International Standard ISO 3700

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

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

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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 3700 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in November 1978.

It has been approved by the member bodies of the following countries :

Australia
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Thailand
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USSR
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The member body of the following country expressed disapproval of the document on technical grounds :

Austria

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Anhydrous hydrogen fluoride for industrial use — Determination of water content — Conductimetric 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 manufacturer's literature should be consulted for further information.

1 Scope

This International Standard specifies a conductimetric method for the determination of the water content of anhydrous hydrogen fluoride for industrial use. The method is suitable for use as a routine alternative to the reference Karl Fischer method specified in ISO 3699, but may give a lower standard of accuracy.

2 Field of application

The method is applicable to products having water contents between 0,01 and 0,4 % (*m*/*m*).

3 References

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

ISO 3138, *Anhydrous hydrogen fluoride for industrial use — Determination of non-volatile acid content — Titrimetric method.*

ISO 3699, *Anhydrous hydrogen fluoride for industrial use — Determination of water content — Karl Fischer method.*

4 Sampling

Prepare the laboratory sample by the method specified in section 1 of ISO 3137.

5 Principle

Measurement of the conductance of anhydrous hydrogen fluoride using a dip cell electrode. Determination of the corresponding water content by means of a calibration graph making a correction for the non-volatile acid content.

6 Reagents

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

6.1 Propan-2-ol.

6.2 Hydrofluoric acid, approximately 40 % (*m*/*m*) aqueous solution.

WARNING — Hydrofluoric acid is toxic and corrosive. Avoid inhalation of fumes. Avoid contact with skin, eyes and clothing.

NOTE — 40 % is the nominal concentration of commercially available hydrofluoric acid solutions. Although the measured concentration may vary from sample to sample, experience indicates that, for the purpose of this determination, such solutions are nevertheless suitable for use in the check test (clause 10).

6.3 Chloroplatinic acid, approximately 30 g/l solution.

Dissolve 4,5 g of chloroplatinic acid hexahydrate ($H_2PtCl_6.6H_2O$) and 0,037 g of lead acetate trihydrate [$(CH_3COO)_2Pb.3H_2O$] in 150 ml of water.

6.4 Sulphuric acid, approximately 10 % (*m*/*m*) solution.

Carefully add 5,5 ml of sulphuric acid, ϱ approximately 1,84 g/ml, to about 80 ml of water and, after cooling, dilute to 100 ml.

6.5 Potassium chloride, standard conductimetric solution, c (KCl) = 0,01 mol/l.

Dissolve 0,745 5 ± 0,000 2 g of potassium chloride, previously dried at about 110 °C for 2 h and cooled in a desiccator, in 1 000 ml of water.

7 Apparatus

Ordinary laboratory apparatus and

7.1 Coupling, stainless steel threaded union, to fit the sampling cylinder (see figure 1 and ISO 3137).

7.2 Delivery tube, stainless steel, (see figure 1).

7.3 Conductivity measuring bridge, including the range 1 to 50 mS.

7.4 Dip cell electrode, as shown in figure 2, with platinized platinum electrodes.

The electrodes shall be platinized or replatinized as follows :

7.4.1 Clean the electrodes by degreasing the surfaces to be plated with diethyl ether, after polishing, if necessary, to remove any irregularities.

7.4.2 Immerse the electrodes in the chloroplatinic acid solution (6.3) and electrolyse for 2 min using a current of approximately 100 mA and a voltage of 4 to 5 V. Reverse the polarity and repeat the electrolysis.

7.4.3 Wash the electrodes thoroughly with water.

7.4.4 Immerse the electrodes in the sulphuric acid solution (6.4) and electrolyse again, for 1 min, using the same current and voltage as in 7.4.2. Reverse the polarity and repeat the electrolysis.

7.4.5 Wash the electrodes with water and then with diethyl ether to dry them completely.

NOTE — Systems for the measurement of conductance are commercially available and may be used instead of the apparatus, specified in 7.3 and 7.4. If so, the manufacturer's instructions should be followed.

7.5 Polyethylene bottle, capacity 250 ml, marked at about 150 ml (as shown in figure 3).

NOTE — The dimensions shown in figure 3 are not critical.

7.6 Thermometer, graduated in 0,1 °C, covering the ambient temperature range and protected from attack by hydrogen fluoride by a thin sheath of polytetrafluoroethylene (PTFE).

