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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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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 2362 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

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

Belgium	Israel	South Africa, Rep. of
Bulgaria	Italy	Spain
Egypt, Arab Rep. of	Netherlands	Switzerland
France	New Zealand	Thailand
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Ireland	Romania	Yugoslavia

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Canada
India

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 $[\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}]$, or the corresponding mass of other hydrates, in water and dilute to 1 000 ml.

4.6.2 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.