A P P E N D I X   A
(*Clause* 4.2)

METHODS OF TEST FOR ANHYDROUS AMMONIA

A-1. GENERAL

A-1.1 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 substances non-volatile 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* Appendix 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

$$\text{Residue on evaporation, percent by mass} = \frac{M_1 - M_2}{0{\cdot}68\ V} \times 100\ F$$

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* Appendix C).

A-3. DETERMINATION OF MOISTURE

A-3.0 General — Two methods have been prescribed below. The appropriate method shall be selected depending upon the moisture content.

A-3.1 Method A (For Low Moisture Content)

A-3.1.0 *Applicability* — This method shall be used for anhydrous ammonia with water content under 200 parts per million.

A-3.1.1 *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 : 2362-1973*.

A-3.1.2 *Apparatus*

A-3.1.2.1 *Sample vessel* — As shown in Fig. 1 A. It is marked to hold 100 ml of sample.

A-3.1.2.2 *Guard tube* — As shown in Fig. 1B.

A-3.1.2.3 *Vaccine cap* — with small slit for burette tip, to close the sample vessel.

A-3.1.2.4 *Dewar-flasks (D)* — two.

A-3.1.2.5 *Manometer (E)* — capable of measuring absolute pressures up to 760 mm Hg.

A-3.1.2.6 *Drying tube (F)* — containing phosphorus pentoxide.

A.3.1.2.7 *Suction pump (G)* — fitted with a trap to prevent ammonia entering the pump.

A-3.1.2.8 *Three-way tap (H)*.

A-3.1.2.9 *Tee-pieces* — two.

A-3.1.2.10 *Titration assembly* — as shown in Fig. 1 of IS : 2362-1973*. The sampling apparatus is assembled as shown in Fig. 2.

A-3.1.3 *Reagents*

A-3.1.3.1 *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.

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*Determination of water by the Karl Fischer method *(first revision)*.

1A   Sample Vessel   1B   Guard Tube
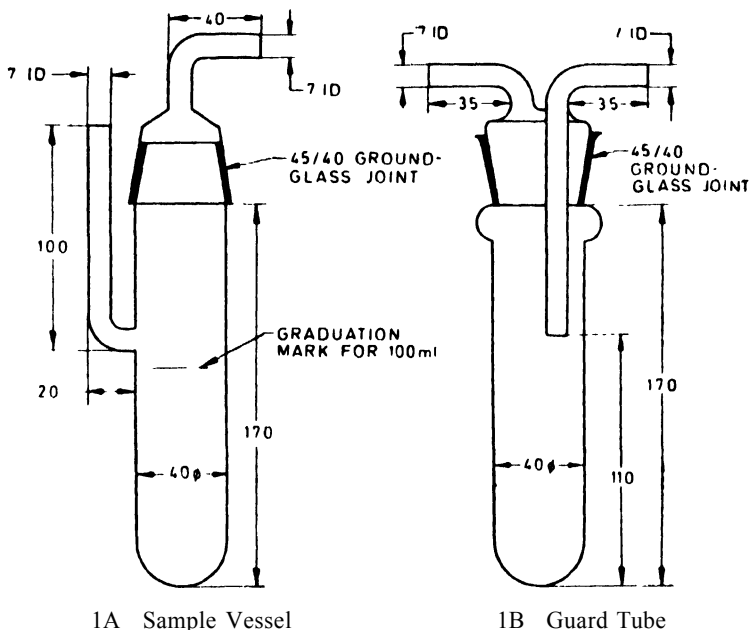
All dimensions in millimetres.

FIG. 1   GLASSWARE FOR THE DETERMINATION OF
WATER IN ANHYDROUS AMMONIA

A-3.1.3.2 *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-3.1.3.3 *Karl Fischer reagent* — having a water equivalent of about 3.5 mg/ml, prepared as prescribed in IS : 2362-1973*.

