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

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FOREWORD

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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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Sodium 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 fluorine content of sodium fluoride for industrial use.

2 REFERENCE

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

3 PRINCIPLE

Separation of the fluorine from a test portion by distillation with sulphuric acid or perchloric acid. Titration with thorium nitrate solution using sodium alizarinsulphonate and methylene blue as indicators.

Alternatively the thorium nitrate titration may be carried out using sodium alizarinsulphonate alone as indicator, the end-point being determined spectrophotometrically 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 Hydrochloric acid, approximately 0,06 N solution.

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

4.2 Sodium hydroxide, 20 g/l solution.

4.3 Sulphuric acid, approximately 24 N solution.

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

or

4.3.1 Perchloric acid, ρ approximately 1,60 g/ml, about 64,5 % (*m/m*) solution.

4.4 Buffer solution, pH 2,7

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

4.5 Thorium nitrate, approximately 0,067 N standard volumetric solution.

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

4.5.1 *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.

4.5.2 *Standardization of the solution*

4.5.2.1 PREPARATION OF THE STANDARD REFERENCE SOLUTION

Weigh, to the nearest 0,000 1 g, 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 (5.2.1) containing several glass balls (2 to 3 mm diameter).

1) In preparation.

Stopper the distillation flask and add, through the dropping funnel (5.2.5), either 50 ml of the sulphuric acid solution (4.3) or 30 ml of the perchloric acid solution (4.3.1), whichever has been selected.

Carry out the distillation as described in 6.3.1.

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. Dissolve about 5 g of pure sodium fluoride in 125 ml of water and, after dissolution, filter under vacuum through a small Buchner funnel. Then evaporate the solution, in a platinum dish, down to approximately 60 ml.

Cool to about 50 °C and separate the sodium fluoride crystals by centrifuging. Wash the crystals three times, always 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 ± 2 °C.

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

4.5.2.2 TITRATION

Transfer a 50,0 ml aliquot portion of the standard reference solution (4.5.2.1) to the beaker (4.5) and titrate as described in 6.3.2.

Towards the end of the titration, add the last few drops of thorium nitrate solution (4.5.1) with extreme caution, stirring vigorously.

4.5.2.3 BLANK TEST

Carry out a blank test at the same time and following the same procedure (distillation as described in 6.3.1 and titration as described in 6.3.2) with the same quantities of the reagents as used in the procedure described in 4.5.2.1. Titrate using the conditions described in 4.5.2.2.

4.5.2.4 CALCULATION OF 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 (4.5.2.1) taken for the titration;

V_1 is the volume, in millilitres, of the thorium nitrate solution (4.5.1) used for the titration of the aliquot portion of the standard reference solution (4.5.2.1) taken for the titration;

V_2 is the volume, in millilitres, of the thorium nitrate solution (4.5.1) used for the titration of a corresponding aliquot portion of the blank test solution (4.5.2.3);

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

4.6 Sodium alizarinsulphonate, 0,5 g/l solution.

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

4.7 Methylene blue, 0,5 g/l solution.

Dissolve 0,05 g of methylene blue in water and dilute to 100 ml.

NOTE — For the visual titration (see 6.3.2.1), instead of using the two indicators 4.6 and 4.7, either the sodium alizarinsulphonate solution (4.6) alone can be used or it can be replaced by a solution of methylthymol blue or any other indicator giving equivalent results in the specified pH range.

5 APPARATUS

Ordinary laboratory apparatus and

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

a) Double bend delivery tube, with parallel limbs, for introducing steam into the distillation flask (5.2.1). One limb shall dip into the distillation flask.

b) Tube for regulating the steam flow, fixed at its outer end with a rubber tube, fitted with a Mohr clip.

c) Safety tube, approximately 1 m in length.

5.2 Borosilicate glass apparatus, with ground glass joints, for the steam distillation, consisting of :

5.2.1 *Distillation flask, Claisen,* 250 ml capacity, with the following preferred dimensions :

— diameter of central neck : 36 mm;

— length of side neck (including the Vigreux column (5.2.2)) : 275 mm;

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

— diameter of side neck : 20 mm.

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

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

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

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