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International Standard 7105

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Liquefied anhydrous ammonia for industrial use — Determination of water content — Karl Fischer method

Ammoniac anhydre liquéfié à usage industriel — Dosage de l'eau — Méthode de Karl Fischer

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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INTERNATIONAL STANDARD ISO 7105-1985 (E)

Liquefied anhydrous ammonia for industrial use — Determination of water content — Karl Fischer method

WARNING — Liquefied anhydrous ammonia is a highly corrosive, toxic substance, which boils at − 33,3 °C at standard atmospheric pressure. Its action on the skin is strongly corrosive, producing severe and painful burns. Contact with the eyes can cause permanent blindness.

Its vapour is strongly irritant to the mucous membrane and eyes, and produces a suffocating effect on the respiratory tract.

In concentrations of 16 to 25 % (*V*/*V*), gaseous anhydrous ammonia forms explosive mixtures with air.

Personnel responsible for handling the product shall be fully informed as to its dangerous character and the precautions to be taken.

Operators shall wear thick rubber gloves, a rubber apron and full face and head protection, and shall be provided with a protective gas-mask fitted with a filter for ammonia.

The operations described shall be carried out only in a well-ventilated fume cupboard.

For further information, see the appropriate sections of ISO 3165.

1 Scope and field of application

This International Standard specifies the Karl Fischer direct electrometric method for the determination of the water content of liquefied anhydrous ammonia for industrial use.

The method is applicable to products having water contents equal to or greater than 50 mg/kg.

NOTE — For water contents greater than 1 000 mg/kg, it is preferable to dilute the evaporation residue with anhydrous methanol in accordance with ISO 4276 and titrate an aliquot portion of the diluted solution.

2 References

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling.*

ISO 4276, *Anhydrous ammonia for industrial use — Evaluation of residue on evaporation — Gravimetric method.*

ISO 7103, *Liquefied anhydrous ammonia for industrial use — Sampling — Taking a laboratory sample.*

3 Principle

Evaporation of a test portion in the presence of ethanediol and determination of the water content of the residue by the Karl Fischer direct electrometric method.

4 Reagents and materials

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

Reagents and materials given in clause 4 of ISO 760, and in addition:

4.1 Freezing mixture, consisting of a mixture of solid carbon dioxide and methanol (technical grade is suitable), capable of reaching a temperature of between − 35 and − 40 °C.

NOTE — Technical grade acetone may be used instead of methanol.

4.2 Sulfuric acid, (ϱ approximately 1,84 g/ml), approximately 10 % (m/m) solution.

4.3 1,2-Ethanediol (ethyleneglycol) ($CH_2OH\text{-}CH_2OH$), having a water content not greater than 0,1 % (m/m).

NOTE — 1,2-Ethanediol is very hygroscopic; prevent absorption of atmospheric moisture.

4.4 Methanol/acetic acid mixture

Mix 100 ml of glacial acetic acid, ϱ approximately 1,05 g/ml, with 900 ml of methanol having a water content not greater than 0,03 % (m/m).

4.5 Methyl red, 1 g/l solution in 95 % (V/V) ethanol.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Apparatus specified in sub-clause 5.1 of ISO 760.

5.2 Apparatus for the sampling of the test portion

A typical example of the apparatus is shown in the figure and consists of the following items:

5.2.1 Glass test tube, with total capacity approximately 150 ml, having a 100 ml reference mark, fitted with a 24/29 ground glass stopper to an arm with a three-way stopcock (3) and a side-arm for connecting it in series to two conical flasks (A and B), each of 1 000 ml capacity.

In the assembled apparatus, the test tube is thus linked to two three-way stopcocks (3 and 4) permitting it to be connected either to the cylinder containing the liquefied anhydrous ammonia or to the two conical flasks (A and B).

The stopcocks are either lubricated with silicone grease or made of polytetrafluoroethylene (PTFE).

5.2.2 Dewar vessel, capable of containing the test tube (5.2.1), leaving the reference mark visible.

6 Sampling

Use the procedure specified in ISO 7103.