A-3.1.3.4 *Cooling mixture* — Solid carbon dioxide in methanol.

A-3.1.4 *Procedure* — Standardize the Karl Fischer reagent as described in **6.1.1** of IS : 2362-1973*. 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.

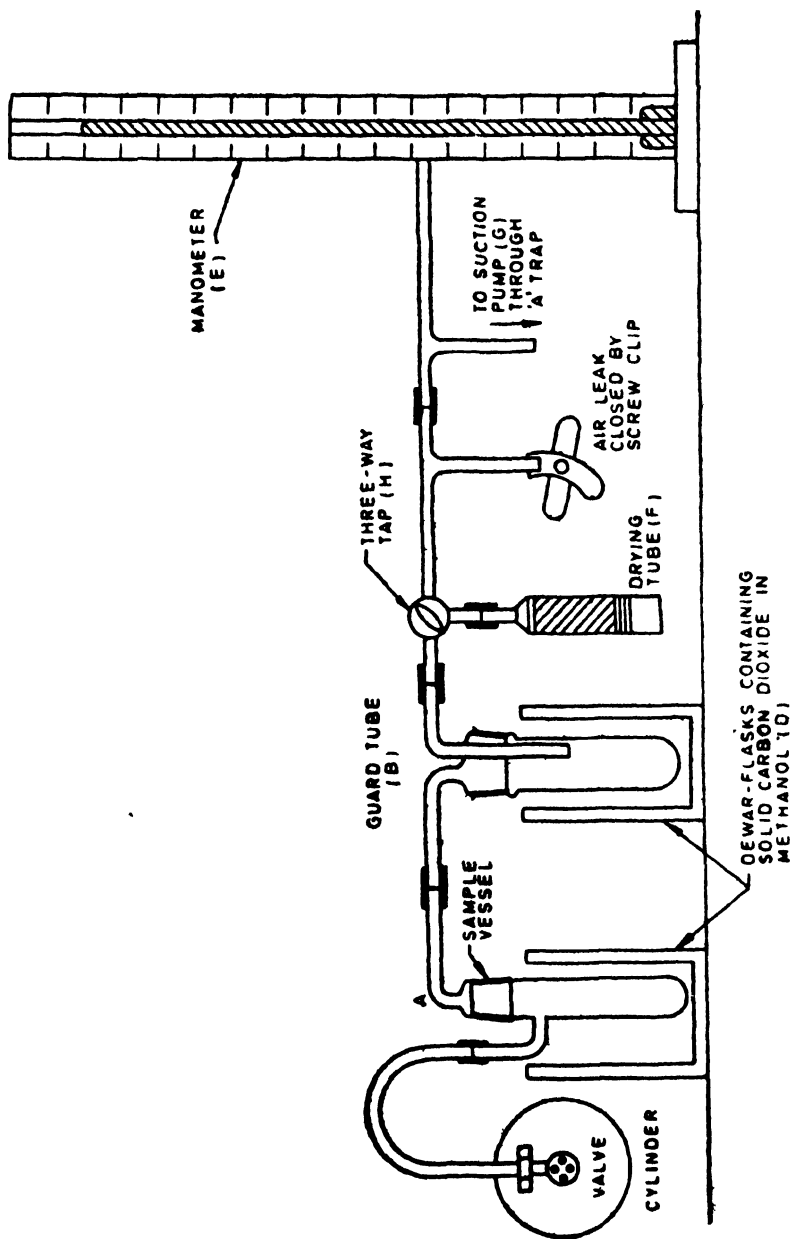*Determination of water by the Karl Fischer method (*first revision*).

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

9

A-3.1.4.1 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 minutes 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 ethanediol. 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, 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-3.1.4.2 When all the ammonia seems to have evaporated, close the air-leak and reduce the pressure in the apparatus to 150 mmHg for 15 minutes 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 ethanediol form 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 : 2362-1973*. Note the volume of the reagent used. Without delay add 2 ml of the ethanediol to the sample vessel and again titrate with the Karl Fischer reagent just to the end point, as described in IS : 2362-1973*. Again note the volume of the reagent used.