8 Procedure

8.1 Determination of dip cell constant

If the constant of the dip cell is not known, obtain it by using the dip cell electrode (7.4) and bridge (7.3) to measure the conductance of the potassium chloride solution (6.5) at 20,0 ± 0,1 °C.

Calculate the dip cell constant (K) from the formula

$$\frac{1,28}{C_m}$$

where

1,28 is the specific conductance, in millisiemens, of the potassium chloride solution (6.5) at 20 ± 0,1 °C;

C_m is the measured conductance, in millisiemens, of the potassium chloride solution (6.5) at 20 ± 0,1 °C.

8.2 Test portion

Cool the sampling cylinder (see figure 1 and ISO 3137) to 10 to 15 °C. Remove the threaded stopper from one end of the transverse passageway and fit the threaded union coupling (7.1) and the delivery tube (7.2), previously dried by heating at 105 °C for 1 h. Transfer about 150 ml of the sample to the clean dry polyethylene bottle (7.5) and bring the bottle and contents to 15 ± 2 °C.

8.3 Measurement of conductance

Wash the dip cell electrode (7.4) with the propan-2-ol (6.1) and wipe and dry thoroughly with filter paper, taking care to avoid touching the platinum black surfaces. Immerse the electrode (7.4) in the test portion (8.2) and measure the conductance (C) using the bridge (7.3).

9 Expression of results

9.1 Calculate the specific conductance (C_s) from the formula

$$C \times K$$

where

C is the conductance, in millisiemens, of the test solution, determined as described in 8.3;

K is the dip cell constant, determined as described in 8.1.

9.2 From the graph (see figure 4), determine the percentage by mass of water corresponding to the specific conductance C_s.

9.3 The true water content, expressed as a percentage by mass, is given by the formula

$$A - \frac{18 \times S}{98} = A - 0,18\,S$$

where

A is the uncorrected water content, expressed as a percentage by mass, obtained as specified in 9.2;

S is the non-volatile acid content, expressed as a percentage by mass as sulphuric acid, determined by the method specified in ISO 3138;

18 is the relative molar mass of water (H_2O);

98 is the relative molar mass of sulphuric acid (H_2SO_4).

Express the result to the nearest 0,01 %.

10 Check test

At least once a month, carry out a check test by determining the water content before and after the addition of a known amount of the hydrofluoric acid solution (6.2) to anhydrous hydrogen fluoride of known low water content to give a total water content of about 0,2 % (m/m).

Alternatively, the check test may be carried out by determining water contents by the reference (Karl Fischer) method specified in ISO 3699. If the check test differs from the expected value by more than 10 %, or if the electrode response is sluggish, replatinize the electrode as specified in 7.4.

11 Test report

The test report shall include the following particulars :

a) the type and identification of the product tested;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

