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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

AMENDMENT 1

Amendment 1 to International Standard ISO 3139-1976 was developed by Technical Committee ISO/TC 47, *Chemistry*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO.

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Sub-clause 4.7 : In the second line of the note concerning the definition of ϱ, replace "1,0 g/ml" by "1,2 g/ml".

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Descriptors : hydrofluoric acid, sampling, chemical analysis, determination of content, fluorosilicic acid, impurities.

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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

Second edition — 1976-04-15

ISO 3139-1976 (E)

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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*. This second edition results from the incorporation of Addendum 1 in the first edition of the Standard.

This Addendum (clause 5 of the present document) was circulated to the Member Bodies in December 1974, and has been approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the document.

This second edition cancels and replaces the first edition (i.e. ISO 3139-1974) which had 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	

No Member Body had 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 the procedure for sampling aqueous hydrofluoric acid for industrial use, together with titrimetric methods for determination of the total acidity, the hexafluorosilicic acid content and the non-volatile acids content, and a method for calculating the hydrogen fluoride content.

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 HEXAFLUOROSILICIC ACID CONTENT — TITRIMETRIC METHOD

3.1 Scope

This clause specifies a titrimetric method for the determination of the total acidity and the hexafluorosilicic acid content 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 hexafluorosilicic acid contents of between 0,2 and 10 % (*m/m*), expressed as hexafluorosilicic acid (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 hexafluorosilicic acid together with the two equivalents of acid that are released by precipitation of the hexafluorosilicic acid. The second titration corresponds to the further four equivalents of acid that are released after redissolution, by heating, of the precipitated potassium hexafluorosilicate.

3.4 Reagents

During the analysis, use only reagents of recognized analytical 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, 1 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 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*

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

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

3.7.2 *Hexafluorosilicic acid content*

The hexafluorosilicic acid content, *b*, expressed as a percentage by mass of hexafluorosilicic acid (H_2SiF_6), is given by the formula

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

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 ACIDS 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 acids 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 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.

4.4.2 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 (4.4.1), drop by drop until a faint permanent pink colour is produced.

4.5 Apparatus

Ordinary laboratory apparatus and

4.5.1 Platinum dish, capacity approximately 150 ml.

4.6 Procedure

4.6.1 *Test portion*

Measure a suitable volume of the test sample (2.1) in a polyolefin measuring cylinder. The volume of the test portion shall be 40 ml for test samples which are expected to contain less than 1 % (*m/m*) of non-volatile acid. For higher concentrations, take proportionately smaller test portions.

4.6.2 *Determination*

Transfer the test portion (4.6.1) quantitatively to the platinum dish (4.5.1). Evaporate on a boiling water bath in a fume cupboard until nearly dry. Add 25 ml of water and again evaporate almost to dryness.

Transfer the residual solution quantitatively to a 250 ml conical flask, using a total volume of about 25 ml of water to rinse the dish.

Add 5 drops of the phenolphthalein solution (4.4.2) and titrate with the standard volumetric sodium hydroxide solution (4.4.1) to the appearance of a faint permanent pink colour.

4.7 Expression of results

The non-volatile acid content, c, expressed as a percentage by mass of sulphuric acid (H_2SO_4), is given by the formula

$$c = \frac{0{,}490\,4\,V_4}{\rho\,V_5} \times \frac{m_1}{m_1 - m_2}$$

where

V_4 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (4.4.1) used for the titration;

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction shall be made.

V_5 is the volume, in millilitres, of the test portion (4.6.1);

ρ is the density, in grams per millilitre, of the test sample (2.1);

NOTE — ρ can be assumed, with sufficient accuracy, to be 1,0 g/ml.

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.

5 CALCULATION OF HYDROGEN FLUORIDE CONTENT

5.1 Scope and field of application

This clause specifies a method for the calculation of the hydrogen fluoride content of 40 to 85 % (*m/m*) aqueous hydrofluoric acid for industrial use, using the results obtained from the determinations specified in clauses 3 and 4.

5.2 Calculation

The hydrogen fluoride content, expressed as a percentage by mass of hydrogen fluoride (HF), is given by the formula

$$a - [(b \times 0{,}833) + (c \times 0{,}408)] = a - 0{,}833\,b - 0{,}408\,c$$

where

a is the total acidity, expressed as a percentage by mass as hydrofluoric acid (HF), determined according to clause 3;

b is the hexafluorosilicic acid content, expressed as a percentage by mass as hexafluorosilicic acid (H_2SiF_6), determined according to clause 3;

c is the non-volatile acids content, expressed as a percentage by mass as sulphuric acid (H_2SO_4), determined according to clause 4;

0,833 is the factor for conversion of hexafluorosilicic acid (H_2SiF_6) to hydrogen fluoride (HF).

0,408 is the factor for conversion of sulphuric acid (H_2SO_4) to hydrogen fluoride (HF).

6 TEST REPORT

The test report shall include the following particulars :

a) the reference of the methods used;

b) the results and the methods of expression used;

c) any unusual features noted during the determinations;

d) any operation not included in this International Standard, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO ANHYDROUS HYDROGEN FLUORIDE AND AQUEOUS HYDROFLUORIC ACID FOR INDUSTRIAL USE

ANHYDROUS HYDROGEN FLUORIDE

ISO 3137 — Sampling.

ISO 3138 — Determination of non-volatile acid content — Titrimetric method.

ISO 3699 — Determination of water content — Karl Fischer method.

ISO 3700 — Determination of water content — Conductimetric method.

ISO 3701 — Determination of hexafluorosilicic acid content — Reduced molybdosilicate photometric method.

ISO 3702 — Determination of sulphur dioxide content — Iodometric method.

AQUEOUS HYDROFLUORIC ACID

ISO 3139 — Sampling and methods of test.