International Standard 5194

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Aluminium and aluminium alloys — Determination of zinc content — Flame atomic absorption spectrometric method

Aluminium et alliages d'aluminium — Dosage du zinc — Méthode par spectrométrie d'absorption atomique dans la flamme

First edition — 1981-11-15

UDC 669.71 : 543.422 : 546.47 **Ref. No. ISO 5194-1981 (E)**

Descriptors : aluminium, aluminium alloys, chemical analysis, determination of content, zinc, atomic absorption spectroscopic method.

Foreword

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International Standard ISO 5194 was developed by Technical Committee ISO/TC 79, *Light metals and their alloys*, and was circulated to the member bodies in December 1980.

It has been approved by the member bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
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The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

Printed in Switzerland

Aluminium and aluminium alloys — Determination of zinc content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the zinc content of aluminium and aluminium alloys.

The method is applicable to products having zinc (Zn) contents between 0,002 and 6 % (*m/m*).

2 Principle

Dissolution of a test portion in hydrochloric acid and hydrogen peroxide. Aspiration of the solution into an air-acetylene flame and comparison of the absorbance of resonance energy by zinc in the test solution (wavelength of 213,9 nm normally) with that of standard solutions.

3 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled or deionized water.

3.1 Aluminium, extra pure (purity 99,99 %), free from zinc.

3.2 Hydrochloric acid, ϱ approximately 1,10 g/ml, 20 % (*m/m*) or approximately 6 mol/l solution.

Dilute 500 ml of hydrochloric acid, ϱ approximately 1,19 g/ml, 38 % (*m/m*) or approximately 12 mol/l solution, with 500 ml of water.

3.3 Hydrogen peroxide, about 30 % (*m/m*) solution.

3.4 Sulphuric acid, ϱ approximately 1,48 g/ml, 58 % (*m/m*) or approximately 9 mol/l solution.

While stirring and cooling, add 50 ml of sulphuric acid, ϱ approximately 1,84 g/ml, 96 % (*m/m*) or approximately 18 mol/l solution, to 40 ml of water. Cool again, dilute to the mark in a 100 ml volumetric flask, and mix.

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

3.6 Nitric acid, ϱ approximately 1,4 g/ml, 68 % (*m/m*) or approximately 15 mol/l solution.

3.7 Aluminium, 20 g/l solution.

Weigh, to the nearest 0,01 g, 20 g of previously pickled extra pure aluminium (3.1), place it in a 1 000 ml beaker and cover with a watch glass. Add, in small portions, 600 ml of the hydrochloric acid solution (3.2) and, if necessary, a drop of metallic mercury to assist the attack. If necessary, warm gently to aid the dissolution, then add a few drops of the hydrogen peroxide solution (3.3) and boil for a few minutes to remove the excess hydrogen peroxide. After cooling, quantitatively transfer to a 1 000 ml volumetric flask, dilute to the mark and mix.

3.8 Aluminium, 2 g/l solution.

Transfer 50,0 ml of the aluminium solution (3.7) to a 500 ml volumetric flask, add 270 ml of the hydrochloric acid solution (3.2) dilute to the mark and mix.

3.9 Zinc, standard solution corresponding to 1 g of Zn per litre.

Weigh, to the nearest 0,001 g, 1 g of zinc (purity ⩾ 99,99 %), transfer it to a 400 ml beaker and cover with a watch glass. Add, in small portions, 50 ml of the hydrochloric acid solution (3.2) and heat gently, if necessary, to complete the dissolution. After cooling, quantitatively transfer to a 1 000 ml volumetric flask, dilute to the mark and mix.

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

3.10 Zinc, standard solution corresponding to 0,05 g of Zn per litre.

Transfer 50,0 ml of the standard zinc solution (3.9) to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,05 mg of zinc.

3.11 Zinc, standard solution corresponding to 0,01 g of Zn per litre.

Transfer 10,0 ml of the standard zinc solution (3.9) to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,01 mg of Zn.

4 Apparatus

Normal laboratory apparatus and

4.1 Burette, graduated in 0,05 ml.

4.2 Atomic absorption spectrometer, fitted with an air-acetylene burner.

4.3 Compressed air (laboratory installation or gas cylinders).

4.4 Acetylene, in gas cylinders.

4.5 Zinc hollow-cathode lamp.

5 Sampling

5.1 Laboratory sample[1)]

5.2 Test sample

Use chips, not more than 1 mm thick, obtained by milling or drilling.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample (5.2).

