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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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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2833 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in June 1972.

It has been approved by the Member Bodies of the following countries :

Australia	Italy	Sweden
Austria	Morocco	Switzerland
Belgium	Netherlands	Thailand
Czechoslovakia	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.S.R.
Hungary	Romania	
India	South Africa, Rep. of	

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.

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 alizarin-sulphonate 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 monochloroacetic 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 $[\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}]$ 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.