
INTERNATIONAL STANDARD



3699

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

Anhydrous hydrogen fluoride for industrial use — Determination of water content — Karl Fischer method

Fluorure d'hydrogène anhydre à usage industriel — Dosage de l'eau — Méthode de Karl Fischer

First edition — 1976-08-15

Sample Document

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UDC 661.487 : 546.212 : 543.257

Ref. No. ISO 3699-1976 (E)

Descriptors : hydrogen fluoride, chemical analysis, determination of content, water, Karl Fischer reagent.

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 3699 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the Member Bodies in February 1975.

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

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

No Member Body expressed disapproval of the document.

Anhydrous hydrogen fluoride for industrial use — Determination of water content — Karl Fischer 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 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 a method for the determination of the water content of anhydrous hydrogen fluoride for industrial use. This method, that of Karl Fischer, is intended for reference purposes.

NOTE — Attention is drawn to ISO 3700¹⁾, which describes an alternative method suitable for routine purposes.

2 FIELD OF APPLICATION

The method is applicable to products having water contents between 0,01 and 0,5 % (m/m).

3 REFERENCES

ISO/R 760, *Determination of water by the Karl Fischer method*.

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

4 SAMPLING

For the preparation of the laboratory sample, use the method specified in section one of ISO 3137.

5 PRINCIPLE

Addition of a cooled test portion to cooled, dry pyridine containing 5 % of anhydrous hydrogen fluoride. Electro-metric titration of the water with Karl Fischer reagent.

6 REAGENTS

During the analysis, use only reagents of recognized analytical grade.

6.1 Karl Fischer reagent, water equivalent 4 to 6 mg/ml (see ISO/R 760).

6.2 Pyridine solvent.

In a polyethylene bottle, cool 1 000 ml of dry pyridine to –50 °C in a mixture of dry ice and methanol. Stir vigorously and continue cooling while slowly adding 50 ml of anhydrous hydrogen fluoride. Store in the polyethylene bottle.

7 APPARATUS

Ordinary laboratory apparatus and

7.1 Apparatus for measurement of test portion and titration (see figures 1 and 2), comprising :

7.1.1 Cooling coil, stainless steel, mounted vertically in a cylindrical cooling bath constructed of mild steel.

7.1.2 Vessel, for the measurement of the test portion, comprising a body constructed of stainless steel fitted with windows made of a sandwich consisting of polytetrafluoroethylene (PTFE) foil/glass sheet/PTFE foil. The front window is marked at a volume of about 40 ml.

The construction is shown in detail in figure 2.

1) At present at the stage of draft.