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Aluminium and its alloys — Determination of zinc — Atomic absorption method

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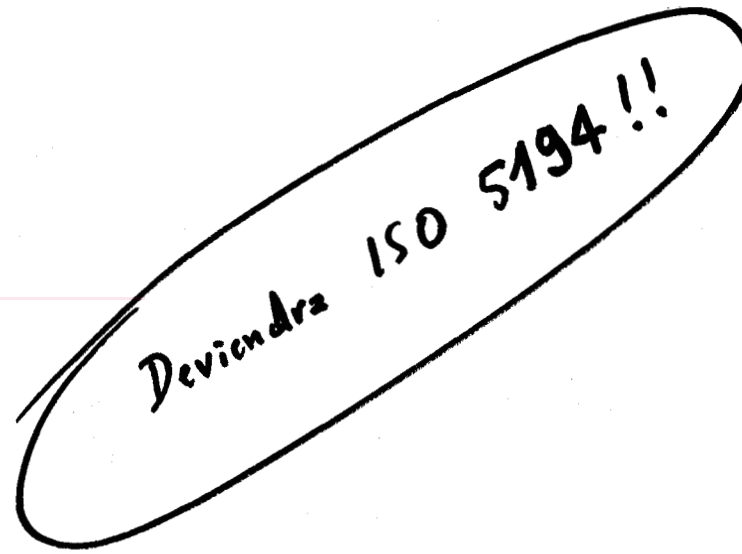

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

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No Member Body expressed disapproval of the document.

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Aluminium and its alloys — Determination of zinc — Atomic absorption method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of zinc in aluminium and its alloys by atomic absorption.

This method is applicable to the determination of zinc contents between 0,002 and 0,2 %. .

2 PRINCIPLE

Dissolution of a test portion in hydrochloric acid. Atomizing the solution in an acetylene-air flame. Determination of zinc by photometric measurement of the absorption of the 213,8 nm line emitted by a zinc hollow cathode lamp.

3 REAGENTS

During the analysis, use only water doubly distilled in borosilicate glassware with ground joints, or water of equivalent purity. Avoid using lead glass.

3.1 Aluminium, extra pure (99,99 % purity), not containing zinc.

The product shall be in chips obtained by milling or drilling.

NOTE — Immediately before use, clean the chips in a little nitric acid (ρ approximately 1,40 g/ml); solution approximately 67 % (*m/m*). Wash the cleaned chips with water then dry them by washing with acetone.

3.2 Hydrochloric acid (ρ approximately 1,1 g/ml); solution approximately 20,4 % (*m/m*).

Dilute 500 ml of hydrochloric acid (ρ approximately 1,19 g/ml), solution approximately 38 % (*m/m*), with water, make up the volume to 1 000 ml and mix.

3.3 Hydrogen peroxide (ρ approximately 1,11 g/ml); solution approximately 30 % (*m/m*).

3.4 Sulphuric acid (ρ approximately 1,48 g/ml); solution approximately 58 % (*m/m*).

Add cautiously 50 ml of sulphuric acid (ρ approximately 1,84 g/ml), solution approximately 95 % (*m/m*), to about 40 ml of water.

After cooling, make up the volume to 100 ml and mix.

3.5 Hydrofluoric acid (ρ approximately 1,13 g/ml); solution approximately 40 % (*m/m*).

3.6 Nitric acid (ρ approximately 1,40 g/ml); solution approximately 67 % (*m/m*).

3.7 Aluminium, acid solution containing 40 g of Al per litre (basic solution).

Weigh, to the nearest 10 mg, 40,0 g of extra pure aluminium (3.1), previously cleaned, and transfer to a beaker of convenient size (for example 2 000 ml). Add, in small portions, 100 ml of hydrochloric acid (3.2), then introduce a drop of metallic mercury to facilitate the attack. Heat gently to start the reaction, then add in small portions a further 400 ml of hydrochloric acid (3.2). Wait until the reaction has subsided, then add gradually 250 ml of hydrochloric acid (ρ approximately 1,19 g/ml) solution approximately 38 % (*m/m*) and heat gently, if necessary, to complete the reaction. Then add a few millilitres of hydrogen peroxide (3.3) and boil for a few minutes to eliminate excess hydrogen peroxide.

