SLOVENSKI STANDARD
SIST EN 12441-7:2005

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Cink in cinkove zlitine ï Kemiļ na analiza ï 7. del: Doloļ evanje kositra ï Metoda plamenske atomske absorpcijske spektrometrije po ekstrakciji

Zinc and zinc alloys - Chemical analysis - Part 7: Determination of tin - Flame atomic absorption spectrometric method after extraction

Zink und Zinklegierungen - Chemische Analyse - Teil 7: Bestimmung von Zinn - FAAS-Verfahren nach Extraktion

Zinc et alliages de zinc - Analyse chimique - Partie 7: Dosage de l'étain - Méthode par spectrométrie d'absorption atomique dans la flamme apres extraction

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Zinc and zinc alloys - Chemical analysis - Part 7: Determination of tin - Flame atomic absorption spectrometric method after extraction

Zinc et alliages de zinc - Analyse chimique - Partie 7: Dosage de l'étain - Méthode par spectrométrie d'absorption atomique dans la flamme après extraction

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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 Central Secretariat has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 12441-7:2004) 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of an identical text or by endorsement, at the latest by June 2005, and conflicting national standards shall be withdrawn at the latest by June 2005.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 12441-7, *Zinc and zinc alloys – Chemical analysis – Part 7: Determination of tin – Flame atomic absorption spectrometric method after extraction.*

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 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*

— *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 flame atomic absorption spectrometric method after extraction for the determination of tin in zinc and zinc alloys. It is applicable to the products specified in EN 988, EN 1179, EN 1774 and EN 12844.

It is suitable for the determination of tin contents (mass fractions) between 0,000 5 % and 0,005 %.

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 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*

3 Terms and definitions

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

3.1
atomic absorption spectrometry
measurement of the absorption of an electromagnetic radiation, emitted by an element at a determined wavelength, by an absorbent medium (flame) formed by atoms of the same element that are in the ground state. Each element absorbs radiation of specific wavelengths and the intensity of the absorbed radiation is proportional to the concentration of said element

4 Principle

A sample of the metal or alloy is dissolved in hydrochloric acid. Tin is extracted in organic phase using an iso-butyl acetate solution of trioctylphosphine oxyde (T.O.P.O.). The content of tin is determined by atomic absorption spectrometry at the wavelength of 286,3 nm, after direct nebulization of the organic phase.

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 Hydrogen peroxide, 30 % (mass fraction)

Hydrogen peroxide solutions are liable to be stabilised with products containing tin. Therefore exactly the same volume of hydrogen peroxide shall be used both when dissolving samples and when preparing the calibration solutions.

5.4 Iso-butyl acetate

5.5 Tri-n-octylphosphinoxide (T.O.P.O.), 10 g/l solution

In a 200 ml volumetric flask, dissolve 2 g of tri-n-octylphosphinoxide in iso-butyl acetate (5.4).

Dilute to the mark with iso-butyl acetate (5.4) and mix.

5.6 Pure zinc 99,995 %, tin free

5.7 Zinc solution, 200 g/l solution

Introduce 200 g of zinc (5.6) into a 2 litre beaker. Add 700 ml of hydrochloric acid (5.2) carefully in small amounts to control the rate of reaction.

When the almost part of the metal is dissolved, add about 100 ml of water and heat gently until zinc is completely dissolved.

Do not allow any concentration of the solution by heating.

After cooling, 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 approximately 200 mg of zinc.

NOTE If dissolution is very difficult, 2 ml of nickel chloride solution (5.10) may be added to expedite the attack.

5.8 Tin, 1 g/l standard solution

Weigh 1,000 g of tin [with a purity of no less than 99,9 % (mass fraction)] to the nearest 0,001 g. Carefully add 100 ml of hydrochloric acid (5.2). When dissolution is complete, cool to room temperature and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of tin.

5.9 Tin, 0,05 g/l standard solution

Transfer 10 ml of tin solution (5.8) into a 200 ml volumetric flask. Add 20 ml of hydrochloric acid (5.2), dilute to the mark with water and mix.

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

5.10 Nickel chloride solution

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

5.11 Nitric acid, ρ = 1,4 g/ml

5.12 Aqua regia

Mix 3 part volumes of hydrochloric acid (5.2) with 1 part volume of nitric acid (5.11).

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 (5.12) prior to use.

6.2 Specific equipment

In addition to standard laboratory apparatus, an atomic absorption spectrometer, equipped with a premix burner, with facilities for using the oxidizer-fuel combination of nitrous oxide-acetylene shall be used.

NOTE 1 Excitation sources should be operated in accordance with the manufacturer's recommendations. The optical path length within the flame should be between 5 cm to 10 cm.

NOTE 2 Organic solutions should be operated in accordance with the manufacturer's recommendations.

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) in a 250 ml beaker fitted with a watch-glass, add 20 ml of water and dissolve by carefully adding 65 ml of hydrochloric acid (5.2). After cooling, oxidize and complete the dissolution by adding 2 ml of hydrogen peroxide (5.3), drop by drop. Do not heat to avoid the loss of $SnCl_4$.

8.2.2 Transfer quantitatively to a 250 ml separatory funnel, rinsing with about 15 ml of water. Add 80 ml of water. Mix well and allow to cool to room temperature.

8.2.3 Add 20 ml of T.O.P.O.-solution (5.5) and shake for 1 minute.

8.2.4 After clear separation of the phases, discard the aqueous phase completely.

8.2.5 Collect the organic phase quantitatively in 25 ml volumetric flask, rinsing with exactly 5 ml of iso-butyl acetate (5.4) and mix.

8.3 Preparation of the calibration solutions

8.3.1 Into each of six 250 ml separatory funnels, introduce 50 ml of zinc solution (5.7), 30 ml of hydrochloric acid (5.2), 2 ml of hydrogen peroxide (5.3) and 100 ml of water. Mix and allow to cool to room temperature.

8.3.2 Than introduce 0,00 ml, 1,00 ml, 2,00 ml, 4,00 ml, 6,00 ml and 10,00 ml of tin standard solution (5.9), corresponding to tin contents (mass fractions) in the test portion of 0,000 0 %; 0,000 5 %; 0,001 0 %; 0,002 %; 0,003 0 % and 0,005 0 %.

8.3.3 Continue as described in 8.2.3 to 8.2.5.

8.4 Spectrometric measurements

Measure the absorbances of the calibration solutions and the test solution(s) by taking alternate readings to ensure that the settings of the burner and of the apparatus do not change during the readings.

The wavelength of the line used shall be 286,3 nm.

To comply with the concentration ranges recommended by the manufacturer of the apparatus, the same dilutions for the calibration solutions and the test solution(s) shall be made if necessary.

NOTE Between each passage of organic solution, nebulize some iso-butyl acetate (5.4) for a few seconds, in order to clean the burner head.

9 Calculation and expression of results

9.1 Method of calculation

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

Determine from the measured absorbances of the test solutions the associated amount of tin from the calibration graph. If a number of determinations are carried out then a mean of all results shall be determined by adding the individual results together and by dividing by the number of individual results.

The results shall be expressed as specified in EN 988, EN 1179, EN 1774 and EN 12844.

9.2 Precision

A planned trial of this method was carried out by 10 laboratories, using 3 samples with 3 levels of tin contents, each laboratory making three determinations of tin 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 5725-1; i.e. one operator, same apparatus, identical operating conditions, same calibration 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 a different calibration.

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.

The data obtained showed a logarithmic relationship between the tin content and the repeatability limit (r) and reproducibility limits (R_w and R) of the test 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.