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

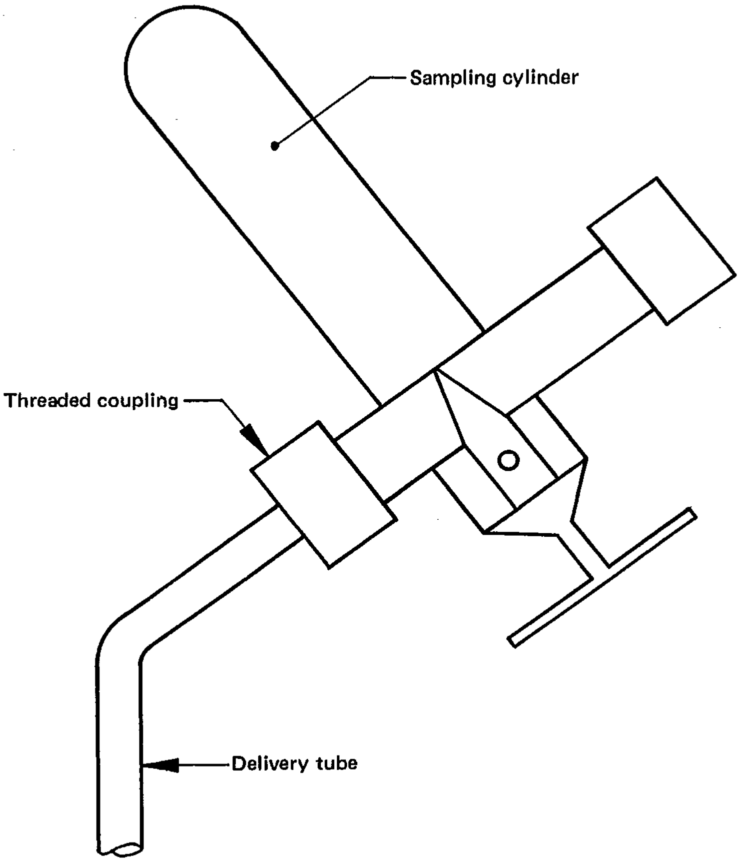

Figure 1 — Sampling cylinder with delivery tube

Dimensions in millimetres

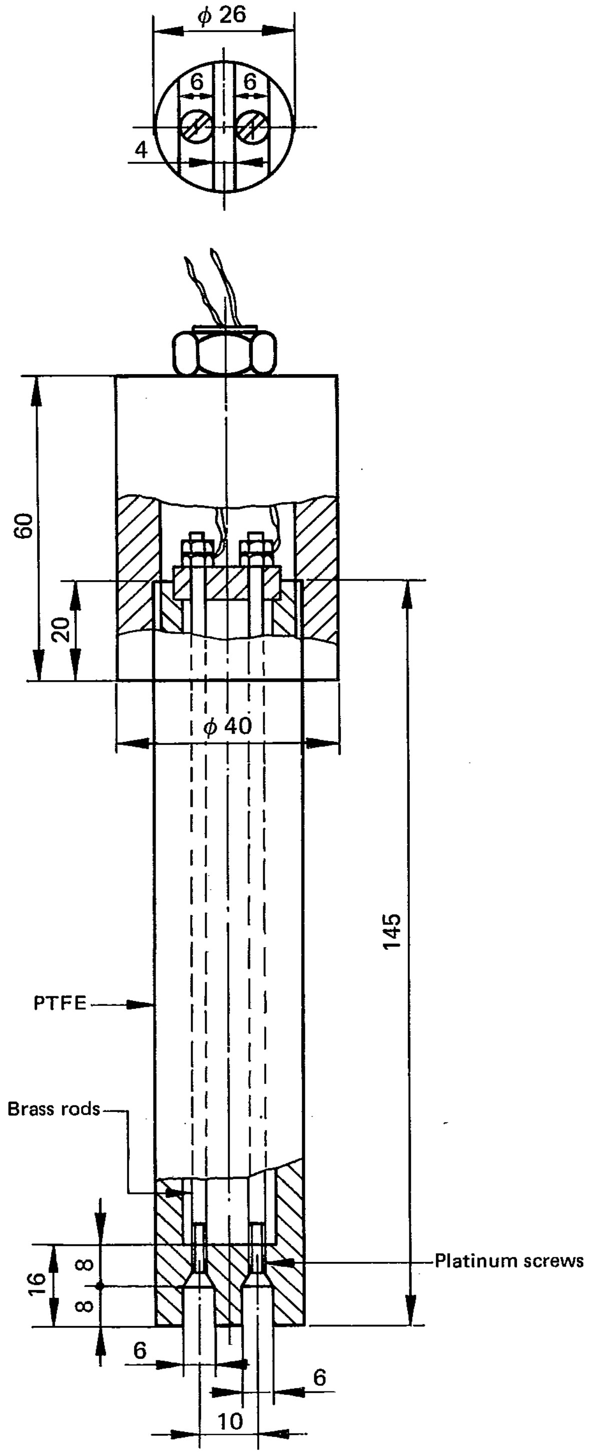

Figure 2 — Dip cell electrode

