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Aluminium fluoride for industrial use — Determination of fluorine content — Modified Willard-Winter method

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FOREWORD

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

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Aluminium fluoride for industrial use — Determination of fluorine content — Modified Willard-Winter method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a modified Willard-Winter method for the determination of the fluorine content of aluminium fluoride for industrial use.

2 REFERENCES

ISO/R 565, *Woven wire cloth and perforated plates in test sieves — Nominal sizes of apertures.*

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

3 PRINCIPLE

Fusion of a test portion with sodium carbonate.

Separation of fluorine by distillation with sulphuric acid. Titration with thorium nitrate solution using sodium alizarinsulphonate-methylene blue as indicator.

Alternatively the thorium nitrate titration may be carried out using sodium alizarinsulphonate alone as indicator, the end point being spectrophotometrically determined under carefully defined conditions when the absorbance at 525 nm reaches the arbitrary value of 0,60.

4 REAGENTS

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

4.1 Sodium carbonate, anhydrous.

4.2 Hydrochloric acid, approximately 0,06 N solution.

Dilute 5 ml of hydrochloric acid, ρ approximately 1,19 g/ml, 38 % (*m/m*) solution, with water to 1 000 ml.

4.3 Sodium hydroxide, 20 g/l solution.

Dissolve 20 g of sodium hydroxide in water and, after cooling, dilute to 1 000 ml.

4.4 Sulphuric acid, approximately 24 N solution.

Carefully add, in small quantities, 200 ml of sulphuric acid, ρ approximately 1,84 g/ml, 96 % (*m/m*), to approximately 100 ml of water, cool and dilute to 300 ml.

4.5 Buffer solution, pH 2,7.

Dissolve 9,45 g of monochloroacetic acid in 50 ml of N sodium hydroxide solution and dilute to 100 ml.

4.6 Thorium nitrate, standardized solution (approximately 0,067 N).

1 ml of this solution is equivalent to approximately 1,3 mg of F.

4.6.1 *Preparation of the solution*

Dissolve 9,45 g of thorium nitrate tetrahydrate [$Th(NO_3)_4.4H_2O$], or the corresponding mass of other hydrates, in water and dilute to 1 000 ml.

4.6.2 *Standardization of the solution*

4.6.2.1 Preparation of the standard reference solution

Weigh, to the nearest 0,1 mg, about 0,2 g of extra pure anhydrous sodium fluoride, previously heated at 600 °C in a platinum dish and cooled in a desiccator. Transfer, using 20 to 30 ml of water, into the distillation flask (5.4.1) containing several glass balls (2 to 3 mm diameter).

Stopper the distillation flask and add, through the dropping funnel (5.4.5), 50 ml of the sulphuric acid solution (4.4).

Carry out the distillation as described in 6.3.2.

Collect the distillate in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

NOTE — If extra pure sodium fluoride is not available, recrystallize the product as follows. Dissolve approximately 5 g of pure sodium fluoride in 125 ml of water and, after dissolution, filter under vacuum through a small Buchner funnel. Evaporate the solution in a platinum dish down to about 60 ml.

Cool to about 50 °C and separate the sodium fluoride crystals by centrifuging. Wash the crystals three times by centrifuging with small quantities of cold water.

Transfer the product to a platinum dish and dry in an electric oven with natural draught, at 110 °C.

Remove the dish from the oven, cool in a desiccator, grind the product in an agate mortar, then sieve through a sieve with a mesh size of 0,355 mm (see ISO/R 565). Place the sieved sodium fluoride in a platinum dish, heat for 2 h at 600 °C and then allow to cool in a desiccator.

1) At present at the stage of draft.

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4.6.2.2 Titration

Transfer a 50,0 ml aliquot portion of the standard reference solution (4.6.2.1) to the beaker (5.7) and titrate as described in 6.3.3.

Towards the end of the titration add the last few drops of thorium nitrate (4.6.1) carefully with vigorous stirring.

4.6.2.3 Blank test

Carry out a blank test following the same procedure (distillation as described in 6.3.2 and titration as described in 6.3.3) with the same quantity of all the reagents as used in the procedure described in 4.6.2.1 and 4.6.2.2.

