INTERNATIONAL STANDARD 2368

G-01-33

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

Aluminium fluoride for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

First edition — 1972-12-15

UDC 661.862.362 : 546.72 : 422

Ref. No. ISO 2368-1972 (E)

Descriptors : aluminium halides, chemical analysis, determination of content, photometry, iron.

Price based on 4 pages

FOREWORD

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International Standard ISO 2368 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in November 1971 by the Member Bodies of the following countries :

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Aluminium fluoride for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

1 SCOPE

This International Standard specifies a photometric method, using 1,10-phenanthroline, for the determination of the iron content of aluminium fluoride for industrial use.

2 FIELD OF APPLICATION

The method is applicable to the determination of iron contents, expressed as Fe_2O_3, greater than 0,020 %, in aluminium fluoride for industrial use.

3 REFERENCE

ISO 2925, *Aluminium fluoride for industrial use — Preparation and storage of test samples.* 1)

4 PRINCIPLE

Dissolution of a test portion by either alkaline or acid fusion.

Preliminary reduction of iron(III) by means of hydroxylammonium chloride.

Formation of the iron(II)-1,10-phenanthroline complex in a buffered medium (pH value between 3,5 and 4,2).

Photometric measurement at a wavelength of about 510 nm.

5 REAGENTS

Distilled water or water of equivalent purity shall be used in the test.

5.1 Sodium carbonate, anhydrous.

5.2 Boric acid (H_3BO_3).

5.3 Potassium pyrosulphate ($K_2S_2O_7$), finely crushed.

5.4 Nitric acid, approximately 8 N solution.

Dilute 540 ml of nitric acid, ρ 1,40 g/ml (approximately 68 % (*m/m*) solution), with water, dilute to 1 000 ml and mix.

5.5 Hydrochloric acid, approximately 6 N solution.

Dilute 515 ml of hydrochloric acid, ρ 1,19 g/ml (approximately 38 % (*m/m*) solution), with water, dilute to 1 000 ml and mix.

5.6 Hydroxylammonium chloride, 10 g/l solution.

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water, dilute to 1 000 ml and mix.

5.7 1,10-phenanthroline hydrochloride, 2,5 g/l solution.

Dissolve 2,5 g of 1,10-phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$) in water, dilute to 1 000 ml and mix.

NOTE — 1,10-phenanthroline hydrochloride monohydrate can be replaced by 1,10-phenanthroline monohydrate. If this product is used, it should be dissolved in 10 ml of ethanol 95 % (*V/V*), before adding water.

5.8 Buffer solution.

Dissolve 272 g of sodium acetate trihydrate in approximately 500 ml of water.

Add 240 ml of glacial acetic acid, approximately 17,4 N, dilute to 1 000 ml and mix.

5.9 Sodium acetate, 500 g/l solution.

Dissolve 50 g of sodium acetate trihydrate in water, dilute to 100 ml and mix.

5.10 Acetic acid, dilute solution.

Dilute 500 ml of glacial acetic acid, approximately 17,4 N, with water, dilute to 1 000 ml and mix.

5.11 Iron standard solution, with an Fe_2O_3 content of 0,200 g/l.

This solution can be prepared by either of the two following methods :

5.11.1 Weigh, to the nearest 1 mg, 0,982 g of iron(II) ammonium sulphate hexahydrate, $[Fe(NH_4)_2.(SO_4)_2.6H_2O]$. Place in a beaker of suitable capacity (100 ml, for example) and dissolve in water.

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1) At present at the stage of draft.

Add 20 ml of sulphuric acid solution, ρ 1,84 g/ml (approximately 96 % (*m/m*) solution), transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

5.11.2 Weigh, to the nearest 1 mg, 0,200 g of iron(III) oxide (Fe_2O_3), previously heated at 600 °C and cooled in a desiccator. Transfer to a beaker of a suitable capacity (100 ml, for example), add 10 ml of hydrochloric acid solution ρ 1,19 g/ml (approximately 38 % (*m/m*) solution) and heat gently to dissolve. Allow to cool, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,200 mg of Fe_2O_3.

5.12 Iron standard solution, with an Fe_2O_3 content of 0,010 g/l.

Transfer 50,0 ml of the standard solution (5.11) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 μg of Fe_2O_3.

The solution shall be prepared immediately before use.