Allow to cool, transfer the solution quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

3.8 Zinc standard solution, containing 1 g of Zn per litre.

Prepare this standard solution by one of the following procedures :

3.8.1 Weigh, to the nearest 0,1 mg, 1,000 g of extra pure zinc (99,99 % purity), transfer to a beaker of convenient size (for example 400 ml) and dissolve with 25 ml of hydrochloric acid (3.2). Dilute the solution and transfer quantitatively to a 1 000 ml volumetric flask. Make up to volume and mix.

1 ml of this standard solution contains 1 mg of Zn.

3.8.2 Weigh, to the nearest 0,1 mg, 1,26 g of zinc oxide (ZnO), previously ignited at 1 000 °C for 1 h and cooled in a desiccator. Transfer to a beaker of convenient size (for example 400 ml) and dissolve with 25 ml of hydrochloric acid (3.2). Dilute the solution and transfer quantitatively to a 1 000 ml volumetric flask. Make up to volume and mix.

1 ml of this standard solution contains 1 mg of Zn.

3.9 Zinc standard solution, containing 0,100 g of Zn per litre.

Transfer 100,0 ml of standard solution (3.8) to a 1 000 ml volumetric flask, make up to volume and mix.

1 ml of this standard solution contains 0,10 mg of Zn.

3.10 Zinc standard solution, containing 0,010 g of Zn per litre.

Transfer 100,0 ml of standard solution (3.9) to a 1 000 ml volumetric flask, make up to volume and mix.

1 ml of this standard solution contains 0,010 mg of Zn.

4 APPARATUS

4.1 Ordinary laboratory apparatus.

NOTE – All glassware, including the reagent flasks, shall be made of borosilicate glass or glass of any other quality which does not release zinc, or of plastics. Do not use rubber stoppers (containing zinc), but exclusively ground glass or plastics stoppers.

4.2 Burette, graduated in 0,05 ml.

4.3 Atomic absorption spectrophotometer fitted with a burner supplied by cylinders of acetylene and compressed air.

4.4 Zinc hollow cathode lamp.

5 SAMPLING

5.1 Laboratory sample[1)]

5.2 Test sample

Chips of thickness less than or equal to 1 mm, obtained by drilling or milling.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 1 mg, 1 g of the test sample (5.2).

6.2 Establishment of calibration curve

6.2.1 *Preparation of standard matching solutions*

6.2.1.1 ZINC CONTENTS BETWEEN 0,002 AND 0,05 %.

Introduce, into a series of eight 100 ml volumetric flasks, 25,0 ml of aluminium acid solution (3.7) then add respectively the volumes of zinc standard solution (3.10) or zinc standard solution (3.9) indicated in Table 1, measured with the burette (4.2).

TABLE 1

Volume of zinc standard solution (3.10)	Corresponding mass of zinc
ml	mg
0*	0
1,0	0,01
5,0	0,05
10,0	0,10
Volume of zinc standard solution (3.9)	
ml	
2,0	0,20
3,0	0,30
4,0	0,40
5,0	0,50

* Blank test of the reagents used for the preparation of the calibration curve.

Make up to volume and mix.

NOTE – For zinc contents between 0,002 and 0,01 %, it is advisable to restrict the calibration curve to the first four dilutions. In this case, for the photometric measurements, use a suitable scale expansion.

6.2.1.2 ZINC CONTENTS BETWEEN 0,05 AND 0,2 %

Introduce, into a series of six 100 ml volumetric flasks, 5,0 ml of aluminium acid solution (3.7) then add respectively the volumes of zinc standard solution (3.9) indicated in Table 2, measured with the burette (4.2).

TABLE 2

Volume of zinc standard solution (3.9)	Corresponding mass of zinc
ml	mg
0*	0
1,0	0,1
2,0	0,2
3,0	0,3
4,0	0,4
5,0	0,5

* Blank test of reagents.

Make up to volume and mix.

1) The sampling of aluminium and its alloys will form the subject of a future International Standard.