4.6.2.4 Calculation of the strength of the solution

The mass, in milligrams, of fluorine (F) corresponding to 1 ml of thorium nitrate solution is given by the formula

$$\frac{m_1 \times 0{,}452\,5}{V_1 - V_2}$$

where

m_1 is the mass, in milligrams, of NaF contained in the aliquot portion of the standard reference solution (4.6.2.1) taken for the titration;

V_1 is the volume, in millilitres, of thorium nitrate solution (4.6.1) used for the titration of the aliquot portion of the standard reference solution (4.6.2.1) taken for the titration;

V_2 is the volume, in millilitres, of thorium nitrate solution (4.6.1) used for the titration of a corresponding aliquot portion of the blank test solution (4.6.2.3);

0,452 5 is the conversion factor from sodium fluoride to fluorine.

4.7 Sodium alizarinsulphonate, 0,5 g/l solution[1)]

Dissolve 0,05 g of sodium alizarinsulphonate in water and dilute to 100 ml.

4.8 Methylene blue, 0,5 g/l solution[1)]

Dissolve 0,05 g of methylene blue (oxidation-reduction indicator grade) in water and dilute to 100 ml.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Flat-bottomed platinum crucible, upper diameter approximately 30 mm, lower diameter approximately 15 mm and height approximately 30 mm, fitted with a platinum lid.

5.2 Electric furnace, capable of being controlled at 800 ± 20 °C.

5.3 Steam generator, for example a flask of approximately 3 000 ml capacity, fitted with a stopper into which are inserted the three glass tubes, a), b), c), of internal diameter about 6 mm.

a) vertical recovery bend tube, for introducing the steam into the distillation flask (5.4.1) (one limb dipping into the distillation flask);

b) tube for regulating the flow of the steam, fitted at its outer end with a rubber tube fitted with a Mohr clip;

c) safety tube, about 1 m long.

5.4 Apparatus of borosilicate glass for steam distillation, with ground glass joints, consisting of :

5.4.1 *Flask,* Claisen, 250 ml capacity, with the following preferred dimensions :

— diameter of central neck : 36 mm;

— length of side neck (including the Vigreux column (5.4.2)) : 275 mm;

— distance between side neck and central neck : 65 mm;

— diameter of side neck : 20 mm.

5.4.2 *Distillation column,* Vigreux, preferably having the following dimensions :

— column, length between the first and last of the series of points : 120 mm;

— 11 groups of 3 points, spaced at 120° on the circumference, at 12 mm separation.

5.4.3 *Thermometer pocket.*

5.4.4 *Thermometer* covering the range 0 to 200 °C, with an effective length of about 250 mm.

5.4.5 *Dropping funnel,* Walter, about 100 ml capacity, for insertion in the Vigreux column.

5.4.6 *Condenser,* Graham, with an effective length of about 400 mm.

For a typical form of apparatus, see the Figure.

5.5 Electric heater for heating the distillation flask (5.4.1) up to a temperature of 150 °C and controlled to within ± 1 °C.

5.6 pH meter, fitted with a glass electrode.

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1) In the visual titration instead of using the two indicators 4.7 and 4.8 (see 6.3.3) the sodium alizarinsulphonate solution (4.7) may be used alone. Alternatively methyl-thymol blue or any other indicator that gives equivalent results in the specified pH range may be used.

5.7 Beaker, of borosilicate glass, 250 ml, tall form.

5.8 Burette, 10 ml with 0,02 ml divisions.

5.9 Stirrer, magnetic.

5.10 Spectrophotometer, adapted for the titration.

5.11 Titration cell, with 5 cm optical path, 5 cm wide and 7,5 cm high.

NOTE – All the glassware shall be carefully rinsed with a hot chromic-sulphuric acid mixture, then thoroughly rinsed with water and finally with distilled water.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,1 mg, about 0,2 g of the dried sample into the platinum crucible (5.1).[1)]

6.2 Blank test

Carry out, using the same procedure, a blank test with the same quantities of all the reagents used for the determination.

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6.3 Determination

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6.3.1 *Preparation of the test solution*

Introduce into the crucible (5.1) containing the test portion (6.1) 2 g of the sodium carbonate (4.1) and mix carefully, preferably by means of a platinum wire. Place the crucible in the electric furnace (5.2), previously heated to about 200 °C, and heat to 800 ± 20 °C. Maintain at this temperature until the carbon dioxide is eliminated and the sample has melted (20 min approximately).

Remove the crucible from the furnace and cool rapidly by plunging the base of the crucible into a bath of cold water.

