

भारतीय मानक
Indian Standard

IS 662 : 2020

निर्जल अमोनिया — विशिष्टि
(दूसरा पुनरीक्षण)

**Anhydrous Ammonia —
Specification**
(*Second Revision*)

ICS 71.060

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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This standard (Second Revision) was first published in 1955 and revised in 1980. The Sectional Committee decided to revise it in the light of actual requirements of the industry. In the first revision the requirements of pyridine, naphthalene, phosphine, sulphur compounds and phenolic compounds were dropped, and the requirements of moisture and oil content were added. In this revision, requirements of oxygen and carbon dioxide along with their method of test have been added. In addition, purity of anhydrous ammonia in terms of percentage by mass has also been incorporated.

By convention, the name 'anhydrous ammonia' is used to describe commercial grades of the product containing approximately 99.90 to 99.98 percent of ammonia (*see 3.1*).

This standard does not contain aspects of safety for handling and transport of ammonia as this is covered under IS 4544:1968.

In the preparation of this standard, reference has been made to 'BS: 4431-1969 Methods of test for anhydrous ammonia', issued by the British Standards Institution.

This standard was formulated by CHD 01 technical committee. The list of experts who had made significant contribution to the formulation of this standard is given at Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ANHYDROUS AMMONIA — SPECIFICATION

(*Second Revision*)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for anhydrous ammonia. The material is used for refrigeration purposes, and also for other industrial purposes such as for the manufacture of nitric acid, in the preparation of liquor ammonia, etc.

1.1.1 This standard document does not cover the requirements of anhydrous ammonia for direct application to soil which are being covered under separate Indian Standard.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (<i>third revision</i>)
1745 : 2018	Petroleum hydrocarbon solvents — Specification (<i>third revision</i>)
2362 : 1993	Determination of water by Karl Fischer method — Test method (<i>second revision</i>)
4544 : 2000	Ammonia — Code of safety (<i>first revision</i>)
5194 : 1969	Method for determination of nitrogen — Kjeldahl method

3 TERMINOLOGY

For the purpose of this standard, anhydrous ammonia refers to the commercial grade of ammonia gas containing 99.90 to 99.98 percent of ammonia. By definition anhydrous ammonia (NH₃) means high purity ammonia without water ('Anhydrous'). Anhydrous ammonia or ammonia is a colourless gas at room temperature and pressure with a strong and pungent odour. Ammonia has a boiling point of approximately negative thirty three degree centigrade (-33°C) at one atmospheric pressure, is partially soluble in water, and is considered both caustic and hazardous.

4 GRADES

4.1 The material shall be of two grades, namely:

- a) *Grade 1* — For refrigeration purposes and other uses where a relatively purer material is essential; and
- b) *Grade 2* — For miscellaneous industrial uses, such as for the manufacture of nitric acid, for the preparation of liquor ammonia, etc.

5 REQUIREMENTS

5.1 Description — The material shall consist essentially of anhydrous ammonia free from foreign matter and visible impurities.

5.2 The material shall comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the table.

6 PACKING AND MARKING

6.1 The material shall be supplied as liquid in cylinders or tank cars of approved design.

6.1.1 The cylinders shall conform to the *Gas Cylinder Rules* 2016 of the Government of India with such modifications as may be ordered from time to time by the Chief Controller of Explosives, Government of India, or other duly constituted authority. The tank cars shall conform to the specification as approved by the appropriate authorities of Indian Railways or other duly constituted authority.

6.2 Packing, marking, painting and transport of cylinders shall be in accordance with the requirements of the notification cited above with such modifications as may be ordered from time to time by the Chief Controller of Explosives, Government of India, or other duly constituted authority.

6.2.1 *Marking*

Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name and description of the material;
- b) Name of the manufacturer and/or his recognized trade-mark, if any;

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- c) Net mass of the material;
- d) Date of filling/packaging of container/cylinder; and
- e) Lot or batch number, in code or otherwise.

For supplies of anhydrous ammonia in tankers, a test report containing the following additional information shall be provided for each tanker:

- 1) Name and grade of the material.
- 2) Name of the manufacturer.
- 3) Supply date.
- 4) Tanker number.
- 5) Quantity.

The test report shall be certified by authorized person of the manufacturer's organization.

6.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity

assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

7 SAMPLING

Representative samples of the material shall be drawn in accordance with the method prescribed in Annex B.

8 TEST METHODS

8.1 Tests shall be carried out according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of Table 1.

8.2 Quality of Reagents

8.2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Anhydrous Ammonia
(Clause 5.2)

SI No.	Characteristic	Requirement		Method OE TEST (Ref To CL No. or Annex A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Residue on evaporation, percent by mass, <i>Max</i>	0.02	0.5	A-2
ii)	Moisture, percent by mass, <i>Max</i>	0.2	0.5	A-3
iii)	Oil, ppm by mass, <i>Max</i>	2.0	10	A-4
iv)	Oxygen (ppm, <i>Max</i>)	1.0	1.0	A-5
v)	Carbon dioxide (ppm, <i>Max</i>)	1.0	1.0	A-6
vi)	Purity (percent by mass, as NH ₃ , <i>Min</i>)	99.95	99.5	A-7

ANNEX A

(Clause 5.2)

METHODS OF TEST FOR ANHYDROUS AMMONIA

A-1 GENERAL

Methods described in this standard indicate that the ammonia is sampled by volume but the calculations are made on mass basis. The density has been taken as 0.68 g/ml, the value at about -33°C, at which temperature liquid ammonia has a vapour pressure of 1 atmosphere absolute.

A-2 DETERMINATION OF RESIDUE ON EVAPORATION

A-2.1 Outline of the Method

The sample is allowed to evaporate from a tared flask and the mass of the residue obtained is determined. This residue will include water, oil and other non-volatile substances

under the conditions of the test.

