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INTERNATIONAL STANDARD ISO 3138

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Anhydrous hydrogen fluoride for industrial use — Determination of non-volatile acid content — Titrimetric method

Fluorure d'hydrogène anhydre à usage industriel — Dosage des acides non volatils — Méthode titrimétrique

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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 3138 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 :

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

No Member Body expressed disapproval of the document.

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Anhydrous hydrogen fluoride for industrial use — Determination of non-volatile acid content — Titrimetric method

WARNING — Anhydrous hydrogen fluoride is a highly corrosive liquid which boils at 19,5 °C. It attacks glass, has a great affinity for water and its 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

This International Standard specifies a titrimetric method for the determination of the content of acids non-volatile at 100 °C of anhydrous hydrogen fluoride for industrial use.

2 FIELD OF APPLICATION

This method is applicable to the determination of non-volatile acid contents of between 0,005 and 0,3 % (*m/m*), expressed as sulphuric acid (H_2SO_4).

3 REFERENCE

ISO 3137, *Anhydrous hydrogen fluoride for industrial use — Sampling.*

4 SAMPLING

Prepare the samples by the method specified in ISO 3137.

5 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.

6 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.

6.1 Sodium hydroxide, 0,01 N standard volumetric solution.

6.2 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 (6.1), drop by drop, until a faint permanent pink colour is produced.

7 APPARATUS

Ordinary laboratory apparatus and

7.1 Platinum dish, capacity approximately 150 ml.

8 PROCEDURE

8.1 Test portion

Measure 20 ml of the test sample (clause 4) in a polyolefin measuring cylinder.

8.2 Determination

Transfer the test portion (8.1) quantitatively to the platinum dish (7.1). Evaporate on a boiling water bath in a fume cupboard until nearly dry. Add 25 ml of water and again evaporate almost to dryness. Repeat the evaporation with two 5 ml portions of water. Finally transfer quantitatively the contents of the dish to a 100 ml conical flask, using about 25 ml of water. Add a few drops of the phenolphthalein solution (6.2) and titrate with the standard volumetric sodium hydroxide solution (6.1) to the appearance of a faint permanent pink colour.

9 EXPRESSION OF RESULTS

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

$$\frac{0{,}000\,49 \times V \times 100}{20\,\rho C/100} = \frac{0{,}245 \times V}{\rho C}$$

where

0,000 49 is the mass, in grams, of sulphuric acid corresponding to 1 ml of 0,01 N sodium hydroxide solution;

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (6.1) used;

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

20 is the volume, in millilitres, of the test portion (8.1);

ρ is the density, in grams per millilitre, of the test sample (see section two of ISO 3137);

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

C is the concentration, as a percentage by mass, of anhydrous hydrogen fluoride in the test sample, calculated by the formula specified in clause 12 of ISO 3137.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or the International Standard to which reference is made, or regarded as optional.

ANNEX

This document forms part of a series of International Standards on methods of test for anhydrous hydrogen fluoride and aqueous hydrofluoric acid for industrial use.

The complete list of the International Standards already prepared or in course of preparation is as follows :

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 fluorosilicic acid content.*

ISO 3702 — *Determination of sulphur dioxide content.*

AQUEOUS HYDROFLUORIC ACID

ISO 3139 — *Sampling and methods of test.*