Transfer the fused mass directly to the distillation flask (5.4.1), containing several glass balls (2 to 3 mm diameter), and wash the crucible carefully with 20 to 30 ml of hot water in order to dissolve that part of the melt still adhering to the crucible, collecting the washings in the distillation flask (5.4.1).

6.3.2 *Distillation*

Place under the condenser (5.4.6) a 500 ml one-mark volumetric flask to collect the distillate.

Connect the distillation flask (5.4.1) to the condenser (5.4.6) and start the water circulation.

Stopper the distillation flask and add, through the dropping funnel (5.4.5), 50 ml of the sulphuric acid solution (4.4).

Fill the flask (5.3) two-thirds full of water and add several small pieces of pumice stone. Heat the flask, leaving the regulating tube b) open until the water boils.

Using the electric heater (5.5), heat the distillation flask (5.4.1) until the solution reaches 150 °C.

Pass the steam (at a rate of 250 to 300 g/h) through tube a), regulating the flow by means of the Mohr clip fitted to tube b) so as to maintain the solution in the distillation flask (5.4.1) at 150 ± 1 °C and collect about 400 ml of the distillate over a period of about 90 min.

Disconnect the distillation flask (5.4.1) from the steam generator (5.3) allowing the steam to escape to atmosphere, and remove the heater (5.5).

Wash the condenser with a jet of water.

Dilute the distillate to the mark in the collecting flask and mix.

6.3.3 *Titration*

6.3.3.1 Visual titration

Transfer 50,0 ml of the solution (6.3.2) to the beaker (5.7). Add to the beaker about 50 ml of water and 0,50 ml of the sodium alizarinsulphonate solution (4.7) and then, in small portions, sodium hydroxide solution (4.3) until a pink coloration appears (pH of the colour change 6,6 to 6,8).

Checking by means of the pH meter (5.6), add the hydrochloric acid solution (4.2) drop by drop until the pH value is between 4,9 and 5,2 (yellow coloration of the solution). Add 3,0 ml of sodium alizarinsulphonate solution (4,7) and then, still checking with the pH meter (5.6), add the buffer solution (4.5) in small portions until the pH is 3,4 ± 0,1 (about 1 ml of the buffer solution is needed). Add 0,50 ml of the methylene blue solution (4.8) (green coloration of the solution).

Place a small glass-encased iron bar in the solution and stir vigorously using the stirrer (5.9).

Fill the burette (5.8) with the thorium nitrate solution (4.6) and titrate just to the development of a blue-violet colour.

Ensure that the same lighting conditions are used as for the standardization of the thorium nitrate solution (4.6.2.2).

NOTE – Carry out the titration in daylight or fluorescent light only. The titration must not be carried out with the illumination provided by a tungsten filament lamp.

6.3.3.2 Spectrophotometric titration

Transfer 50,0 ml of the solution (6.3.2) to the titration cell (5.11) and dilute to about 100 ml. Add 3,0 ml of the sodium alizarinsulphonate solution (4.7) and then, in small portions, sodium hydroxide solution (4.3) until a pink coloration appears (pH of the colour change 6,6 to 6,8).

1) See ISO 2925.

Checking by means of the pH meter (5.6), add the hydrochloric acid solution (4.2) drop by drop until the pH value is between 4,9 and 5,2 (yellow coloration of the solution). Still checking with the pH meter (5.6), add the buffer solution (4.5) in small portions until the pH is 3,4 ± 0,1 (about 1 ml of the buffer solution is needed).

Transfer the cell to the adapted spectrophotometer (5.10). Place a small glass-encased iron bar in the solution. Place the tip of the burette (5.8), filled with the thorium nitrate solution (4.6), into the solution and stir.

Cover the titration assembly, adjust the wavelength to 525 nm and choose an appropriate sensitivity. Close the shutter and adjust to zero transmittance. Open the shutter and adjust the slit width to give a transmittance of 100. Using the burette (5.8), titrate with the thorium nitrate solution (4.6) until the absorbance is 0,60 (25 % transmittance). Read the volume of titrant to the nearest 0,01 ml.

7 EXPRESSION OF RESULTS

The fluorine (F) content is expressed, as a percentage by mass, by the formula :

$$\frac{m_2 \times (V_3 - V_4)}{m_0} \times 1\,000$$

where

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

m_2 is the mass, in grams, of fluorine corresponding to 1 ml of standardized thorium nitrate solution (4.6);

V_3 is the volume, in millilitres, of thorium nitrate solution (4.6) used for the titration of the aliquot portion of the sample solution (6.3.2);

V_4 is the volume, in millilitres, of thorium nitrate solution (4.6) used for the titration of a corresponding aliquot portion of the blank test solution (6.2).