6.2.2 *Photometric measurements*

6.2.2.1 ADJUSTMENT OF APPARATUS FITTED WITH ZINC HOLLOW CATHODE LAMP (4.4)

Put the apparatus (4.3) under pressure for the time required to stabilize it. Adjust the wavelength to about 213,8 nm as well as the sensitivity and the slit width according to the characteristics of the apparatus. Adjust the pressure of the air and acetylene in accordance with the characteristics of the atomizer-burner so that a clear non-luminescent oxidizing flame is obtained.

6.2.2.2 PHOTOMETRIC MEASUREMENTS

Atomize in the flame the series of control solutions (6.2.1) and for each one measure the intensity of the unabsorbed radiation. Take care that the quantity of the solution atomized in the flame remains constant per unit of time during the whole operation of establishing the calibration curve.

NOTE – Purge with water after each measurement.

6.2.3 *Plotting of the curve*

Plot a graph on millimetre paper placing, for example, on the abscissa the values, expressed in milligrams, of the quantities of zinc contained in 100 ml of the standard matching solutions and on the ordinate the values corresponding to the intensities measured, less the value measured for the zero term of the standard matching solutions (blank test of the reagents of the calibration curve).

6.3 Determination

6.3.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a beaker of convenient size (for example 400 ml), cover with a watch glass and add, in small portions 25,ml of hydrochloric acid (3.2); if necessary, heat gently to complete the dissolution. Add a few millilitres of hydrogen peroxide solution (3.3), then heat to eliminate the excess of hydrogen peroxide and complete the preparation of the test solution, filtering if necessary, according to 6.3.1.1 or 6.3.1.2 depending on the zinc content to be determined.

NOTES

1 In the case of products which are difficult to attack with hydrochloric acid, add a drop of metallic mercury.

2 For silicon contents greater than 1 %, proceed as follows :

Transfer the filter containing the silicon dioxide to a platinum crucible and dry. Ignite the filter, then heat at about 800 to 1 000 °C for about 15 min. Allow to cool, then add to the crucible 2 ml of sulphuric acid (3.4), 5 ml of hydrofluoric acid (3.5) and, drop by drop, about 1 ml of nitric acid (3.6). Heat gently, then, when the acids have evaporated, heat again at 800 to 1 000 °C for a few minutes. Allow to cool, add to the crucible a few millilitres of hydrochloric acid (3.2) and heat, if necessary, to complete the dissolution of the residue; filter, if necessary. Add this solution quantitatively to the test solution.

6.3.1.1 ZINC CONTENTS BETWEEN 0,002 AND 0,05 %

Transfer the test solution (6.3.1) quantitatively to a 100 ml volumetric flask. Cool, make up to volume and mix.

6.3.1.2 ZINC CONTENTS BETWEEN 0,05 AND 0,2 %

Transfer the test solution (6.3.1) quantitatively to a 500 ml volumetric flask. Cool, make up to volume and mix.

6.3.2 *Blank test*

Carry out in parallel a blank test following the same procedure and using the same quantities of all the reagents as are used for the determination, replacing the test portion by 1 g, weighed to the nearest 1 mg, of extra pure aluminium (3.1).

6.3.3 *Photometric measurements*

Carry out the measurements relating to the test solution (6.3.1.1 or 6.3.1.2), to the blank test solution (6.3.2) and to the standard matching solutions specified in 6.2.1.1 or 6.2.1.2 according to the method described in 6.2.2.2.

Take care to make the measurements of the test solution between those of two standard matching solutions containing quantities of zinc as close as possible to the quantity being determined.

7 EXPRESSION OF RESULTS

By means of the calibration curve (see 6.2.3), determine the quantities of zinc corresponding to the intensities measured for the test solution (6.3.1.1 or 6.3.1.2) and for the blank test solution (6.3.2).

The zinc (Zn) content is given, as a percentage by mass, by the formula :

$$\frac{(m_1 - m_2) \times V}{m_0 \times 10 \times V_1}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in milligrams, of zinc found in the test solution;

m_2 is the mass, in milligrams, of zinc found in the blank test solution;

V is the volume, in millilitres, of the test solution;

V_1 is the volume, in millilitres, of the control solutions.

8 TEST REPORT

The test report shall include the following information :

a) the reference of the method used;

b) the results, as well as the form in which they are expressed;

c) any particular details noted in the course of the test;

d) any operations not specified in this International Standard or any optional operations which may have affected the results.

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