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Aqueous hydrofluoric acid for industrial use — Sampling and methods of test

Acide fluorhydrique en solution à usage industriel — Échantillonnage et méthodes d'essai

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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 3139 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in June 1973.

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

Austria	India	South Africa, Rep. of
Belgium	Israel	Spain
Bulgaria	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.S.R.
Hungary	Romania	

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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Aqueous hydrofluoric acid for industrial use — Sampling and methods of test

WARNING — Aqueous hydrofluoric acid is a highly corrosive liquid which attacks glass; the vapour is irritant and toxic. Its action on the skin and eyes is strongly corrosive, producing severe and painful burns which may not be immediately evident and which respond slowly to treatment.

Samples should be handled only inside a well-ventilated fume cupboard. Rubber gloves, boots and gown of a suitable size to give adequate protection to the individual, and full head and face protection must be worn when handling the material.

In the event of contact or suspected contact, flood with water and seek immediate medical attention. The manufacturers' literature should be consulted for further information.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods of sampling and test for aqueous hydrofluoric acid for industrial use.

2 SAMPLING

For this dangerous material, a test sample shall be prepared by dilution, if required, of a bulk sample as specified in 2.1.

2.1 Test sample

2.1.1 *Reagent*

Distilled water, or water of equivalent purity, and **ice** obtained from such water.

2.1.2 *Apparatus*

Ordinary laboratory apparatus and

2.1.2.1 Screw-capped polyolefin sample bottle of capacity 150 ml, graduated at 100 ml.

2.1.3 *Procedure*

Weigh, to the nearest 0,01 g, a mass of a mixture of the ice and water (2.1.1) depending on the concentration of hydrofluoric acid in the bulk sample, as shown in the following table, into the tared sample bottle (2.1.2.1).

TABLE — **Mass of mixture of ice and water for preparation of test sample**

Concentration of bulk sample	Mass of mixture of ice and water
HF % (*m*/*m*)	g
40 to 50	0
50 to 60	15
60 to 70	35
> 70	50

Carefully fill the sample bottle (2.1.2.1) to the mark with the bulk sample, cool if necessary and reweigh to the nearest 0,01 g.

3 DETERMINATION OF TOTAL ACIDITY AND FLUOROSILICIC ACID CONTENT — TITRIMETRIC METHOD

3.1 Scope

This clause specifies a titrimetric method for the determination of the total acidity and fluorosilicic acid contents of 40 to 85 % (*m*/*m*) commercial hydrofluoric acid for industrial use.

3.2 Field of application

This method is applicable to the determination of fluorosilicic acid contents of between 0,2 and 10 % (*m*/*m*), expressed as H_2SiF_6.

3.3 Principle

Titration of an ice-cold test portion with standard volumetric sodium hydroxide solution, in the presence of potassium nitrate and using phenolphthalein as indicator, followed by a titration after heating.

NOTE — The first titration corresponds to the acids other than fluorosilicic acid together with the two equivalents of acid that are released by precipitation of the fluorosilicic acid. The second titration corresponds to the further four equivalents of acid that are released after redissolution, by heating, of the precipitated potassium fluorosilicate.

3.4 Reagents

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water, or water of equivalent purity.

3.4.1 Crushed ice, obtained from distilled water or from water of equivalent purity.

3.4.2 Potassium nitrate, saturated solution at room temperature.

3.4.3 Sodium hydroxide, N standard volumetric solution.

Store this solution in a plastics bottle.

3.4.4 Sodium hydroxide, 0,1 N standard volumetric solution.

Store this solution in a plastics bottle.

3.4.5 Phenolphthalein, 10 g/l ethanolic solution.

Dissolve 1 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol. Add the standard volumetric sodium hydroxide solution (3.4.4), drop by drop, until a faint permanent pink colour is produced.

3.5 Apparatus

Ordinary laboratory apparatus.

3.6 Procedure

3.6.1 *Test portion*

Weigh, to the nearest 0,001 g, about 2 g of the test sample (2.1) into a stoppered PTFE or polyolefin weighing bottle.

3.6.2 *Determination*

Transfer the test portion (3.6.1) quantitatively to a 250 ml polyolefin beaker containing a slurry of 20 ml of the potassium nitrate solution (3.4.2) and the crushed ice (3.4.1). Use ice-cold water to rinse the weighing bottle and to rinse the washings into the beaker.

Add 5 drops of the phenolphthalein solution (3.4.5) and, while keeping the solution ice-cold, titrate first with the standard volumetric sodium hydroxide solution (3.4.3) until the end-point is approached. Complete the titration with the standard volumetric sodium hydroxide solution (3.4.4) to the appearance of a faint permanent pink colour.

Transfer the contents of the polyolefin beaker quantitatively to a 400 ml glass beaker, heat just to boiling, and titrate immediately with the standard volumetric sodium hydroxide solution (3.4.4) to the appearance of a faint permanent pink colour.

3.7 Expression of results

3.7.1 *Total acidity*

Total acidity, expressed as a percentage by mass of hydrofluoric acid (HF), is given by the formula

$$0{,}200\,1 \times \frac{10V_1 + V_2 + V_3}{m_0} \times \frac{m_1}{m_1 - m_2}$$

3.7.2 *Fluorosilicic acid content*

Fluorosilicic acid content, expressed as a percentage by mass of fluorosilicic acid (H_2SiF_6), is given by the formula

$$\frac{0{,}360\,3\,V_3}{m_0} \times \frac{m_1}{m_1 - m_2}$$

where

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4.3) used in the first titration;

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4.4) used to complete the first titration;

V_3 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4.4) used in the second titration;

NOTE – If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, appropriate corrections should be made.

m_0 is the mass, in grams, of the test portion (3.6.1);

m_1 is the mass, in grams, of the test sample (2.1);

m_2 is the mass, in grams, of the ice/water mixture used to prepare the test sample.

4 DETERMINATION OF NON-VOLATILE ACID CONTENT – TITRIMETRIC METHOD

4.1 Scope

This clause specifies a titrimetric method for the determination of the content of acids non-volatile at 100 °C of 40 to 85 % (*m/m*) commercial hydrofluoric acid for industrial use.

4.2 Field of application

This method is applicable to the determination of non-volatile acid contents of between 0,025 and 5 % (*m/m*), expressed as sulphuric acid (H_2SO_4).

4.3 Principle

Removal of volatile acids by evaporation, and titration of the remaining non-volatile acids with standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

4.4 Reagents

During the analysis use only reagents of recognized analytical reagent grade and only distilled water, or water of equivalent purity, freshly boiled and cooled.

4.4.1 Sodium hydroxide, 0,1 N standard volumetric solution.