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

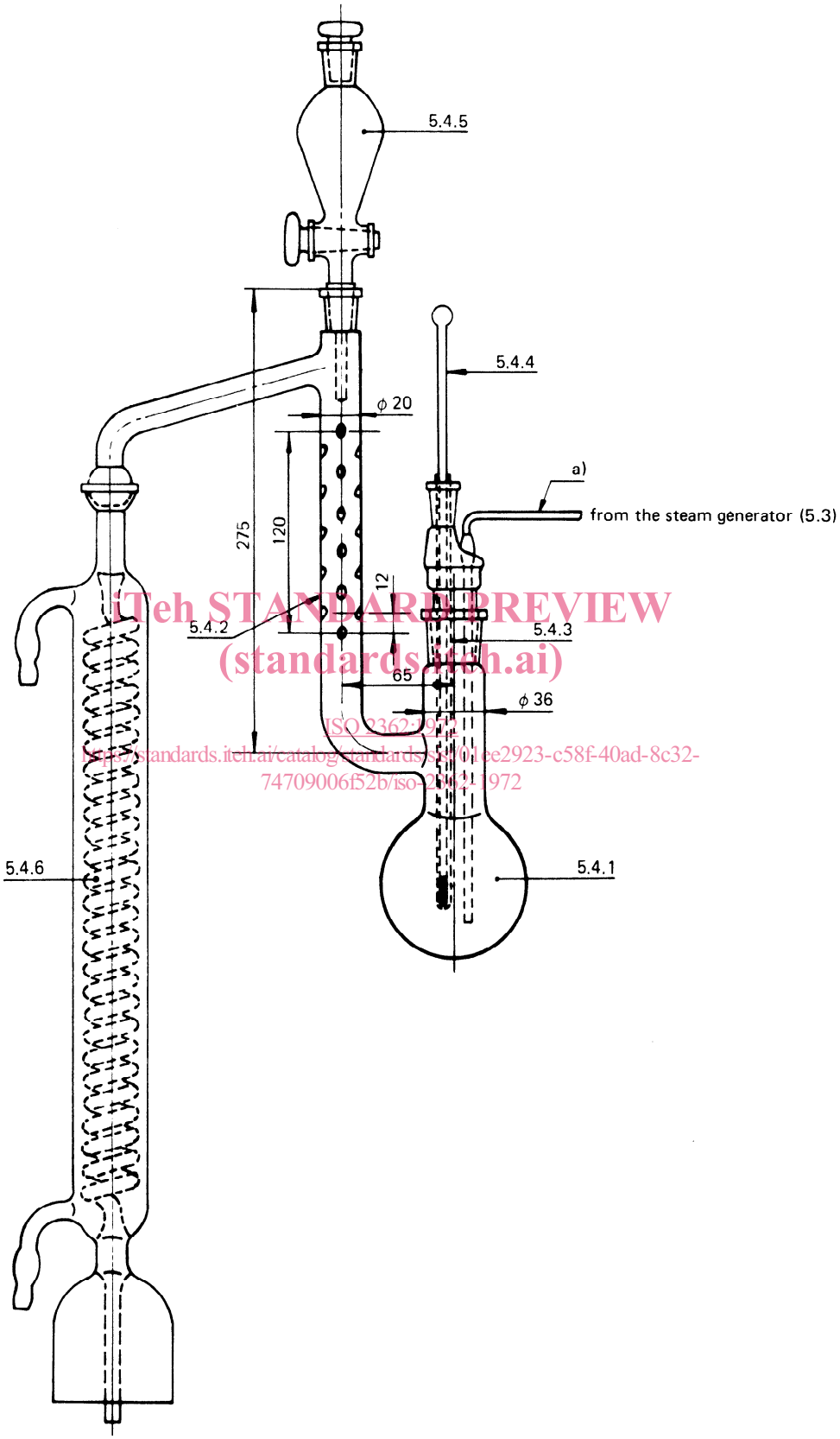
b) the results and the method of expression used;

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.

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Dimensions in millimetres



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FIGURE – **Typical form of apparatus for steam distillation**

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