5.13 Indicator paper, covering the pH range 3,5 to 4,2 at intervals of 0,2 unit.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Platinum dish, flat-bottomed, approximately 80 mm in diameter and approximately 35 mm deep, fitted with a platinum lid.

6.2 Electric furnace, capable of being controlled at 550 ± 25°C.

6.3 Electric furnace, capable of being controlled at 750 ± 25 °C.

6.4 Electric furnace, capable of being controlled at 700 ± 25 °C.

6.5 pH meter, fitted with a glass electrode.

6.6 Spectrophotometer, or

6.7 Photoelectric absorptiometer.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 1 mg, 1 g of the dried test sample.[1]

7.2 Blank test

7.2.1 *Alkaline fusion*

Weigh into the platinum dish (6.1), 12 g of the sodium carbonate (5.1) and 4 g of the boric acid (5.2). Mix thoroughly using a platinum spatula. Cover the dish with its lid and place in the electric furnace (6.2), controlled at 550 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support to avoid the risk of contamination. Leave the dish in the furnace until the reaction subsides.

Then transfer the dish to the electric furnace (6.3) controlled at 750 ± 25 °C, again isolating it from the furnace floor, and allow to remain for a maximum of 5 min.

Remove the dish from the furnace and allow to cool in air. Add boiling water to the dish and heat gently until dissolution is complete.

After cooling slightly, transfer the contents of the dish to a beaker of suitable capacity containing 20 ml of the nitric acid solution (5.4). Carefully wash the dish and lid with 18 ml of the nitric acid solution (5.4) and then with hot water, collecting the washings in the beaker; simmer gently for a few minutes until complete dissolution is obtained.

Allow to cool slightly and transfer quantitatively to a one-mark volumetric flask of the same capacity as that used for the preparation of the test solution (7.4.1). After cooling, dilute to the mark and mix.

Alternatively the following procedure may be used :

7.2.2 *Acid fusion*

Weigh into the platinum dish (6.1), 10 g of the potassium pyrosulphate (5.3). Cover the dish with its lid and place in the electric furnace (6.4) controlled at 700 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support to avoid the risk of contamination, and leave at the temperature of 700 ± 25 °C for a maximum of 10 min.

Remove the dish from the furnace and allow to cool in air. Add 10 ml of water and 10 ml of the hydrochloric acid solution (5.5) to the dish. Simmer gently until complete dissolution is obtained, taking care, at the same time, to rinse the lid and walls of the dish with hot water, and collect the washings in the same dish.

Allow to cool slightly and transfer the solution quantitatively to a one-mark volumetric flask of the same capacity as that used for the preparation of the test solution (7.4.1). After cooling, dilute to the mark and mix.

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1) See ISO 2925.

7.3 Preparation of calibration curve

7.3.1 *Preparation of the standard matching solutions* for photometric measurements with a 1 cm cell.

Into each of a series of eight 100 ml one-mark volumetric flasks transfer respectively the volumes of the iron standard solution (5.12) shown in the following table.

Volume of iron standard solution (5.12)	Corresponding mass of Fe_2O_3
ml	µg
0*	0
1,0	10
2,5	25
5,0	50
10,0	100
15,0	150
20,0	200
25,0	250

* Compensation solution.

Add to each flask an amount of water sufficient to dilute to approximately 50 ml, then add 5 ml of the hydroxylammonium chloride solution (5.6), 5 ml of the 1,10-phenanthroline solution (5.7) and 25 ml of the buffer solution (5.8). Dilute to the mark and mix.

7.3.2 *Photometric measurements*

After 10 min, carry out the photometric measurements with the spectrophotometer (6.6) at a wavelength of about 510 nm or with the photoelectric absorptiometer (6.7) with suitable filters, adjusting the instrument to zero absorbance against the compensation solution.

7.3.3 *Preparation of the calibration chart*

Prepare a calibration chart having, for example, the Fe_2O_3 contents in milligrams per 100 ml of standard matching solutions as abscissae and the corresponding values of absorbance as ordinates.

7.4 Determination

7.4.1 *Preparation of the test solution*

7.4.1.1 Alkaline fusion

Weigh into the platinum dish (6.1), 12 g of the sodium carbonate (5.1) and 4 g of the boric acid (5.2). Mix thoroughly using a platinum spatula. Add the test portion (7.1) to the mixture and mix thoroughly. Cover the dish with its lid and place it in the electric furnace (6.2), controlled at 550 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support to avoid the risk of contamination. Maintain at 550 ± 25 °C until the reaction subsides (about 30 min).