6.2 Preparation of the calibration curves

6.2.1 Preparation of the standard solutions

6.2.1.1 Zinc contents between 0,002 and 0,03 % (m/m)

Into a series of nine 100 ml volumetric flasks, introduce, by means of the burette (4.1), the volumes of standard zinc solutions (3.10 and 3.11) indicated in table 1. Add to each flask, 50 ml of the aluminium solution (3.7), dilute to the mark and mix.

Table 1

Standard zinc solution (3.11)	Mass of zinc contained	Zinc content
ml	mg	% (m/m)
0*	0	0
1,0	0,01	0,001
3,0	0,03	0,003
5,0	0,05	0,005
10,0	0,10	0,010
Standard zinc solution (3.10)		
ml		
3,0	0,15	0,015
4,0	0,20	0,020
5,0	0,25	0,025
6,0	0,30	0,030

* Blank test of calibration curve reagents.

6.2.1.2 Zinc contents between 0,02 and 0,3 % (m/m)

Into a series of nine 100 ml volumetric flasks, introduce, by means of the burette (4.1), the volumes of standard zinc solutions (3.10 and 3.11) indicated in table 2. Add to each flask, 5 ml of the aluminium solution (3.7), dilute to the mark and mix.

Table 2

Standard zinc solution (3.11)	Mass of zinc contained	Zinc content
ml	mg	% (m/m)
0*	0	0
1,0	0,01	0,01
3,0	0,03	0,03
5,0	0,05	0,05
10,0	0,10	0,10
Standard zinc solution (3.10)		
ml		
3,0	0,15	0,15
4,0	0,20	0,20
5,0	0,25	0,25
6,0	0,30	0,30

* Blank test of calibration curve reagents.

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

6.2.1.3 Zinc contents between 0,2 and 3 % (m/m)

Into a series of nine 100 ml volumetric flasks, introduce, by means of the burette (4.1), the volumes of standard zinc solutions (3.10 and 3.11) indicated in table 3. Add to each flask, 5 ml of the aluminium solution (3.8), dilute to the mark and mix.

Table 3

Standard zinc solution (3.11)	Mass of zinc contained	Zinc content
ml	mg	% (m/m)
0*	0	0
1,0	0,01	0,10
3,0	0,03	0,30
5,0	0,05	0,50
10,0	0,10	1,00
Standard zinc solution (3.10)		
ml		
3,0	0,15	1,50
4,0	0,20	2,00
5,0	0,25	2,50
6,0	0,30	3,00

* Blank test of calibration curve reagents.

6.2.1.4 Zinc contents between 3 and 6 % (m/m)

Into a series of six 100 ml volumetric flasks, introduce, by means of the burette (4.1), the volumes of standard zinc solution (3.10) indicated in table 4. Add to each flask, 2,5 ml of the aluminium solution (3.8), dilute to the mark and mix.

Table 4

Standard zinc solution (3.10)	Mass of zinc contained	Zinc content
ml	mg	% (m/m)
0*	0	0
2,0	0,10	2,0
3,0	0,15	3,0
4,0	0,20	4,0
5,0	0,25	5,0
6,0	0,30	6,0

* Blank test of calibration curve reagents.

6.2.2 Spectrometric measurements

Switch on the spectrometer (4.2), fitted with the zinc hollow-cathode lamp (4.5), sufficiently in advance to allow it to stabilize. Adjust the wavelength to about 213,9 nm, and the sensitivity and the slot according to the characteristics of the apparatus. Adjust the pressure of the air and the acetylene according to the characteristics of the aspirator-burner, so as to obtain a clear, non-luminous, oxidizing flame.

Aspirate the standard solutions (6.2.1.1, 6.2.1.2, 6.2.1.3 or 6.2.1.4) into the flame and measure their absorbances. Take care to ensure that the volume of standard solutions aspirated per unit time into the flame is kept constant throughout the procedure for preparation of the calibration curve.

NOTE — Aspirate water into the burner after each measurement.

