INTERNATIONAL STANDARD 3702

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Anhydrous hydrogen fluoride for industrial use — Determination of sulphur dioxide content — Iodometric method

Fluorure d'hydrogène anhydre à usage industriel — Dosage du dioxyde de soufre — Méthode iodométrique

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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 3702 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and was circulated to the Member Bodies in January 1975.

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

Austria	Israel	Switzerland
Belgium	Italy	Turkey
Brazil	Poland	United Kingdom
France	Portugal	U.S.S.R.
Germany	Romania	Yugoslavia
Hungary	South Africa, Rep. of	
India	Spain	

No Member Body expressed disapproval of the document.

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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 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

This International Standard specifies an iodometric method for the determination of the sulphur dioxide content of anhydrous hydrogen fluoride for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having sulphur dioxide (SO_2) contents between 0,002 and 0,20 % (*m/m*).

3 REFERENCE

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

4 SAMPLING

For the preparation of the laboratory and test samples, use the methods specified in ISO 3137.

5 PRINCIPLE

Addition of a measured volume of iodine solution to a test portion. Back-titration of the excess iodine using a standard volumetric sodium thiosulphate solution, and calculation of the sulphur dioxide content from the amount of iodine used.

6 REAGENTS

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

6.1 Iodine, approximately 0,1 N solution.

Dissolve 20 g of potassium iodide in approximately 30 ml of water. Add 12,7 g of iodine and stir until dissolved. Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Renew this solution at least every month.

6.2 Iodine, approximately 0,01 N solution.

Place 100 ml of the iodine solution (6.1) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

6.3 Sodium thiosulphate, 0,01 N standard volumetric solution.

6.4 Starch indicator, 5 g/l solution.

Disperse 0,5 g of soluble starch in a few millilitres of cold water and add the dispersion to 100 ml of boiling water. Stir for 1 min.

Prepare a fresh solution daily.

7 APPARATUS

Ordinary laboratory apparatus and

7.1 Polyethylene weighing bottle.

7.2 Polyethylene beaker, capacity 500 ml.

7.3 Stirrer, consisting of a polyethylene rod.

8 PROCEDURE

8.1 Test portion

Transfer about 25 ml of the test sample, prepared in accordance with the method specified in section two of ISO 3137, into the weighing bottle (7.1), weighed to the nearest 0,1 g. Replace the lid and weigh again to the nearest 0,1 g.

8.2 Standardization of the iodine solution

Carry out the standardization determination, as indicated in 8.3, using 100 ml of water and 50,0 ml of the iodine solution (6.2), but omitting the test portion.

8.3 Determination

Place 100 ml of water in the polyethylene beaker (7.2) and add 50,0 ml of the iodine solution (6.2). Transfer the test portion (8.1) quantitatively to the mixture. Allow to stand for a few minutes and then titrate the excess iodine with the standard volumetric sodium thiosulphate solution (6.3) until the solution is pale yellow in colour. Add 2 ml of the starch indicator (6.4) and continue the titration until the solution is colourless. Use the rod (7.3) for stirring.

9 EXPRESSION OF RESULTS

The sulphur dioxide content, expressed as a percentage by mass of SO_2, is given by the formula

$$(V_0 - V_1) \times 0{,}000\,32 \times \frac{100}{m} \times \frac{100}{C} = \frac{3{,}2\,(V_0 - V_1)}{m \times C}$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (6.3) used in the standardization determination (8.2);

V_1 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (6.3) used in the test (8.3);

m is the mass, in grams, of the test portion (8.1);

C is the concentration of anhydrous hydrogen fluoride, expressed as a percentage by mass, in the test sample (see ISO 3137, clause 12);

0,000 32 is the mass, in grams, of sulphur dioxide (SO_2) corresponding to 1 ml of exactly 0,01 N sodium thiosulphate solution.

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.

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 in the International Standard to which reference is made, 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.