A-2.2 Apparatus

A-2.2.1 Conical Flask — 250 ml calibrated to hold 150 ml (about 100 g) of sample and with a bung fitted with a glass tube through which the exit gas can be led away to a safe place. Ensure that the glass tube and exit line are free from constrictions.

A-2.3 Procedure

Weigh the calibrated conical flask to the nearest milligram. Quickly run in the sample (*see* Annex B) up to the calibration mark and insert the bung. Immerse the flask in a continuous stream of cold water and allow the ammonia to evaporate slowly. When the evaporation is complete, remove the bung briefly and gently blow out the residual ammonia vapour with a small jet of dry, filtered air. Dry the outside of the flask thoroughly and weigh again to the nearest milligram.

A-2.4 Calculation

Residue on evaporation percent by mass =

$$\frac{M_1 - M_2}{0.68 V} \times 100 F$$

where

M_1 = mass in g, of the flask and residue;

M_2 = mass in g, of the flask;

V = volume in ml, of the sample taken; and

F = evaporation factor (*see* Annex C).

A-3 DETERMINATION OF MOISTURE

A-3.1 General

Two methods have been prescribed below. The appropriate method shall be selected depending upon the moisture content.

A-3.2 Method A (For Low Moisture Content)

A-3.2.1 Applicability

This method shall be used for anhydrous ammonia with water content under 200 parts per million.

A-3.2.2 Outline of the Method

A small volume of ethanediol is added to the vessel in which the sample is to be measured in order to retain the water. The ammonia is allowed to evaporate, the process being assisted by reduced pressure in the later stages. A solution of acetic acid in methanol is added to neutralize any residual ammonia and the water is then titrated by Karl Fischer method as described in IS 2362.

A-3.2.3 Apparatus

A-3.2.3.1 Sample vessel — As shown in Fig. 1A. It is marked to hold 100 ml of sample.

A-3.2.3.2 Guard tube — As shown in Fig. 1B.

A-3.2.3.3 Vaccine cap — with small slit for burette tip, to close the sample vessel.

A-3.2.3.4 Dewar-flasks (D) — two.

A-3.2.3.5 Manometer (E) — capable of measuring absolute pressures up to 760 mm Hg.

A-3.2.3.6 Drying tube (F) — containing phosphorus pentoxide.

A. 3.2.3.7 Suction pump (G) — fitted with a trap to prevent ammonia entering the pump.

A-3.2.3.8 Three-way tap (H).

A-3.2.3.9 Tee-pieces — two.

A-3.2.3.10 Titration assembly — as shown in Fig. 1 of IS 2362. The sampling apparatus is assembled as shown in Fig. 2.

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A-3.2.4 Reagents

A-3.2.4.1 *Ethanediol* — Preferably containing less than 0.1 percent of water.

NOTE — Ethanediol is hygroscopic and care should be taken to avoid absorption of water from the atmosphere.

A-3.2.4.2 *Methanolic solution of acetic acid* — Mix 100 ml of glacial acetic acid with 900 ml of methanol with water content less than 0.03 percent.

A-3.2.4.3 *Karl Fischer reagent* — Having a water equivalent of about 3.5 mg/ml, prepared as prescribed in IS 2362.

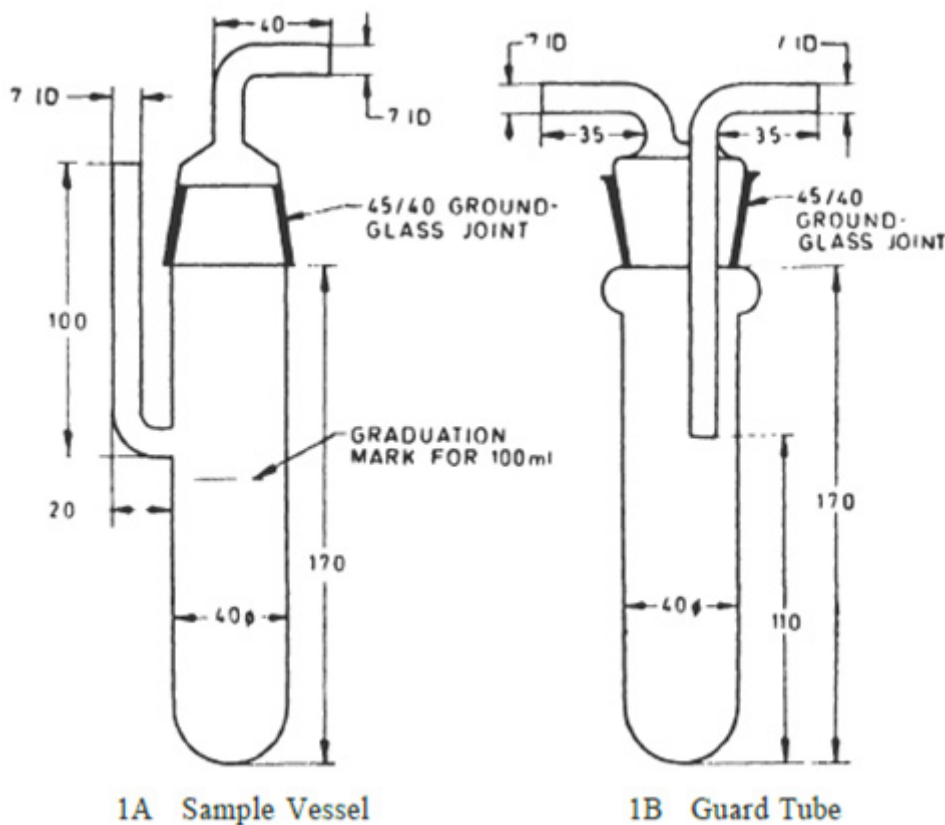
A-3.2.4.4 *Cooling mixture* — Solid carbon dioxide in methanol.