6.2.3 Plotting the graphs

Plot graphs, having, for example, the masses, in milligrams, of zinc contained in 100 ml of the standard solutions as abscissae and the corresponding values of absorbance, corrected for the blank test of the calibration curve reagents (zero term), as ordinates.

6.3 Determination

6.3.1 Preparation of the test solution

Place the test portion (6.1) in a 250 ml beaker and cover with a watch glass. Add about 30 to 40 ml of water, and then in small portions, 30 ml of the hydrochloric acid solution (3.2), warming gently, if necessary, in order to complete the dissolution. Add a few drops of the hydrogen peroxide solution (3.3) and boil for 10 min to remove the excess hydrogen peroxide.

If undissolved matter remains, indicating the presence of silicon, filter the solution, transfer the filter paper and undissolved matter to a platinum crucible and incinerate, taking care that it does not inflame. Calcine at about 550 °C. After cooling, add 2 ml of the sulphuric acid solution (3.4), 5 ml of the hydrofluoric acid solution (3.5) and, drop by drop, the nitric acid solution (3.6) in such a manner as to obtain a clear solution (about 1 ml). Evaporate to dryness and calcine again, at about 700 °C, for a few minutes, to completely volatilize the silicon. After cooling, bring the non-volatile matter into solution with the least possible quantity of the hydrochloric acid solution (3.2), filter, if necessary, and quantitatively add this filtrate to that already obtained.

6.3.1.1 Zinc contents between 0,002 and 0,03 % (m/m)

Quantitatively transfer the solution (6.3.1) to a 100 ml volumetric flask, dilute to the mark and mix.

Use calibration curve 6.2.1.1 for this solution.

6.3.1.2 Zinc contents between 0,02 and 0,3 % (m/m)

Quantitatively transfer the solution (6.3.1) to a 1 000 ml volumetric flask, dilute to the mark and mix.

Use calibration curve 6.2.1.2 for this solution.

6.3.1.3 Zinc contents between 0,2 and 3 % (m/m)

Quantitatively transfer the solution (6.3.1) to a 500 ml volumetric flask, dilute to the mark and mix. Then transfer 50,0 ml of this solution to a 1 000 ml volumetric flask, add 27 ml of the hydrochloric acid solution (3.2), dilute to the mark and mix.

Use calibration curve 6.2.1.3 for this solution.

6.3.1.4 Zinc contents between 3 and 6 % (m/m)

Quantitatively transfer the solution (6.3.1) to a 1 000 ml volumetric flask, dilute to the mark and mix. Then transfer 50,0 ml of this solution to another 1 000 ml volumetric flask, add 13,5 ml of the hydrochloric acid solution (3.2), dilute to the mark and mix.

Use calibration curve 6.2.1.4 for this solution.

6.3.2 Blank test

Carry out a blank test, in parallel with the analysis, using the same procedure and the same quantities of all reagents used in the determination, but replacing the test portion by 1 g, weighed to the nearest 0,001 g, of the extra-pure aluminium (3.1).

6.3.3 Spectrometric measurements

Measure the absorbances of the test solution (6.3.1.1, 6.3.1.2, 6.3.1.3 or 6.3.1.4), the blank test solution (6.3.2), and the appropriate standard solutions (6.2.1.1, 6.2.1.2, 6.2.1.3 or 6.2.1.4), proceeding as specified in 6.2.2, and taking care to bracket the measurement of the absorbance of the test solution and of the blank test solution between two standard solutions having zinc contents as close as possible, respectively, to that to be determined.

7 Expression of results

By means of the calibration curves, determine the quantity of zinc corresponding to the spectrometric measurements of the test solution and of the blank test solution.

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

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

where

m_0 is the mass, in grams, of the test portion (i.e. 1 g);

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

m_2 is the mass, in miligrams, of zinc found in the test portion (totality or aliquot) used for the spectrometric measurement;

R is the ratio between the dilution-volume of the test solution and the volume of the standard solutions for calibration ($R = 1$ for test solutions prepared in accordance with 6.3.1.1, $R = 10$ for test solutions prepared in accordance with 6.3.1.2, $R = 100$ for test solutions prepared in accordance with 6.3.1.3 and $R = 200$ for test solutions prepared in accordance with 6.3.1.4).

8 Test report

The test report shall include the following particulars :

a) identification of the test sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard, or regarded as optional.