Then transfer the dish to the electric furnace (6.3) controlled at 750 ± 25 °C, again taking care to isolate it from the floor of the furnace. Keep the dish in the furnace for 30 min, making sure that the temperature of 750 ± 25 °C is maintained for at least 20 min.

Remove the dish from the furnace and allow to cool in air. Add boiling water to the dish and heat gently until dissolution is complete.

After cooling slightly, transfer the contents of the dish to a beaker of suitable capacity, containing 20 ml of the nitric acid solution (5.4).

Dissolve the residue (which consists essentially of iron(III) oxide) adhering to the walls of the dish with 18 ml of the nitric acid solution (5.4) and carefully wash the dish and its lid with hot water, collecting the washings in the beaker.

Simmer the solution for a few minutes to ensure complete dissolution. Allow to cool slightly and transfer quantitatively to a one-mark volumetric flask of either 250 or 500 ml capacity, according to the iron content to be determined.

After cooling, dilute to the mark and mix.

Alternatively, the following procedure may be used :

7.4.1.2 Acid fusion

Weigh into the platinum dish (6.1), 10 g of the potassium pyrosulphate (5.3). Add the test portion (7.1) and mix carefully with a platinum spatula. Cover the dish with its lid and place in the electric furnace (6.4) controlled at 700 ± 25 °C, taking care to isolate it from the floor of the furnace by means of a support to avoid the risk of contamination. Maintain for 30 min at the temperature of 700 ± 25 °C.

Remove the dish from the furnace and allow to cool in air. Add 10 ml of water and 10 ml of the hydrochloric acid solution (5.5) to the dish. Simmer the solution gently until complete dissolution is obtained, taking care at the same time to rinse the lid and walls of the dish with hot water and collect the washings in the dish. Allow to cool slightly and transfer the solution quantitatively to a one-mark volumetric flask of either 250 or 500 ml capacity according to the iron content to be determined.

After cooling, dilute to the mark and mix.

7.4.2 *Colour reaction*

7.4.2.1 Taking of aliquot portions

Take two aliquot portions of the test solution (7.4.1), each containing between 50 and 250 µg of Fe_2O_3, and place one in a beaker of suitable capacity and the other in a 100 ml one-mark volumetric flask.

7.4.2.2 Preliminary test to adjust pH

Dilute the aliquot in the beaker to approximately 50 ml with water. Then add 5 ml of the hydroxylammonium chloride solution (5.6), wait for 2 or 3 min, then add 5 ml of the 1,10-phenanthroline solution (5.7) and 25 ml of the buffer solution (5.8). Check the pH value of the solution using either the indicator paper (5.13) or the pH meter (6.5). This value should be between 3,5 and 4,2; if not, adjust the pH value by slowly adding the required volume of the sodium acetate solution (5.9) or acetic acid solution (5.10), as appropriate; stir after each addition.

Note the volume of reagent used to adjust the pH and discard the solution.

7.4.2.3 Colour development

To the aliquot portion placed in the 100 ml one-mark volumetric flask, add the same quantities of all the reagents used in the preliminary test (7.4.2.2).

Dilute to the mark and mix.

7.4.2.4 Photometric measurements

After 10 min carry out the photometric measurements of the test solution and blank solution following the procedure described in 7.3.2, adjusting the instrument to zero absorbance against water.

8 EXPRESSION OF RESULTS

By reference to the calibration curve (see 7.3.3), read the iron contents corresponding to the values of the photometric measurements.

The iron content, expressed as iron(III) oxide (Fe_2O_3), is given, as a percentage by mass, by the formula :

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

where

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

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

m_1 is the mass, in milligrams, of iron(III) oxide determined in the aliquot portion of the test solution;

m_2 is the mass, in milligrams, of iron(III) oxide determined in the corresponding aliquot portion of the blank test solution.

9 NOTES ON PROCEDURE

Generally, the aliquot portion of the blank solution after treatment for photometric measurement shows a slight coloration. In this case, it is advisable to use it as the compensation solution.

In this case, the formula for calculation becomes :

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

where the symbols have the same meaning as those given in section 8.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or those documents to which reference is made, or regarded as optional.