A-3.2.5 Procedure

Standardize the Karl Fischer reagent as described in 6.1.1 of IS 2362. Assemble the apparatus as shown in Fig. 2. Before connecting the ammonia cylinder to the sample vessel purge the valve and sampling tube with about 1 litre of ammonia in the liquid form. Ensure that the ammonia is purged to a safe place. Quickly dry the

end of the tube (for example, with clean absorbent paper such as filter paper) and then immediately connect the sampling tube to the sample vessel.

A-3.2.5.1 With the air leak closed and tap *H* closed to the drying tube, evacuate the apparatus up to the valve of the cylinder, with the guard tube *B* (but not the sample vessel *A*) immersed in the cooling mixture contained in Dewar flask. After about 5 min open the tap *H* and allow air to pass into the apparatus through the drying tube until atmospheric pressure is attained. Open the air-leak and shut off the suction pump. Close the drying tube to atmosphere. Remove the ground glass cowl of the sample vessel *A*, and add as quickly as possible 2 ml of the ethane diol. Replace the cowl. Place the sample vessel in the other Dewar flask. Fill the Dewar flask with the cooling mixture. After the sample vessel has become thoroughly cooled, very carefully open the valve of the ammonia cylinder and collect 100 ml of sample in vessel *A* at such a rate that not more than about 2 ml of sample collects in the guard tube *B*. Close the cylinder valve again. Remove the Dewar flask from the sample vessel and replace with a beaker of methanol. Allow the ammonia to evaporate,



All dimensions in millimetres

FIG. 1 GLASSWARE FOR THE DETERMINATION OF WATER ANHYDROUS AMMONIA

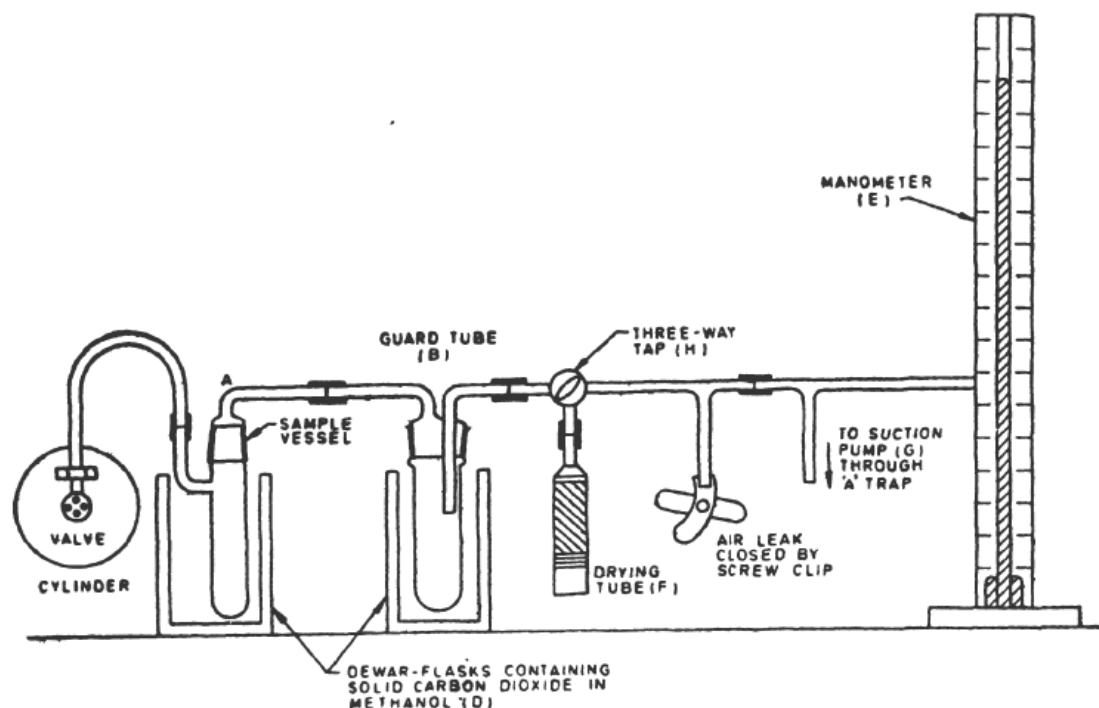


FIG. 2 APPARTUS ASSEMBLED FOR SAMPLING ANHYDROUS AMMONIA FOR THE DETERMINATION OF WATER

most of it will condense in *B*, but some will escape from the air-leak. It may be necessary to adjust the temperature of the methanol to speed up evaporation.

A-3.2.5.2 When all the ammonia seems to have evaporated, close the air-leak and reduce the pressure in the apparatus to 150 mm Hg for 15 min by means of the suction pump. At the end of this time allow air to pass through the drying tube into the apparatus to restore atmospheric pressure. Disconnect the sample vessel *A* from the sampling train, and close its die side-arm with a rubber stopper. Measure 10 ml of the methanolic acetic acid solution into a small flask, quickly run in the Karl Fischer reagent just to the end point and then transfer it to the sample vessel *A*. Stopper the sample vessel and shake gently to wash down the ethane diol from the sides of the tube. Fit the sample vessel with the vaccine cap and titrate the contents with Karl Fischer reagent just to the end point, as described in IS 2362. Note the volume of the reagent used. Without delay add 2 ml of the ethane diol to the sample vessel and again titrate with the Karl Fischer reagent just to the end point, as described in IS 2362. Again note the volume of the reagent used.

A-3.2.6 Calculation

$$\text{Water content, percent by mass} = \frac{W(V_1 - V_2) \times 10^3 \times F}{0.68V_3}$$

where

W = water equivalent in mg/ml of the Karl Fischer reagent;

*V*₁ = volume in ml, of Karl Fischer reagent required for the first titration;

*V*₂ = additional volume in ml, of Karl Fischer reagent required for the second titration;

*V*₃ = volume in ml, of sample taken; and

F = evaporation factor (see Annex C).

