International Standard 6827

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Aluminium and aluminium alloys — Determination of titanium content — Diantipyrylmethane photometric method

Aluminium et alliages d'aluminium — Dosage du titane — Méthode photométrique au diantipyrylméthane

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Foreword

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Aluminium and aluminium alloys — Determination of titanium content — Diantipyrylmethane photometric method

1 Scope and field of application

This International Standard specifies a diantipyrylmethane photometric method for the determination of the titanium content of aluminium and aluminium alloys.

The method is applicable to products having titanium contents between 0,003 and 0,3 % *(m/m)*.

2 Principle

Dissolution of a test portion in hydrochloric acid, reduction of the iron(III) and vanadium(V) interferences by the addition of ascorbic acid in the presence of copper(II) sulphate, and addition of diantipyrylmethane to form the yellow titanium complex. Photometric measurement of the complex at a wavelength of about 400 nm.

3 Reagents

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

3.1 Hydrochloric acid, ϱ approximately 1,1 g/ml, about 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.

3.2 Sulphuric acid, ϱ approximately 1,84 g/ml, 96 % *(m/m)* or approximately 18 mol/l solution.

3.3 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 the sulphuric acid solution (3.2) to 40 ml of water. Cool again, then dilute to 100 ml and mix.

3.4 Nitric acid, ϱ approximately 1,40 g/ml, 68 % *(m/m)* or approximately 15 mol/l solution.

3.5 Hydrogen peroxide, approximately 30 % *(m/m)* solution.

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

3.7 Potassium permanganate, 1 g/l solution.

Dissolve 1 g of potassium permanganate ($KMnO_4$) in water, dilute to 1 000 ml and mix.

3.8 Copper(II) sulphate, 50 g/l solution.

Dissolve 5 g of anhydrous copper(II) sulphate ($CuSO_4$) in water, dilute to 100 ml and mix.

3.9 Ascorbic acid, 20 g/l solution.

Prepare just before use.

Dissolve 2 g of ascorbic acid ($C_6H_8O_6$) in water, dilute to 100 ml and mix.

3.10 Diantipyrylmethane, 50 g/l solution in approximately 0,1 mol/l hydrochloric acid solution.

Dissolve 5 g of diantipyrylmethane in approximately 17 ml of the hydrochloric acid solution (3.1) and 70 ml of water, dilute to 100 ml with water and mix.

3.11 Aluminium, 10 g/l solution.

Weigh, to the nearest 0,01 g, 10 g of previously pickled aluminium (purity ⩾ 99,99 %) free from titanium, transfer it to a 1 000 ml beaker and cover with a watch glass. Add, in small portions, 300 ml of the hydrochloric acid solution (3.1) and, if necessary, a drop of metallic mercury to assist the attack. If necessary, warm gently to help dissolution, then add a few drops of the hydrogen peroxide solution (3.5) and boil for a few minutes to remove the excess of hydrogen peroxide. Dilute to about 500 ml with water. After cooling, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

50 ml of this solution contain 0,5 g of aluminium.

3.12 Titanium, standard solution corresponding to 0,5 g of titanium per litre.

Prepare this standard solution by one of the following methods.

3.12.1 Weigh, to the nearest 1 mg, 0,5 g of pure titanium (purity ⩾ 99,5 %) and transfer it to a 600 ml beaker. Dissolve it in 125 ml of the sulphuric acid solution (3.2) and oxidize with a few drops of the nitric acid (3.4). Boil the solution gently until all nitric acid fumes have been given off. Cool, dilute suitably, transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,5 mg of titanium.

3.12.2 Weigh, to the nearest 0,1 mg, 1,848 5 g of potassium titanyl oxalate dihydrate $[K_2TiO(C_2O_4)_2.2H_2O]$ and transfer to a 100 ml Kjeldahl flask. Add 1,8 g of ammonium sulphate $(NH_4)_2SO_4$ and 15 ml of the sulphuric acid solution (3.2). Heat gently until the reaction subsides and boil gently for 10 min.

Cool and transfer the solution to a beaker of suitable capacity (for example 250 ml) already containing 100 ml of water. Add a few drops of the potassium permanganate solution (3.7) until a persistent pink colour is obtained. Transfer the solution to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,5 mg of titanium.

3.13 Titanium, standard solution corresponding to 0,01 g of titanium per litre.

Transfer 10 ml of the standard titanium solution (3.12) to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

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

4 Apparatus

Normal laboratory apparatus, and

4.1 Spectrophotometer, or

4.2 Electrophotometer.

5 Sampling

5.1 Laboratory sample[1]

5.2 Test sample

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

6 Procedure

6.1 Test portion

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

6.2 Preparation of the calibration graph

6.2.1 Preparation of standard matching solutions, related to photometric measurements carried out in cells of thickness 1 cm

Into a series of seven 100 ml one-mark volumetric flasks, introduce the volumes of standard titanium solution (3.13) indicated in table 1.

