International Standard 4192

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

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

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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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Australia
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Aluminium and aluminium alloys — Determination of lead 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 lead content of aluminium and its alloys.

The method is applicable to products having lead (Pb) contents between 0,01 and 1,5 % (*m*/*m*).

2 Principle

Dissolution of a test portion in a mixture of hydrochloric acid and nitric acid. Aspiration of the solution into an air-acetylene flame and comparison of the absorbance of resonance energy of lead by the test solution (wavelength of 217,0 or 283,3 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 lead.

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

3.3 Hydrochloric acid/nitric acid mixture.

Cautiously mix together 500 ml of water, 375 ml of the hydrochloric acid solution, [ϱ approximately 1,19 g/ml, 38 % (*m*/*m*)], and 125 ml of the nitric acid solution (3.2).

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

3.5 Aluminium, 20 g/l solution.

Weigh, to the nearest 0,01 g, 10 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, 200 ml of the hydrochloric acid/nitric acid mixture (3.3) and, if necessary, a drop of metallic mercury to assist the attack. If necessary, warm gently to aid the dissolution and, when the aluminium has dissolved, boil until brown fumes have been expelled and the volume of the solution has been reduced to about 100 ml. After cooling, quantitatively transfer the solution to a 500 ml volumetric flask, dilute to the mark and mix.

3.6 Lead, standard solution corresponding to 1 g of Pb per litre.

Weigh, to the nearest 0,001 g, 1 g of lead (purity ⩾ 99,99 %), transfer it to a 250 ml beaker and cover with a watch glass. Add the minimum amount (10 ml, for instance) of the nitric acid solution (3.2) and warm gently until all the lead has dissolved. Then boil for a few minutes to remove nitrous fumes and, after cooling, transfer to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 1 mg of lead.

3.7 Lead, standard solution corresponding to 0,1 g of Pb per litre.

Transfer 100,0 ml of the standard lead solution (3.6) to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,1 mg of lead.

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 Lead 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 Lead contents between 0,01 and 0,10 % (*m*/*m*)

Into a series of six 100 ml volumetric flasks, introduce the volumes of the standard lead solution (3.7) shown in table 1, using the burette (4.1). Add to each flask 50 ml of the aluminium solution (3.5) and 5 ml of the nitric acid solution (3.2). Dilute to the mark and mix.

Table 1

Standard lead solution (3.7)	Corresponding mass of lead	Lead content
ml	mg	% (*m*/*m*)
0*	0	0
1	0,1	0,01
3	0,3	0,03
5	0,5	0,05
7	0,7	0,07
10	1,0	0,10

* Blank test of calibration curve reagents.

6.2.1.2 Lead contents between 0,10 and 1,5 % (*m*/*m*)

Into a series of eight 100 ml volumetric flasks, introduce the volumes of the standard lead solutions (3.7 and 3.6) shown in table 2, using the burette (4.1). Add to each flask 10 ml of the aluminium solution (3.5) and 5 ml of the nitric acid solution (3.2). Dilute to the mark and mix.

Table 2

Standard lead solution (3.7)	Corresponding mass of lead	Lead content
ml	mg	% (*m*/*m*)
0*	0	0
2,0	0,2	0,10
6,0	0,6	0,30
10,0	1,0	0,50
Standard lead solution (3.6)		
ml		
1,5	1,5	0,75
2,0	2,0	1,00
2,5	2,5	1,25
3,0	3,0	1,50

* Blank test of calibration curve reagents.

6.2.2 Spectrometric measurements

Switch on the spectrometer (4.2), fitted with the lead hollow cathode lamp (4.5), sufficiently in advance to allow it to stabilize. Adjust the wavelength to about 217,0 or 283,3 nm and the sensitivity and the slot according to the characteristics of the apparatus. Adjust the pressure of the air and 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 and 6.2.1.2) 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.

NOTES

1 Aspirate water into the burner after each measurement.

2 If necessary, appropriately amplify the signal of the spectrometer.

6.2.3 Plotting the graph

Plot a graph having, for example, the masses, in milligrams, of lead 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.

[1)] The sampling of aluminium and aluminium alloys will be the subject of a future International Standard.

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, in small portions, 20 ml of the hydrochloric acid/nitric acid mixture (3.3) and warm gently, if necessary, to complete the dissolution. With the beaker covered, evaporate the solution to about 10 ml and allow to cool. Take up the residue with 25 ml of water and boil in order to complete the dissolution of the salts.

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

6.3.1.1 Lead contents between 0,01 and 0,10 % (*m/m*)

Quantitatively transfer the solution (6.3.1) to a 100 ml volumetric flask, add 5 ml of the nitric acid solution (3.2), dilute to the mark and mix.

Use calibration curve 6.2.1.1 for this solution.

6.3.1.2 Lead contents between 0,10 and 1,5 % (*m/m*)

Quantitatively transfer the solution (6.3.1) to a 500 ml volumetric flask, add 25 ml of the nitric acid solution (3.2), dilute to the mark and mix.

Use calibration curve 6.2.1.2 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 (6.1) by 0,5 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 or 6.3.1.2), the blank test solution (6.3.2), and the appropriate standard solutions (6.2.1.1 or 6.2.1.2), proceeding as specified in 6.2.2, and taking care to bracket the measurements of absorbance of the test solution and of the blank test solution between two standard solutions having lead 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 lead corresponding to the spectrometric measurements of the test solution and of the blank test solution.

The lead, Pb, content, expressed as a percentage by mass, is given by the formula

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

where

m is the mass, in milligrams, of lead found in the test solution;

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

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

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, and $R = 5$ for test solutions prepared in accordance with 6.3.1.2).

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 included in this International Standard or regarded as optional.

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