A-3.3 Method B (for Higher Moisture Content)

A-3.3.1 Applicability

This method shall be used for anhydrous ammonia with higher moisture content (more than 200 parts per million).

A-3.3.2 Outline of the Method

This method is rapid routine method which is suitable for the determination of water at the higher concentration. The liquid ammonia is evaporated in the presence of a small volume of ethanediol which retains the water. A solution of acetic acid in methanol is then added to neutralize any residual ammonia and the water is titrated by the Karl Fischer method (see IS 2362).

A-3.3.3 Apparatus

A.3.3.3.1 Conical flask — 250 ml calibrated to hold 150 ml (about 100 g) of the sample and with a bung fitted with a glass tube through which the exit gas can be led away to a safe place. Ensure that the glass tube and exit line are free from constrictions.

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A-3.3.3.2 Titration assembly — As shown in Fig. 1 of IS 2362.

A-3.3.4 Reagents

A-3.3.4.1 Ethanediol — Preferably containing less than 0.1 percent of water.

NOTE — Ethanediol is hygroscopic and care should be taken to avoid absorption of water from the atmosphere.

A-3.3.4.2 Methanolic acetic acid solution — Mix 100 ml of glacial acetic acid with 900 ml of methanol with moisture content less than 0.03 percent.

A-3.3.4.3 Karl Fischer reagent — Having a water equivalent of about 3.5 mg/ml, prepared (see IS 2362).

A-3.3.5 Procedure

Transfer to the clean, dry conical flask 2.0 ml of the ethanediol, quickly run in the sample (see Annex B) up to the calibration mark and insert the bung. Immerse the flask in a continuous stream of cold water, taking care not to allow any water to enter the flask, and allow the ammonia to evaporate slowly. Remove the bung and gently blow out the residual ammonia with a small jet of dry air.

A-3.3.5.1 Measure 10 ml of the methanolic acetic acid solution into a small flask and quickly run in the Karl Fischer reagent just to the end point, as described in IS 2362.

A-3.3.5.2 Without delay, transfer this titrated solution to the sample flask and then titrate the contents with the Karl Fischer reagent. Note the volume of the reagent used. Immediately add 2 ml of the ethanediol to the sample vessel and again titrate to the end point as described in IS 2362. Again note the volume of the reagent used.

A-3.3.6 Calculation

Moisture content, percent by mass =

$$\frac{W(V_1 - V_2) \times 10^3 \times F}{0.68V_3}$$

where

W = water equivalent in ml/g, of the Karl Fischer reagent;

V_1 = volume in ml, of Karl Fischer reagent required for the first titration;

V_2 = additional volume in ml, of Karl Fischer reagent required for the second titration;

V_3 = volume in ml, of sample taken; and

F = evaporation factor (see Annex C).

A-4 DETERMINATION OF OIL CONTENT

A-4.1 General

Two methods have been prescribed namely, gravimetric method and spectrophotometric method. The spectrophotometric method is preferred and shall be used if an infra-red spectrophotometer is available. The gravimetric method is not suitable for oil contents below 30 parts per million, but improved precision can be obtained for low contents by using larger samples.

A-4.2 Gravimetric Method

A-4.2.1 Outline of the Method

The sample is allowed to evaporate from a flask, any oil in the residue is dissolved in petroleum spirit, and the solution evaporated to dryness in a platinum dish.

A-4.2.2 Apparatus

A-4.2.2.1 Conical flask — 500 ml calibrated to hold 300 ml (about 200 g) of sample and with a bung fitted with a glass tube through which the exit gas can be led away to a safe place. Ensure that the glass tube and exit line are free from constrictions.

A-4.2.2.2 Platinum dish — 75 mm in diameter.

A-4.2.3 Reagent

A-4.2.3.1 Petroleum spirit — Boiling range 40°C to 60°C (see IS 1745).

A-4.2.4 Procedure — Quickly run in the sample (see Annex B) up to the calibration mark and insert the bung.

A-4.2.4.1 Immerse the flask in a continuous stream of cold water and allow the ammonia to evaporate slowly. When the evaporation is complete, remove the bung and gently blow out the last traces of ammonia with a small jet of filtered air. Dry the outside of the flask. Place the flask in an oven at 105 ± 1°C for 15 min to remove the moisture, blowing out the last traces with a gentle stream of filtered air at the end of this period, allow to cool.

A-4.2.4.2 Add to the flask approximately 10 ml of the petroleum spirit and swirl to dissolve the oil. Filter the solution through a small filter paper (Whatman No. 41 or equivalent) directly into the platinum dish previously tared to the nearest 0.1 mg. Repeat the operation with two more successive 10 ml portions of petroleum spirit, filtering through the same paper as before into the same dish.

A-4.2.4.3 Evaporate the combined extracts to dryness on a water bath in a fume cupboard, taking care to avoid naked lights, and finally dry in an oven at $105 \pm 1^\circ\text{C}$ for 30 min. Allow the dish to cool thoroughly in a desiccator and weigh again to the nearest 0.1 mg.

A-4.2.4.4 At the same time carry out a blank determination on the petroleum spirit and filter paper. Make an appropriate correction in the calculation.

A-4.2.5 Calculation

Oil content, parts per, million by mass =

$$\frac{(M_1 - M_2 - M_3)}{0.68 V} \times 10^6 \times F$$

where

M_1 = mass in g, of platinum dish and residue;

M_2 = mass in g, of platinum dish alone;

M_3 = mass in g, of oil in blank determination;

V = volume in ml, of sample taken; and

F = evaporation factor (*see* Annex C).

A-4.3 Spectrophotometric Method

A-4.3.1 Outline of the Method

The sample is evaporated and any oil present is dissolved in carbon tetrachloride. The oil content of the solution is then measured photometrically at 346 nm (the C-H stretching frequency) using an infra-red spectrophotometer.

