



SLOVENSKI STANDARD
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Zinc and zinc alloys - Chemical analysis - Part 11: Determination of silicon in zinc alloys - Spectrophotometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 11: Bestimmung von Silicium in Zinklegierungen - Spektrophotometrisches Verfahren

iTeh STANDARD PREVIEW

Zinc et alliages de zinc - Analyse chimique - Partie 11: Dosage du silicium dans les alliages de zinc - Méthode spectrophotométrique

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English Version

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Bestimmung von Silicium in Zinklegierungen -
Spektrophotometrisches Verfahren

This European Standard was approved by CEN on 7 July 2006.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 12441-11:2006) 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of an identical text or by endorsement, at the latest by February 2007, and conflicting national standards shall be withdrawn at the latest by February 2007.

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 12441-11, *Zinc and zinc alloys – Chemical analysis – Part 11: Determination of silicon in zinc alloys – Spectrophotometric method.*

This standard is a part of a series of eleven standards. The other standards 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 10: Determination of chromium and titanium 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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

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

It is suitable for the determination of silicon contents (mass fractions) between 0,01 % and 0,1 %.

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 European Standard, the terms and definitions given in EN 12060:1997 apply.

4 Principle

Direct spectrophotometric determination of silicon molybdenum blue after dissolution of the sample in hydrochloric and hydrofluoric acid.

5 Reagents

5.1 General

During the test, use only reagents of known analytical grade.

5.2 Bidistilled water (for preparation and dilution of all solutions)

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

5.4 Hydrofluoric acid, $\rho = 1,13$ g/ml

5.5 Boric acid

5.6 Ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$

5.7 Sodium metabisulfite $\text{Na}_2\text{S}_2\text{O}_5$

5.8 Sodium sulfite Na_2SO_3

5.9 1-amino-2-naphtol-4-sulfonic acid

5.10 Molybdate solution

Dissolve 100 g of boric acid (5.6) in 3 litres of water and dissolve 25 g of ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ in 250 ml of water.

Transfer both solutions into a 5 litre volumetric flask. Dilute to the mark with water and mix.

5.11 Reducing solution

Dissolve 100 g of $\text{Na}_2\text{S}_2\text{O}_5$ (5.7) in 600 ml of tepid water (fraction 1) and 4 g of Na_2SO_3 (5.8) in 40 ml of tepid water (fraction 2). Introduce 2 g of 1-amino-2-naphthol-4-sulfonic acid (5.9) in 40 ml of tepid water (fraction 3) and mix. Add fraction 2 to fraction 3 and mix until the solution is clear. Dilute with fraction 1.

Transfer to a 1 l volumetric flask. Dilute to the mark with water and mix.

5.12 Silicon standard solution

In hot water dissolve, 0,6 342 g of ammonium hexafluoro-silicate $[(\text{NH}_4)_2\text{SiF}_6]$, dried beforehand between 105 °C and 110 °C and cooled in a dessiccator. Let cool, then transfer in a 500 ml volumetric polyethylene flask. Dilute to the mark with water and mix.

1 ml of this solution contains 200 µg of silicon.

6 Apparatus

6.1 Plastic bottles with screw cap, volumetric flasks, pipettes etc.,

made of polytetrafluoroethylene (PTFE), polypropylene (PP), polyethylene (PE), polymethylpentene or other equivalent polymers.

New vessels have to be cleaned with warm hydrofluoric acid (5.4)

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

- magnetic stirrer and stirring bars (magnetic core with PTFE jacket);
- spectrophotometer, set at a wavelength of 820 nm and using 1 cm optical cells.

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 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 Introduce the test portion (8.1) in a 50 ml plastic bottle, add 5 ml of water and then 12 ml of hydrochloric acid (5.3) in portions of 1 ml or 2 ml. Cool if the reaction is too violent.

8.3.2 After effervescence has stopped, add 2 ml of hydrofluoric acid (5.4).

8.3.3 After effervescence has stopped, add a magnetic stirrer, close the bottle with a screw cap and stir until complete dissolution. Depending of the alloy this may take between 0,5 h and 3 h.

8.3.4 Transfer the solution quantitatively into a 50 ml plastic volumetric flask. Dilute to the mark with water and mix.

8.3.5 Transfer 1 ml of solution 8.3.4 into a 50 ml plastic volumetric flask, let stand for 1 h, then add 45 ml of molybdate solution (5.10).

8.3.6 After 15 minutes, add 2 ml of reducing solution (5.11). Dilute to the mark with water and mix.

8.3.7 After 15 minutes, 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 820 nm, after first measuring the calibration solutions as in (8.4).

8.4 Calibration

NOTE The following is valid for 1 cm cells and for 0 mg, 0,1 mg, 0,2 mg, 0,4 mg, 0,6 mg, 0,8 mg, 1,0 mg and 1,2 mg of silicon corresponding to contents (mass fractions) in the test portion of 0 %, 0,01 %, 0,02 %, 0,04 %, 0,06 %, 0,08 %, 0,10 % and 0,12 %. It is necessary to apply the appropriate modifications in the case of cells of different lengths.

8.4.1 Introduce into a series of 50 ml plastic flasks, 0,00 ml, 0,50 ml, 1,00 ml, 2,00 ml, 3,00 ml, 4,00 ml, 5,00 ml and 6,00 ml respectively of the silicon standard solution (5.12).

8.4.2 Add 5 ml of water, 12 ml of hydrochloric acid (5.3) and 2 ml of hydrofluoric acid (5.4). Dilute to the mark with water and mix.

8.4.3 Transfer 1 ml of solution (8.4.2) into 50 ml plastic flasks and add 45 ml of Molybdate solution (5.10).

8.4.4 Wait for 15 minutes, then add 2 ml of reducing solution (5.11). Dilute to the mark with water and homogenise.

8.4.5 Wait for 15 minutes, then measure the absorbance of each solution against the zero standard of the calibration range, in 1 cm cells, using the spectrophotometer (6.2), at a wavelength of 820 nm.

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

9 Calculation and expression of results

9.1 Method of calculation

Determine from the measured absorbances of the test solution the associated amount of silicon from the calibration curve (8.4). If a number of determinations (n) are carried out then a mean of all results shall be determined.

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 6 samples with 6 levels of silicon contents, each laboratory making three determinations of silicon 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 i.e., one operator, same apparatus, identical operating conditions, same calibrations 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 different calibrations.

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 5725-1.

The data obtained showed a logarithmic relationship between the silicon content and the repeatability limit [r] and reproducibility limits [R_w and R] of the 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 1 — Results for repeatability limit and reproducibility limits

Silicon content % (mass fraction)	Repeatability limit		Reproducibility limits	
	r	R_w	R	
0,01	0,0 013	0,0 013	0,0 020	
0,02	0,0 017	0,0 019	0,0 031	
0,05	0,0 024	0,0 034	0,0 054	
0,1	0,0 030	0,0 051	0,0 082	
0,2	0,0 038	0,0 078	0,0 126	

10 Test report

The test report shall include the following details:

- identification of sample;
- test method used (i.e. reference to this standard);
- silicon content, expressed as percentage by mass, giving where possible the results for the individual and the mean value;
- any unusual occurrence during the determination;
- any steps in the procedure beyond those specified in this standard, and any circumstances that may have affected the results;
- date of the test report;
- name of the laboratory or testing organisation;
- signature of the laboratory manager or other responsible person.