A-3.1.5 *Calculation*

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

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,

---

*Determination of water by the Karl Fischer method (*first revision*).

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* Appendix C).

A-3.2 Method B (for Higher Moisture Content)

A-3.2.0 *Applicability* — This method shall be used for anhydrous ammonia with higher moisture content (more than 200 parts per million).

A-3.2.1 *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 as described in IS : 2362-1973*.

A-3.2.2 *Apparatus*

A.3.2.2.1 *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.

A-3.2.2.2 *Titration assembly* — as shown in Fig. 1 of IS : 2362-1973*.

A-3.2.3 *Reagents*

A-3.2.3.1 *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-3.2.3.2 *Methanolic acetic add solution* — Mix 100 ml of glacial acetic acid with 900 ml of methanol with moisture content less than 0.03 percent.

A-3.2.3.3 *Karl Fischer reagent* — having a water equivalent of about 3.5 mg/ml, prepared as described in IS : 2362-1973*.

A-3.2.4 *Procedure* — Transfer to the clean, dry conical flask 2.0 ml of the ethanediol, quickly run in the sample (*see* Appendix 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.

---

*Determination of water by the Karl Fischer method (*first revision*).

A-3.2.4.1 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 : 2362-1973*.

A-3.2.4.2 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 : 2362-1973*. Again note the volume of the reagent used.

A-3.2.5 *Calculation*

$$\text{Moisture content, percent by mass} = \frac{W\,(V_1 - V_2)}{0\cdot 68\,V_3} \times 10^5 \times F$$

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* Appendix C).

A-4. DETERMINATION OF OIL CONTENT

A-4.0 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-4.1 Gravimetric Method

A-4.1.1 *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-4.1.2 *Apparatus*

A-4.1.2.1 *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.

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*Determination of water by the Karl Fischer method (*first revision*).

A-4.1.2.2 *Platinum dish* — 75 mm in diameter.

A-4.1.3 *Reagent*

A-4.1.3.1 *Petroleum spirit* — boiling range 40°C to 60°C (*see* IS : 1745-1978*).

A-4.1.4 *Procedure* — Quickly run in the sample (*see* Appendix B) up to the calibration mark and insert the bung.

A-4.1.4.1 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 minutes 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-4.1.4.2 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-4.1.4.3 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 ± 1°C for 30 minutes. Allow the dish to cool thoroughly in a desiccator and weigh again to the nearest 0.1 mg.

A-4.1.4.4 At the same time carry out a blank determination on the petroleum spirit and filter paper. Make an appropriate correction in the calculation.

A-4.1.5 *Calculation*

$$\text{Oil content, parts per million by mass} = \frac{(M_1 - M_2) - M_3}{0.68\ V} \times 10^6 \times F$$

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* Appendix C).

*Specification for petroleum hydrocarbon solvents (*second revision*).

A-4.2 Spectrophotometric Method

A-4.2.1 *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 3.46 μm (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-4.2.2 *Apparatus*

A-4.2.2.1 *Infra-red spectrophotometer and accessories*

A-4.2.2.2 *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-4.2.3 *Reagents*

A-4.2.3.1 *Carbon tetrachloride* — redistilled. Check that each fresh supply of this reagent shows no absorption at 3.46 µm.

A-4.2.3.2 *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-4.2.4 *Procedure*

A-4.2.4.1 *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 ± 1°C for 15 minutes 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-4.2.4.2 *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 3.46μm) 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-4.2.4.3 *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-4.2.5 *Calculation*

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

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* Appendix C).

APPENDIX B

(*Clause* 6.1)

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 : 4544-1968*.

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

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*Code of safety for ammonia.

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.

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* Appendix 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 sche-dule 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 recomm-endations can be made here.