NOTE — This method is very sensitive and great care is necessary to avoid contamination of any part of the apparatus with oil or grease.

A-4.3.2 Apparatus

A-4.3.2.1 Infra-red spectrophotometer and accessories

A-4.3.2.2 Conical flask — 500 ml capacity, calibrated to hold 300 ml (about 200 g) of sample and with a bung fitted with a glass tube through which the exit gas can be led away to a safe place. Ensure that the glass tube and exit lines are free from constrictions.

A-4.3.3 Reagents

A-4.3.3.1 Carbon tetrachloride — Redistilled. Check that each fresh supply of this reagent shows no absorption at 346 nm.

A-4.3.3.2 Standard oil solution — 0.5 g/l of liquid paraffin in carbon tetrachloride. One millilitre of this solution is equivalent to 500 µg of oil.

A-4.3.4 Procedure

A-4.3.4.1 Preparation of test solution — Thoroughly clean the 500 ml conical flask so that it is oil-free. Quickly run in the sample up to the calibration mark

and insert the bung. Immerse the flask in a continuous stream of cold water and allow the ammonia to evaporate slowly. When the evaporation is complete, remove the bung and gently blow out the last traces of ammonia with a small jet of filtered air. Dry the outside of the flask.

Place the flask in an oven at $105 \pm 1^\circ\text{C}$ for 15 min to remove the moisture, blowing out the last traces with a gentle stream of filtered air at the end of this period, allow to cool. Rinse out the flask with several 10 ml portions of the carbon tetrachloride and combine these washings in a 100 ml volumetric flask. Dilute to the mark with carbon tetrachloride and mix well. Any other suitable dilution may be made at this stage and allowed for in the calculation.

A-4.3.4.2 Preparation of calibration chart

In a series of 100 ml volumetric flasks prepare suitable dilutions of the standard oil solution to cover the range of values within which the mass of oil in the sample is expected to be found. Measure the optical densities of this series of solutions at the wavelength of maximum absorption (approximately 3.46 µm) with the carbon tetrachloride as the reference liquid.

Prepare a calibration chart by plotting the mass of oil at the different dilutions against the corresponding optical densities.

A-4.3.4.3 Determination

Measure the optical density of the test solution at the wavelength used in the calibration with the carbon tetrachloride as the reference liquid. From the calibration chart determine the mass of oil present in the total volume of the test solution.

A-4.3.5 Calculation

Oil content, parts per, million by mass = $\frac{M}{0.68 V} \times F$

where

M = mass in g, of oil in the total volume of the test solution;

V = volume in ml, of sample taken; and

F = evaporation factor (*see* Annex C).

A-5 DETERMINATION OF OXYGEN

A-5.1 Scope

The method is useful for the determination of oxygen present in trace amount (from 0-10 ppm to 0-10,000 ppm).

A-5.2 Principle

Liquid ammonia is vapourized by passing it through stainless steel coil. Oxygen is passed further and diffused into the fuel cell of the analyzer. Oxygen reacts chemically and electric current is produced which is

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proportional to the concentration of oxygen in the gas phase. Further, signal produced by the fuel cell from the trace oxygen is amplified by two stage solid state amplifier.

A-5.3 Apparatus

A-5.3.1 Trace Oxygen Analyzer — Analytical instrument “Teledyne”, model 311-1 or similar is suitable. Such type of instruments are adapted for use with anhydrous ammonia.

A-5.3.2 Stainless Steel Coil

A-5.3.3 Sample Line — It is made of stainless steel with 3/8” outside diameter. It has a purge line and double isolation valve.

A-5.4 Calibration

Stand the analyzer on a flat level surface upright. Keep the range switch in OFF position. Check if the meter pointer is aligned with the zero mark on the scale. Keep the range switch set in “CAL” position. Arrange and install the plastic tube equipped male disconnect fitting to either of sample ports for analysis and in the other port install blank disconnect fitting. Suck air gently via plastic tube with the help of hand aspirator and observe the reading of the meter. Suck the air from the tube regularly till we get a stable reading. Meter pointer should coincide with the ‘CAL’ mark on the meter scale. Further, connect a source of inert gas with a trace amount of oxygen and allow it to purge at the rate of 5L/min till the reading is obtained at a low value of 10 ppm V/V. Purging should be continued to obtain such a low value.

A-5.5 Procedure

Purging: In order to determine oxygen in ammonia, purge an inert gas using a purge rate of 5L/min.

Withdraw disconnect fittings as soon as satisfactory low oxygen level is achieved. It is to be ensured that purge gas supply is removed first. Fuel cell should not be subjected to excessive pressure since leak in the cell may occur.

A-5.6 Analysis

With the help of stainless steel compression fitting, connect the coil to the sample point and immerse it in a container of warm water. Open the isolation valve carefully and purge sample line and coil free of air. With the help of suitable flow meter, establish a flow rate of 5 L/min. In order to ensure that the system is delivering a sample of pure ammonia, continue the purging for few minutes. Carefully connect disconnect fittings to the analyzer and make sure that the vent line is fitted before ammonia line. If all connections are made correctly, a result can be expected within 15 min of starting to pass ammonia. In order to maintain

the temperature for ammonia evaporation, periodically change the warm water in which the coil is dipped. At such low level of oxygen, the response of fuel cell to oxygen is slow. Direct reading of oxygen can be obtained in ppm. Purge ammonia from the instrument after the determination is complete.

A-5.7 Expression of Results

The concentration of oxygen in ammonia in ppm level can be given as:

$$A \times 32/17$$

where

A = the concentration of oxygen which is obtained directly from the analyzer (in ppm); and

32 = the relative molecular mass of oxygen and 17 is the relative molecular mass of ammonia.

A-6 DETERMINATION OF CARBON DIOXIDE

A-6.1 Principle

Sample is evaporated in the presence of excess of barium hydroxide solution. Back titration is carried out with excess of barium hydroxide which is titrated with standard hydrochloric acid using thymolphthalein indicator.