7 Procedure

WARNING — Carry out all operations in a well-ventilated fume cupboard.

7.1 Test portion

Weigh, to the nearest 0,1 g, the assembly comprising the two conical flasks (A and B), each containing approximately 500 ml of the sulfuric acid solution (4.2) and several drops of the methyl red solution (4.5), together with the connecting tubes shown in the figure, starting at point 5.

Immerse the unstoppered test tube (5.2.1) in the Dewar vessel (5.2.2), three-quarters filled with the freezing mixture (4.1). Place 2 ml of the 1,2-ethanediol (4.3) in the test tube, stopper it and connect it to the conical flasks.

Adjust stopcock 3 so as to isolate the test tube and to connect arms 1 and 2 to the atmosphere.

Connect arm 1 to the cylinder containing the laboratory sample, using rubber tubing. Carefully open the valve of the cylinder and allow the ammonia to escape slowly into the atmosphere, until arms 1 and 2 are well cooled and the ammonia emerges in the form of droplets.

Immediately adjust stopcock 3 so as to isolate arm 2, at the same time connecting arm 1 with the test tube and leaving arm 6 open by means of stopcock 4.

Quickly adjust stopcock 4 so as to connect the two conical flasks to the rest of the apparatus, taking care to isolate arm 6. The liquefied anhydrous ammonia is thus collected in the test tube, while any ammonia gas is absorbed by the sulfuric acid solution (4.2) contained in the two conical flasks.

As soon as the liquefied ammonia in the test tube reaches the 100 ml mark, adjust stopcock 4 so as to connect the test tube to the atmosphere and to isolate the two conical flasks.

Immediately adjust stopcock 3 so as to isolate the test tube and allow the ammonia gas to escape into the atmosphere through arm 2.

Then close the valve of the cylinder containing the laboratory sample and disconnect it from the apparatus.

As soon as the test portion has been obtained, disconnect the assembly comprising the two conical flasks and the connecting tubes shown in the figure, starting at point 5. Allow it to attain ambient temperature and weigh it, to the nearest 0,1 g.

7.2 Blank test

Carry out a blank test at the same time as the determination, using the same procedure and the same quantities of all the reagents (except the Karl Fischer reagent) as used for the determination, but omitting the test portion.

7.3 Determination

Remove the test tube containing the test portion from the Dewar vessel (5.2.2) and allow the ammonia to evaporate slowly at ambient temperature, through arm 2 (see the figure), until a residue consisting of a water/ethanediol solution is obtained at the bottom of the test tube. Unstopper the test tube, add 10,0 ml of the methanol-acetic acid mixture (4.4), ensuring that it runs slowly down the walls of the test tube. Stir carefully and transfer the solution quantitatively into the titration vessel specified in sub-clause 5.1.1.2 of ISO 760, rinsing the test tube using 10,0 ml portions of the methanol/acetic acid mixture, to a maximum total of 50,0 ml, and collecting the washings quantitatively in the titration vessel.

Titrate as specified in sub-clause 7.2 of ISO 760 (direct electrometric titration).

8 Expression of results

The water content, expressed as a percentage by mass of H_2O, is given by the formula

$$\frac{T(V_1 - V_2)}{m \times 10}$$

where

T is the equivalent in water of the Karl Fischer reagent (4.5 of ISO 760), calculated in accordance with sub-clause 7.3.1 of ISO 760;

V_1 is the volume, in millilitres, of the Karl Fischer reagent used for the titration;

V_2 is the volume, in millilitres, of the Karl Fischer reagent used for the blank test;

m is the mass, in grams, of the test portion (7.1). This is the sum of the volumes, in millilitres, of the liquefied anhydrous ammonia collected in the test tube multiplied by 0,68 (0,68 g/ml being the density of liquefied anhydrous ammonia), and the increase in mass, in grams of the assembly comprising the two conical flasks and the connecting tubes shown in the figure, starting at point 5.

9 Test report

The test report shall include the following particulars:

a) an identification of the sample;

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 in the International Standards to which reference is made, or regarded as optional.