Dimensions in millimetres

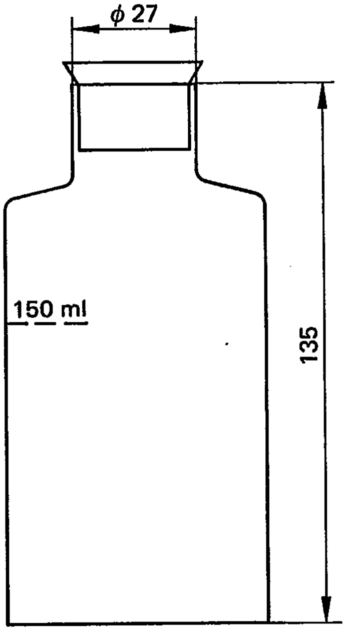

Figure 3 — Polyethylene bottle

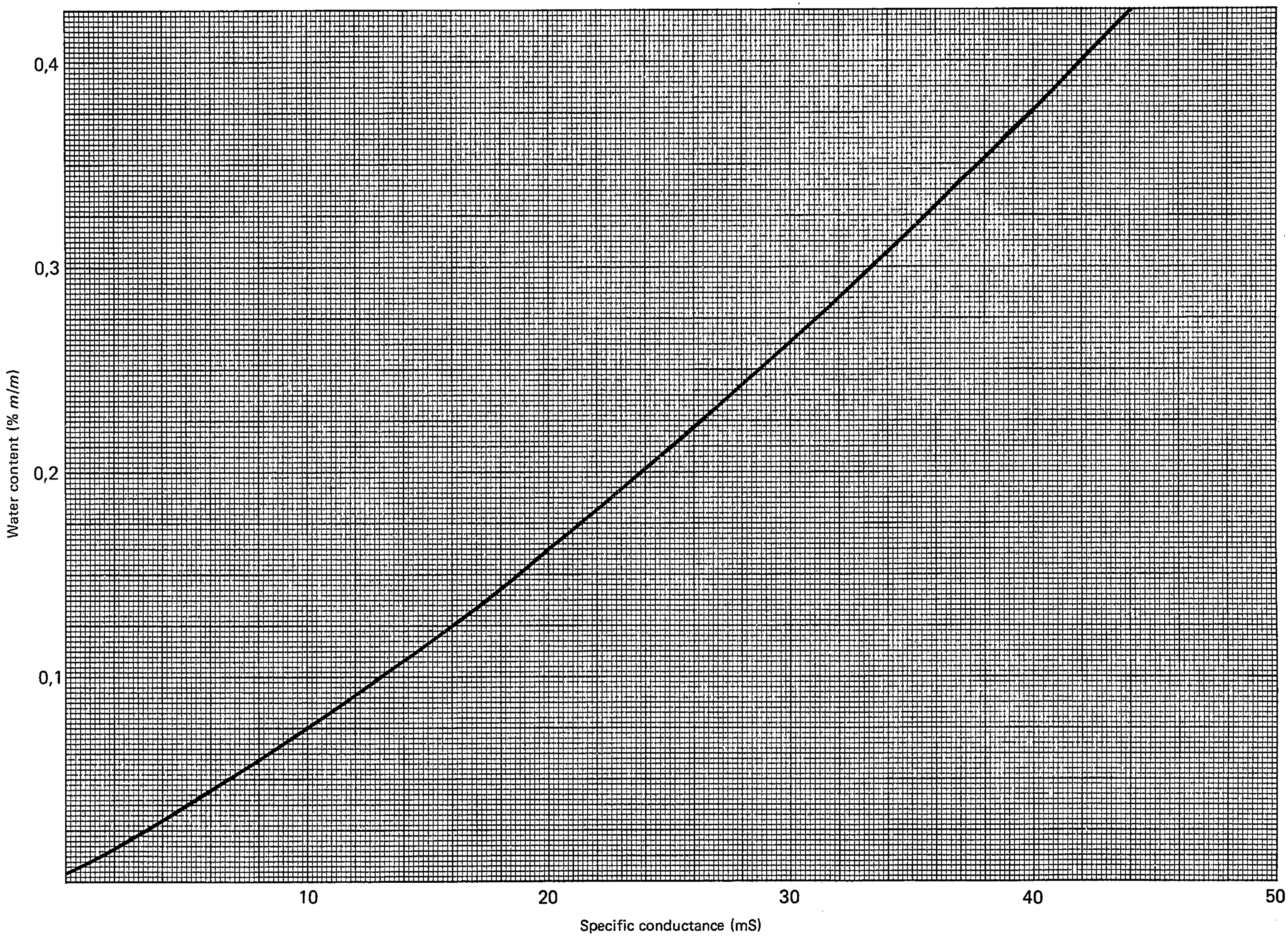

Figure 4 — Graph of specific conductance against water content

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.