A-6.2 Reagents

A-6.2.1 Standard Hydrochloric Acid — 0.100 mol/l,

A-6.2.2 Thymolphthalein Indicator — Prepared in 95 percent ethanol.

A-6.2.3 Barium Hydroxide Solution (Concentration of 5 g/l).

A-6.3 Procedure

Prepare the test sample by adding to it excess of Barium Hydroxide solution. Take the sample and evaporate ammonia. Immerse the flask in a continuous stream of cold water and allow the ammonia to evaporate slowly. After the completion of evaporation, remove the bung and gently blow out the residual ammonia with a small jet of dry filtered air. After this, add carbon dioxide free water (10 ml) in the test tube. Remove the last traces of ammonia by heating on water bath. Close the test tube using rubber stopper which is fitted with soda lime. Allow it to cool. Back titrate excess of barium hydroxide against standard volumetric hydrochloric acid solution using thymolphthalein indicator. Titrate till the blue colour of the solution disappears.

A-6.4 Blank Test

Carry out the blank determination using the same procedure with same quantities of all reagents (except standard hydrochloric acid solution).

The amount of carbon dioxide expressed in mg/kg (ppm) is given as-

$$\frac{(A-B) \times D \times 1\,000 \times F}{G}$$

where

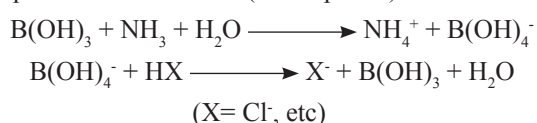
- A = Volume of HCl solution used for blank (in ml),
- B = Volume of hydrochloric acid solution used for the determination,
- D = is the mass of carbon dioxide corresponding to 1.00 ml of volumetric HCl solution,
- F = Evaporation factor; and
- G = Mass of test sample.

A-7 DETERMINATION OF PURITY

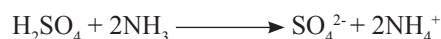
A-7.1 Principle

The reaction vessel is filled with absorbing acidic solution in order to capture ammonia gas. Used up acid is then converted back to free acid quantitatively by titrating against strong acid and concentration of Ammonia is determined.

Common absorbing solutions used are aqueous boric acid $[B(OH)_3]$ of 2-4 percent concentration. Ammonia is trapped by boric acid quantitatively forming solvated ammonium ions. Addition of strong acid converts this de-protonated boric acid (used up acid).



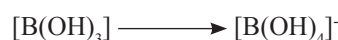
Alternately, acids such as sulphuric acid or hydrochloric acid can be used to capture ammonia and the excess can be titrated with a base.



This type of titration is known as back titration.

A-7.2 Procedure

Take about 50 ml of 4 percent boric acid solution to capture ammonia. Add about 6-7 drops of Tashiro's indicator. Tashiro's indicator has a pH around (4.4 - 6.2). It is a mixed indicator which is composed of a solution of 0.1 percent methylene blue and 0.03 percent methyl red in ethanol or methanol. Around 150 ml of ammonia condensate is captured in boric acid solution in 5 min. As soon as ammonia reacts with boric acid the solution turns from red violet to green due to change of medium from acidic to basic.



Titrate the solution with 0.25 mol/l of HCl till the solution has slightly violet colour. Using the volume and concentration of HCl, we can calculate the number of mol of Ammonia in the sample.

A-7.3 Calculation

$$\text{Purity (as } NH_3\text{), percent by mass} = \frac{1.703 \times V_1 \times N}{V_2 \times D}$$

where

- V_1 = is the volume of HCl taken, in mL;
- N = is the Normality of HCl taken;
- V_2 = Volume of ammonia, in mL; and
- D = Density of ammonia, in g/mL.

ANNEX B

(Clause 7.1)

SAMPLING OF ANHYDROUS AMMONIA

B-1 GENERAL

B-1.1 Anhydrous ammonia is stored and transported as liquefied gas either without cooling, under a gauge pressure which, at 20°C, is approximately 0.76 MPa (7.6 bar) or with refrigeration, at lower temperatures and pressures. It is strongly irritant as liquid or as vapour, and can cause serious burns to the respiratory tract, skin and eyes. Appropriate protective clothing, gloves, goggles and self-contained breathing equipment should be worn. Mixture of ammonia gas and air can explode, if ignited, when the proportion of ammonia gas is within the limits of 16 to 26 percent by volume; therefore, sources of ignition should be prohibited during the sampling of ammonia.

B-1.2 For further precautionary details, reference may be made to IS 4544.

B-2 PROCEDURE

B-2.1 From Cylinders

B-2.1.1 The contents of a cylinder or other small transport container at or near atmospheric temperature may be sampled near the laboratory but in the open air, by means of a sampling line and connection assembly attached to the cylinder outlet valve. In the event of corrosion and wear it may be necessary to use a suitable washer to ensure a good seal between the cylinder and the sample apparatus; a compressed asbestos fibre washer is suitable for this purpose. The cylinder is securely supported in the position for liquid ammonia discharge.

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B-2.1.2 A separate sample shall be taken for each determination, and no attempt made to divide a sample.

B-2.1.3 The gauge pressure of the container shall be read at the time of sampling in order to determine the evaporation factor (see Annex C).

B-2.2 From Tank Wagons and Storage Tanks

B-2.2.1 It is usual, for maximum safety, to avoid taking liquid ammonia samples direct from a road or rail tank wagon, but to take them instead from a sampling point installed specifically for the purpose in the fixed pipeline through which the ammonia is discharged from the mobile tank to the storage tank. Sampling technique then follows the same procedure as in sampling from cylinders.