Table 1

Standard titanium solution (3.13)	Corresponding mass of titanium
ml	mg
0*	0
1,5	0,015
3	0,030
5	0,050
8	0,080
12	0,120
16	0,160

* Compensation solution.

Add, to each flask, 50 ml of the aluminium solution (3.11).

6.2.2 Colour reaction

Add 25 ml of the sulphuric acid solution (3.3), dilute to approximately 60 to 70 ml, add 2 drops of the copper(II) sulphate solution (3.8) and 2 ml of the ascorbic acid solution (3.9) and mix.

Finally, add 10 ml of the diantipyrylmethane solution (3.10), dilute to the mark with water and mix.

6.2.3 Photometric measurements

Measure the absorbances of the standard matching solutions after 30 min, using the spectrophotometer (4.1) at the maximum of the absorption curve (wavelength approximately 400 nm), or the electrophotometer (4.2) fitted with suitable filters, after having first adjusted the apparatus to zero absorbance against the compensation solution.

6.2.4 Plotting of the calibration curve

Plot a graph having, for example, the masses of titanium (Ti), in milligrams, contained in 100 ml of standard matching solution as abscissae and the corresponding values of absorbance as ordinates.

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

6.3 Determination

6.3.1 Preparation of the test solution

Transfer the test portion (6.1) to a 250 ml beaker and cover it with a watch glass. Add 30 ml of water and then, in small portions, 30 ml of the hydrochloric acid solution (3.1) and, drop by drop, 3 ml of the hydrogen peroxide solution (3.5) (see 9.1). If necessary, warm gently to start the reaction. As soon as the attack is completed, boil for 10 min and rinse the watch glass and the walls of the beaker with a little warm water.

If undissolved matter remains, which denotes the presence of silicon, filter, place the filter in a platinum crucible and incinerate, taking care that it does not inflame. Then calcine at about 550 °C.

After cooling, add 2 ml of the sulphuric acid solution (3.2), 5 ml of the hydrofluoric acid solution (3.6) and, drop by drop, some nitric acid solution (3.4) in such a manner as to obtain a limpid 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 (3.1), filter if necessary, and quantitatively add this filtrate to that previously obtained. Evaporate the solution in the beaker to reduce the volume to not more than about 50 ml. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

According to the expected titanium content, proceed using the volumes of test solution and standard aluminium solution (3.11) indicated in table 2.

Table 2

Expected titanium content	Volume of test solution	Quantity of titanium present	Volume of standard aluminium solution (3.11)
% (*m*/*m*)	ml	mg	ml
0,003 to 0,03	50,0	0,015 to 0,15	—
0,03 to 0,08	20,0	0,06 to 0,16	30,0
0,08 to 0,30	5,0	0,04 to 0,15	45,0

6.3.2 Colour reaction

Place in a 100 ml one-mark volumetric flask, using a pipette, the appropriate volumes of test solution and standard aluminium solution (3.11) according to table 2, and continue as specified in 6.2.2 (see 8.2).

6.3.3 Reference solution

Place in a 100 ml one-mark volumetric flask, using a pipette, the appropriate volumes of test solution and standard aluminium solution (3.11) according to table 2, and continue as specified in 6.2.2, but omitting the diantipyrylmethane (see 8.3).

6.3.4 Photometric measurements

Measure the absorbance of the solution (6.3.2) after 30 min, using the spectrophotometer (4.1) at the maximum of the absorption curve (wavelength approximately 400 nm), or the electrophotometer (4.2) fitted with suitable filters, after having first adjusted the apparatus to zero absorbance against the corresponding reference solution (6.3.3).

7 Expression of results

By means of the calibration curve (see 6.2.4), determine the mass of titanium corresponding to the absorbance of the treated test solution.

The titanium (Ti) content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 \times D}{10 \times m_0}$$

where

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

m_1 is the mass, in milligrams, of titanium found in the aliquot portion of the test solution (6.3.2) measured against the reference solution (6.3.3);

D is the ratio of the volume of the test solution to that of the aliquot portion taken for the colour reaction.

8 Notes on procedure

8.1 Instead of 3 ml of the hydrogen peroxide solution (3.5), 1 ml of the nitric acid solution (3.4) may be used to oxidize the test solution.

8.2 For alloys with relatively high zinc contents, some precipitate may occur in the solution. In this case, filter part of the solution into a dry container before making photometric measurements.

8.3 The corresponding value of absorbance of this "basic" colour should be deducted from the measured absorbance of the test solution in order to avoid interference from elements which form coloured ions in the solution.

9 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 special features noted during the determination;

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

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