc alloys - Spectrophotometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 10: Bestimmung von Chrom und Titan in Zinklegierungen - Spektrophotometrisches Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 10: Dosage du chrome et du titane dans les alliages de zinc - Méthode spectrophotométrique

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

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Contents

Foreword

This document (EN 12441-10:2004) 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of an identical text or by endorsement, at the latest by June 2005, and conflicting national standards shall be withdrawn at the latest by June 2005.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 12441-10, *Zinc and zinc alloys – Chemical analysis – Part 10: Determination of chromium and titanium in zinc alloys – Spectrophotometric 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 9: Determination of nickel in zinc alloys – Flame atomic absorption spectrometric 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 spectrophotometric method for the determination of chromium and titanium in zinc alloys. It is applicable to the products specified in EN 988, EN 1774 and EN 12844.

It is suitable for the determination of chromium and titanium contents (mass fractions) between 0,05 % and 0,50 %.

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 apply.

4 Principle

For both determinations the same dissolution and oxidation step is used.

Chromium is determined by spectrophotometry of the violet chromium diphenylcarbazide complex.

Titanium is determined by spectrophotometry of the red chromotropic acid titanium complex, formed in a solution buffered at pH 2,9.

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 Perchloric acid, ρ = 1,6 g/ml

5.5 Sulphuric acid, ρ = 1,83 g/ml

5.6 Chloroacetic acid

5.7 Sodium hydroxide

5.8 Sulphuric acid, (1+1)

Carefully add 1 part volume of sulphuric acid (5.5) to 1 part volume of water.

5.9 Sulphuric acid, 2M

Carefully pour 110 ml of sulphuric acid (5.5) into water and dilute to a final volume of 1 litre.

5.10 Aqua regia

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

5.11 Pure titanium

5.12 Titanium oxalate potassium, pure and dry $[K_2TiO(COO)_4]\ 2H_2O$

5.13 Chromotropic acid, 60 g/l (fresh solution)

5.14 Buffer solution, pH 2,9 (fresh solution)

Dissolve separately in 600 ml beakers, each with about 400 ml of water:

— 236 g of chloracetic acid (5.6);

— 50 g of sodium hydroxide (5.7).

Transfer both solutions quantitatively to a 1 litre volumetric flask. Dilute to the mark with water and mix.

5.15 Ascorbic acid, 20 g/l (fresh solution)

5.16 Titanium, 1 g/l standard solution

Introduce either 1,848 g of titanium oxalate potassium (5.12), either 0,250 g of pure titanium (5.11), weighted to the nearest 0,001 g, into a 400 ml beaker. Add 50 ml of sulphuric acid (5.8). Heat gently to complete dissolution. Cool to room temperature and transfer to a 250 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of titanium.

5.17 Titanium, 25 mg/l standard solution (fresh solution)

Transfer 25 ml of the titanium standard solution (5.16) to a 1 litre volumetric flask.

Dilute to the mark with water and mix.

1 ml of this solution contains 0,025 mg of titanium.

5.18 Potassium dichromate, dried at 105 °C.

5.19 Acetone

5.20 Diphenyl carbazide, 10 g/l (fresh solution)

Dissolve 1 g of diphenyl carbazide in 100 ml of acetone (5.19).

5.21 Chromium, 500 mg/l standard solution

Introduce 1,414 5 g of potassium dichromate (5.18) weighted to the nearest 0,000 5 g into a 400 ml beaker. Add 200 ml of water and 50 ml of sulphuric acid (5.8). Heat gently to complete dissolution. Cool to room temperature and transfer to a 1 litre volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 0,5 mg of chromium.

5.22 Chromium, 25 mg/l standard solution

Transfer 25 ml of the chromium standard solution (5.21) to a 500 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 0,025 mg of chromium.

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 540 nm for chromium, 470 nm for titanium and using 1 cm optical cells shall be used.

NOTE The dilution and aliquot parts defined in this document 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 1 g of the test sample to the nearest 0,001 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 General

Introduce the test portion (8.1) into a 250 ml beaker fitted with a watch-glass and carefully add 5 ml of hydrochloric acid (5.2). Complete the dissolution by adding 2 ml of nitric acid (5.3). Add 10 ml of perchloric acid (5.4) and heat by raising the temperature gradually [in order to allow complete oxidation to Cr (VI)], until strong white fumes are evolved. Cool to room temperature, add 50 ml of water and heat gradually to boiling to dissolve salts. Cool to room temperature and transfer quantitatively to a 200 ml volumetric flask. Dilute to the mark with water and mix.

8.3.2 Chromium determination

Transfer a 10 ml aliquot of the sample solution (8.3) to a 200 ml volumetric flask and add successively:

— 5 ml of sulphuric acid (5.9);

— 80 ml of water;

— 1 ml of diphenyl carbazide solution (5.20).

Dilute to the mark with water and mix.

Wait for 15 minutes, then measure the absorbance of this solution against the blank test solution (8.2) in 1 cm cells using the spectrophotometer (6.2) set at a wavelength of 540 nm.

8.3.3 Titanium determination

Transfer a 10 ml aliquot of the sample solution (8.3) to a 200 ml volumetric flask and add successively:

— 10 ml of ascorbic acid (5.15);

— 2 ml of chromotropic acid (5.13);

— 20 ml of buffer solution (5.14).

Dilute to the mark with water and mix.

Wait for 15 minutes, then measure the absorbance of this solution against the blank test solution (8.2) in 1 cm cells using the spectrophotometer (6.2) set at a wavelength of 470 nm.

8.4 Calibration

NOTE The following is valid for 1 cm cells and for 0 mg, 0,025 mg, 0,05 mg, 0,10 mg, 0,15 mg, 0,20 mg and 0,25 mg of chromium or titanium corresponding to contents (mass fractions) in the test portion of 0 %, 0,05 %, 0,1 %, 0,2 %, 0,3 %, 0,4 % and 0,5 %. It is necessary to apply the appropriate modifications in the case of cells of different lengths.

8.4.1 Chromium

Introduce into a series of 200 ml volumetric flasks, 0,00 ml, 1,00 ml, 2,00 ml, 4,00 ml, 6,00 ml, 8,00 ml and 10,00 ml of the chromium standard solution (5.22) and proceed as specified in 8.3.2.

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

8.4.2 Titanium

Introduce into a series of 200 ml volumetric flasks, 0,00 ml, 1,00 ml, 2,00 ml, 4,00 ml, 6,00 ml, 8,00 ml and 10,00 ml of the titanium standard solution (5.17) and proceed as specified in 8.3.3.

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