B-2.2.2 Although it is recommended that mobile transport tanks should not be sampled directly, there may be occasions when it is unavoidable. In this case, it is essential that a clear, readily understood sampling schedule shall be issued and the samplers properly briefed. Since the technique will be mainly decided by the design of the tank, it shall be agreed with the owner of the tank and the supplier of ammonia.

B-2.3 From Tanks into Cylinders

B-2.3.1 Only when the sample has to be sent to a distant testing station or it is necessary to store it for some time, it will be necessary to sample into a cylinder. Unless very carefully carried out, this procedure may increase the possibility of contamination of the sample and it may also involve danger to personnel.

B-2.3.2 In circumstances which make it essential to use cylinders to take ammonia samples, it will be necessary to have a properly designed cylinder-filling installation for sampling; a make shift arrangement is likely to be unsafe. The design and operation of such installations demand special care and consideration in detail, and no specific recommendations can be made here.

B-3 SAMPLING LINE

B-3.1 General

The sampling of anhydrous ammonia is based on the assumption that the material to be sampled is as claimed and contains only a small amount of impurity primarily water. Duplicate samples shall be taken from each tank or vessel sampled.

B-3.2 Apparatus

B-3.2.1 Weathering Flask — 2 litre capacity graduated from 0 to 10 ml and made of heat resistant glass as shown in Fig. 3.

B-3.2.2 Stopper — Rubber with bend tube vent as shown in Fig. 4.

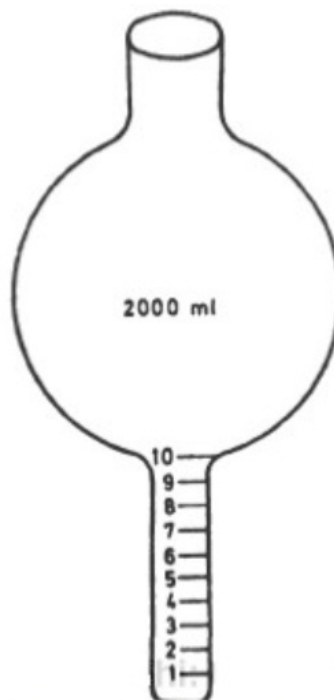


FIG. 3 WEATHERING FLASK

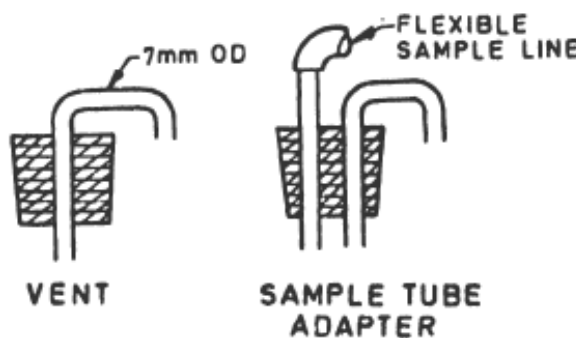


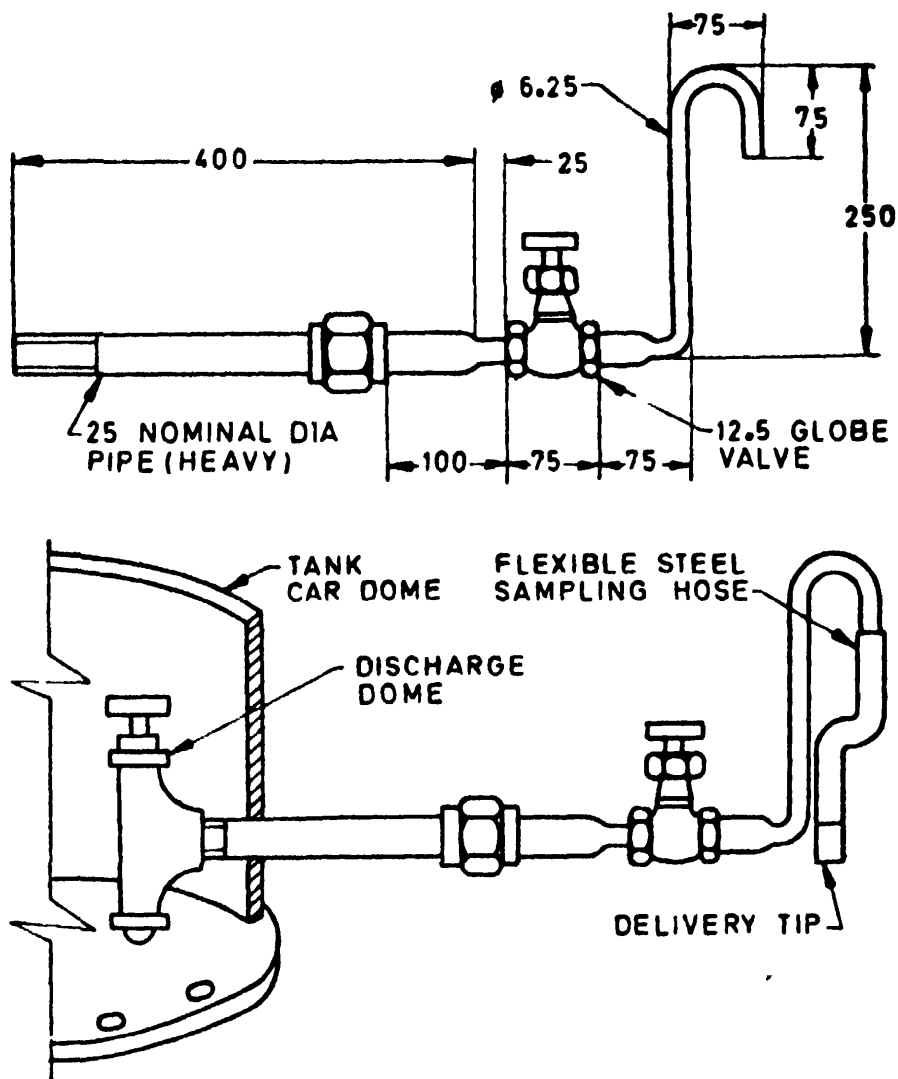
FIG. 4 WEATHERING FLASK ADAPTER

B-3.2.3 Sampling Line and Connection Assembly — (see Fig. 5) in which flexible steel sampling nose 1.2 m, 6 mm NPT coupling at each end and about 3 mm insulated steel tubing delivery tip at one end.

