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INTERNATIONAL STANDARD



4276

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## Anhydrous ammonia for industrial use — Evaluation of residue on evaporation — Gravimetric method

*Ammoniac anhydre à usage industriel — Évaluation du résidu à l'évaporation — Méthode gravimétrique*

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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 4276 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in November 1975.

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

Austria	India	Switzerland
Belgium	Israel	Thailand
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Czechoslovakia	New Zealand	U.S.S.R.
France	Poland	Yugoslavia
Germany	Romania	
Hungary	South Africa, Rep. of	

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

Netherlands

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

# Anhydrous ammonia for industrial use – Evaluation of residue on evaporation – Gravimetric method

**WARNING** – Liquefied anhydrous ammonia is a highly corrosive, toxic substance which boils at  $-33,3^{\circ}\text{C}$  at standard atmospheric pressure. Its action on the skin and eyes is strongly corrosive, producing severe and painful burns.

Its vapour is strongly irritant to the mucous membranes 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 adequately instructed and fully informed as to its dangerous character.

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

Samples shall be handled only inside a well-ventilated fume cupboard.

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the evaluation of the residue on evaporation of anhydrous ammonia for industrial use.

The method is applicable to products leaving a residue on evaporation equal to or greater than 0,02 % (m/m).

NOTE – For products obtained in modern plants, leaving a residue on evaporation less than the limit indicated, the evaluation specified in this International Standard is not usually required. If it is carried out, it is sufficient to state that the result obtained is less than 0,02 % (m/m).

## 2 REFERENCE

ISO . . . , *Anhydrous ammonia (liquefied) for industrial use – Taking of a laboratory sample.*<sup>1)</sup>

## 3 PRINCIPLE

Evaporation, at ambient temperature, of a test portion of liquefied anhydrous ammonia, and weighing of the residue.

Titration of the ammonia contained in the residue with a standard volumetric sulphuric acid solution in the presence of an indicator.

Subtraction of the mass of ammonia determined by titration from that of the residue.

## 4 REAGENTS

During the analysis, use only reagents of recognized

analytical reagent grade and only distilled water or water of equivalent purity.

**4.1 Cooling mixture**, consisting of solid carbon dioxide and technical grade methanol, in proportions allowing a temperature between  $-35$  and  $-40^{\circ}\text{C}$  to be attained.

As an alternative to methanol, technical grade acetone may be used.

**4.2 Sulphuric acid**, approximately 2 N solution.

**4.3 Sulphuric acid**, 0,1 N standard volumetric solution.

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

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Glass test tube**, of total capacity about 150 ml, fitted with a ground glass or polytetrafluoroethylene (PTFE) 24/29 stopper and having a graduation mark at 100 ml (mass of test tube and stopper about 110 g).

The test tube may be connected to two three-way stopcocks (3 and 4) which allow it to communicate on one side with the vessel containing the anhydrous ammonia and on the other side with two conical flasks (A and B), of capacity 1 000 ml, connected in series.

The stopcocks are greased with silicone or made of PTFE.

1) In preparation.

**5.2 Dewar flask** capable of containing the test tube (5.1).

An example of an assembled apparatus is shown in the figure.

**6 PROCEDURE**

Carry out all the following operations in a well-ventilated fume cupboard.

**6.1 Test portion**

Weigh, on a technical balance, to the nearest 0,1 g, the two conical flasks (A and B), each containing about 500 ml of the sulphuric acid solution (4.2) and a few drops of the methyl red solution (4.4), together with the connecting tubes as from the point 5 in the figure.

Weigh the stoppered test tube (5.1), to the nearest 0,000 1 g, on an analytical balance, immerse it in the Dewar flask (5.2) three-quarters full of the cooling mixture (4.1) and, after removing the stopper, connect it to the conical flasks.

Turn stopcock 3 so as to close off the test tube and open tubes 1 and 2 to atmosphere.

Connect tube 1 to the vessel containing the laboratory sample, taken according to the requirements of ISO . . . , using a rubber tube. Carefully open the sample valve, allowing the ammonia vapour to escape slowly to atmosphere until tubes 1 and 2 are well cooled and the ammonia begins to emerge in droplet form.

At this point, turn stopcock 3 so as to close off tube 2 and connect tube 1 to the test tube, leaving tube 6 open, by means of the stopcock 4.

At almost the same time, turn stopcock 4 so as to connect the two conical flasks with the rest of the apparatus, taking care to close off tube 6.

The anhydrous liquid ammonia is thus collected in the test tube, while the vapour released is absorbed by the sulphuric acid solution (4.2) contained in the two conical flasks.

When the liquefied ammonia collected in the test tube reaches the 100 ml graduation mark, turn stopcock 4 so as to connect the test tube to atmosphere, closing off the two conical flasks.

Immediately turn stopcock 3 so as to close off the test tube and allow the ammonia to escape to atmosphere through tube 2.

Then close the sampling valve of the vessel containing the anhydrous ammonia and disconnect the apparatus from it.

**6.2 Determination**

Remove the test tube and its contents from the Dewar flask and allow the ammonia to evaporate slowly at ambient temperature, until a residue is obtained composed of aqueous ammoniacal solution, oil and other substances non-volatile at ambient temperature.

Allow the test tube and contents to reach ambient temperature (approximately 20 °C) and then pass a gentle current of dry air (about 100 l/h) for 30 s. Stopper the test tube, wipe with a clean dry cloth and weigh it with its contents to the nearest 0,000 1 g.