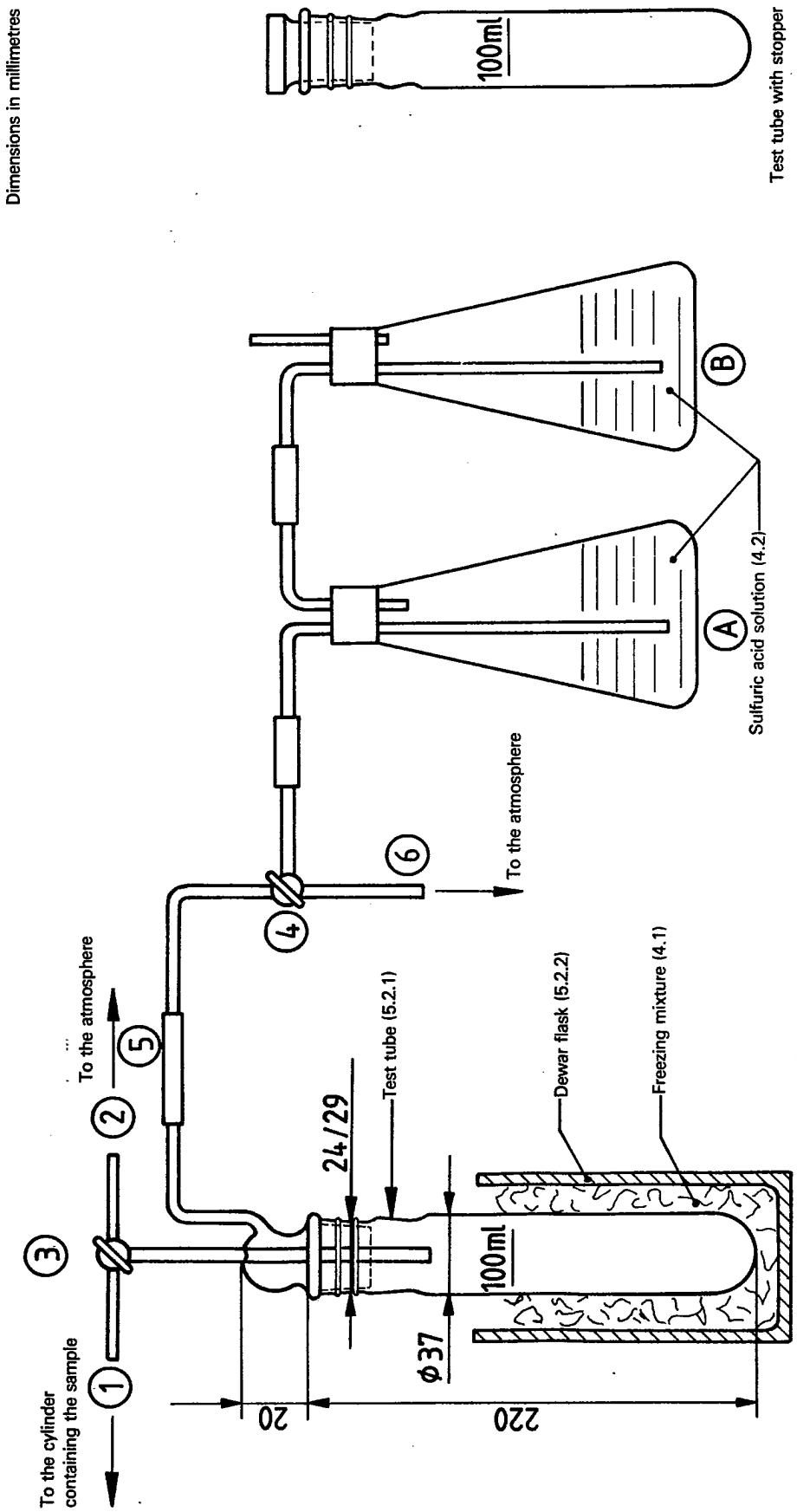

Figure — Typical apparatus for the sampling of the test portion