B-3.3 Reagents

B-3.3.1 Charcoal — Reagent, 14 to 20 mesh.

NOTE — If the sample is expected to contain excessive amount of water (one percent or more), one piece of the charcoal may be added to each tube before introduction of the sample.



All dimensions in millimetres.

FIG. 5 SAMPLING LINE AND CONNECTION ASSEMBLY

B-3.4 Procedure

B-3.4.1 Place two dry, clean weathering flasks. Connect the sampling line connection assembly to the unloading valve of the tank, vessel, or line to be sampled. Open the valves slowly and purge the sampling line connection assembly thoroughly by venting 3 to 4 litres of ammonia. Close the sample line globe valve. Remove the vented stoppers from the tubes and insert the adapter end of the sampling line connection assembly. Open the sample line valve and slowly fill the weathering flask to the 100 ml mark, close the sample line valve. Remove

the sampling line adapter and insert the vented stopper in the weathering flask. Repeat as above and fill the second tube. Close tank discharge valve and remove the sampling line connection assembly. Either know or note the vessel, container, or line pressure of material sampled. Tag the collected samples for identification and submit to the laboratory for processing.

B-3.4.2 The gauge pressure of the container shall be read at the time of sampling to determine the evaporation factor. The pressure *versus* evaporation factor is given in Table 2.

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Table 2 Evaporation Factor for Different Pressure in Vessel
(Clause B-3.4.2)

Sl No.	Pressure in Vessel in kg/cm ²	Evaporation Factor (<i>F</i>)
(1)	(2)	(3)
i)	0.0	1.000
ii)	0.70	0.963
iii)	1.41	0.940
iv)	2.10	0.920
v)	2.81	0.900
vi)	3.52	0.885
vii)	4.22	0.870
viii)	4.92	0.860
ix)	5.82	0.850
x)	6.33	0.840
xi)	7.03	0.830
xii)	7.73	0.821
xiii)	8.44	0.813
xiv)	9.14	0.805
xv)	9.91	0.797
xvi)	10.55	0.789
xvii)	11.25	0.782
xviii)	11.95	0.776
xix)	12.64	0.770
xx)	13.36	0.764
xxi)	14.06	0.758

ANNEX C

(Clauses A-2.4 and B-2.1.3)

EVAPORATION FACTOR

C-1 PROCEDURE

C-1.1 When a sample of liquid ammonia is transferred to an open flask from a container in which the pressure is higher than atmospheric, that sample quickly reaches thermodynamic equilibrium with its new environment. Some of the liquid ammonia will evaporate, and since the ammonia so lost as vapour contains none of the non-volatile constituents present in the original product, the concentration of those constituents will increase in the liquid sample taken. This may be a significant factor in subsequent analysis, and fortunately it is possible to calculate from the known thermodynamic properties of ammonia an appropriate correction. This correction is

usually known in this context as the ‘evaporation factor’ or ‘flash factor’, and is simply that fraction by mass of the original liquid ammonia which remains as liquid in the sample. Multiplication of the determined quantity of a ‘non-volatile’ constituent (oil, water, dissolved or suspended solids, etc) by the evaporation factor gives a result which will be closer to the true figure. A diagram of evaporation factors is given in Fig. 6.

C-1.2 Where a sample is transferred (for example, by gravity) from a container to a cylinder at the same pressures there should be no loss by evaporation and in this case it is not necessary to use an evaporation factor.

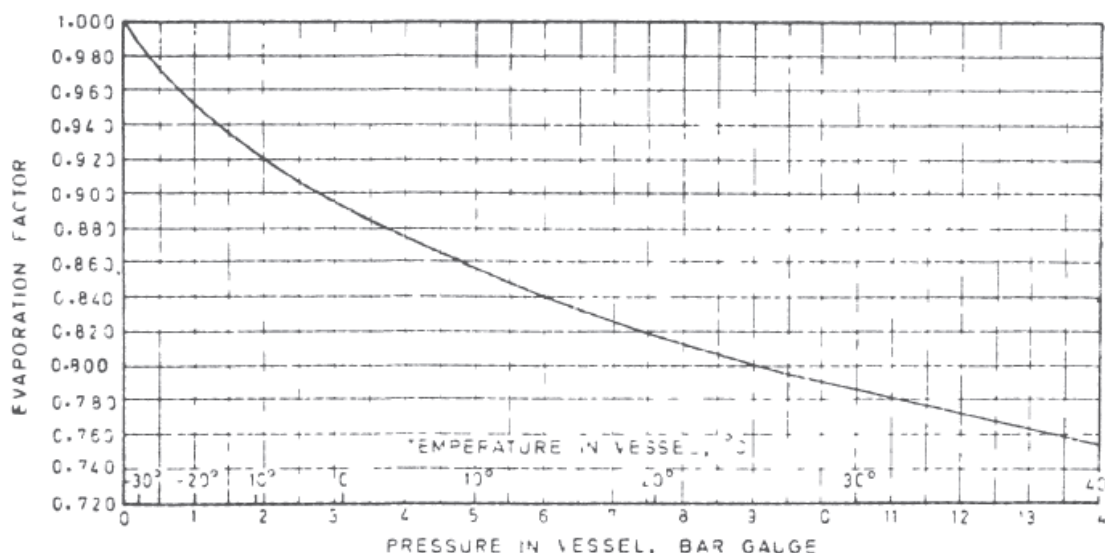


FIG. 6 EVAPORATION FACTOR

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