Add to the test tube 50 ml of water and a few drops of the methyl red solution (4.4). Swirl and titrate with the sulphuric acid solution (4.3) until the indicator changes from yellow to red.

Reweigh, to the nearest 0,1 g, the two conical flasks and associated tubing after they have reached ambient temperature.

**7 EXPRESSION OF RESULTS**

The residue on evaporation is given, as a percentage by mass, by the formula

$$\frac{m_2 - m_1 - m_3}{m_0} \times 100$$

where

$m_0$  is the mass, in grams, of the test portion (6.1) consisting of : the volume, in millilitres, of liquefied anhydrous ammonia collected in the test tube, multiplied by 0,68 (0,68 g/ml being the density of liquefied anhydrous ammonia) plus the increase in mass, in grams, of the two conical flasks and associated tubing;

$m_1$  is the mass, in grams, of the empty stoppered test tube (5.1);

$m_2$  is the mass, in grams, of the stoppered test tube containing the residue after evaporation;

$m_3$  is the mass, in grams, of ammonia in the residue after evaporation, determined by titration, calculated using the formula

$$V \times 0,001 7$$

where  $V$  is the volume, in millilitres, of the sulphuric acid solution (4.3) used for the titration.

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

**8 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.

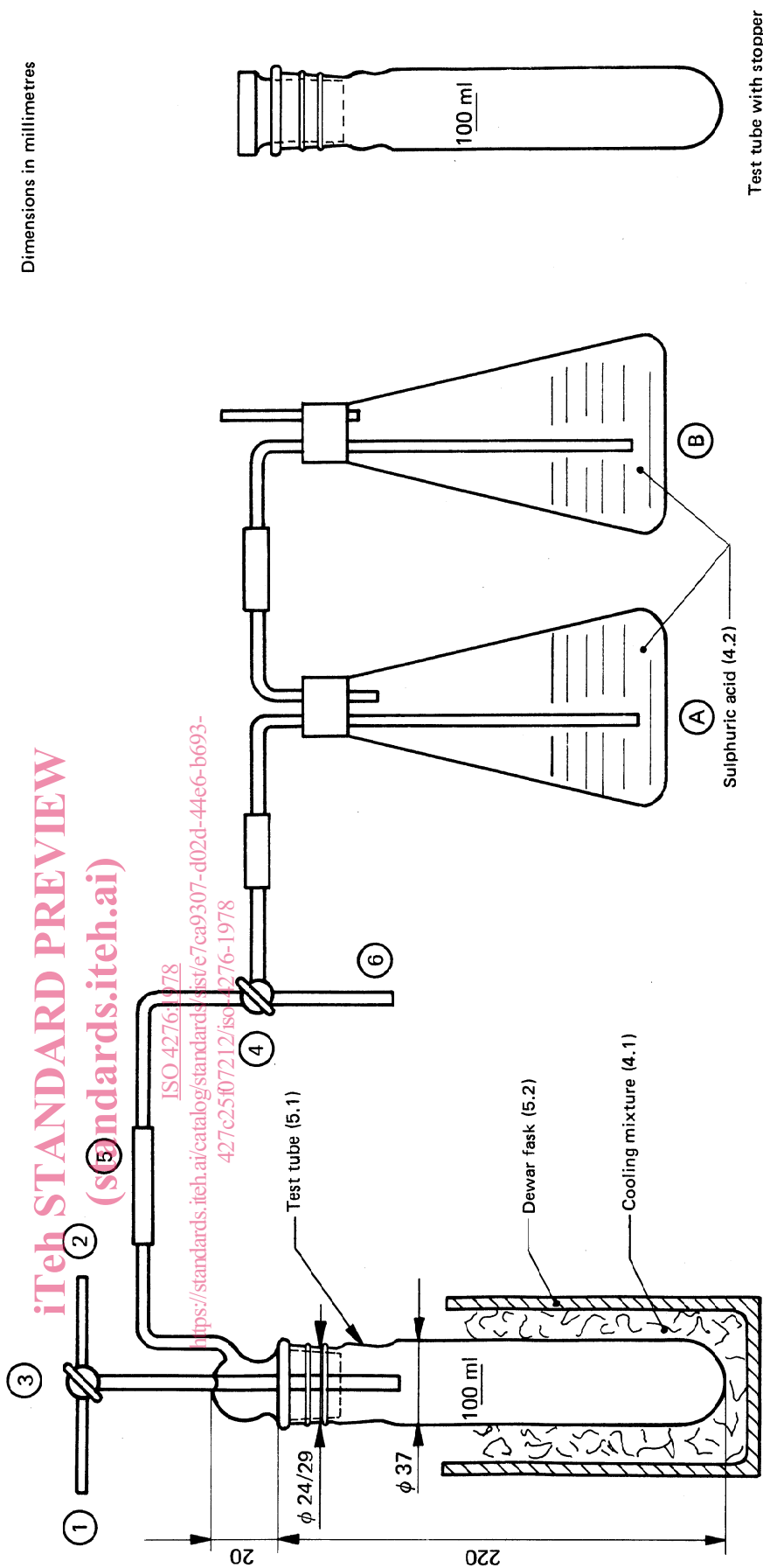


FIGURE — Typical apparatus for the evaluation of residue on evaporation of anhydrous ammonia

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