B-3. SAMPLING LINE

B-3.0 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-3.1 Apparatus

B-3.1.1 *Weathering Flask* — 2 litre capacity graduated from 0 to 10 ml and made of heat resistant glass as shown in Fig. 3.
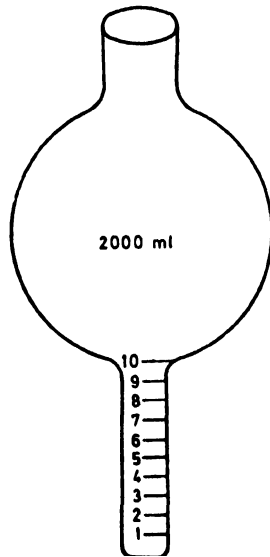

FIG. 3   WEATHERING FLASK

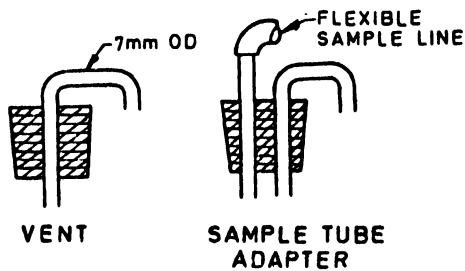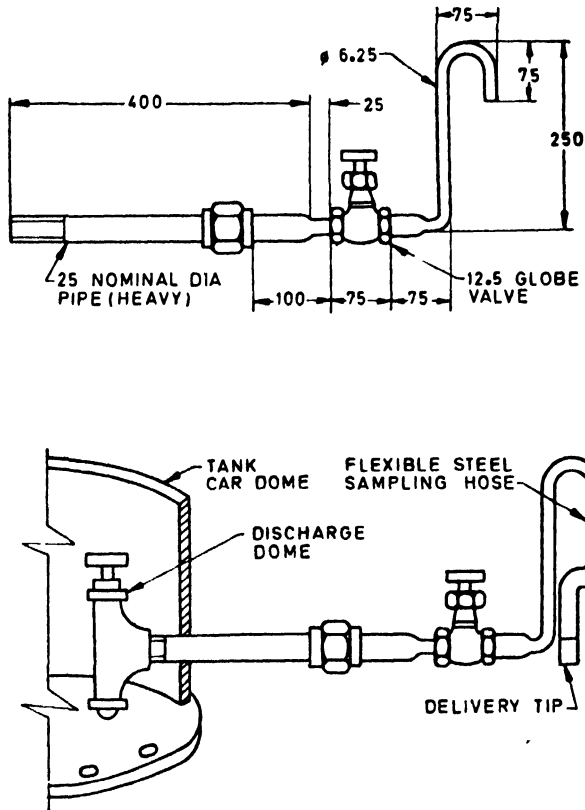**B-3.1.2** *Stopper —* rubber with bend tube vent as shown in Fig. 4.

FIG. 4   WEATHERING FLASK ADAPTER

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B-3.1.3 *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.

All dimensions in millimetres.

FIG. 5   SAMPLING LINE AND CONNECTION ASSEMBLY

B-3.2 Reagents

B-3.2.1 *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 intro-duction of the sample.

B-3.3 Procedure

B-3.3.1 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-3.3.2 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.

TABLE 2 EVAPORATION FACTOR FOR DIFFERENT PRESSURE IN VESSEL

PRESSURE IN VESSEL in kg/cm^2	EVAPORATION FACTOR *(F)*
(1)	(2)
0.0	1.000
0.70	0.963
1.41	0.940
2.10	0.920
2.81	0.900
3.52	0.885
4.22	0.870
4.92	0.860
5.82	0.850
6.33	0.840
7.03	0.830
7.73	0.821
8.44	0.813
9.14	0.805
9.91	0.797
10.55	0.789
11.25	0.782
11.95	0.776
12.64	0.770
13.36	0.764
14.06	0.758

A P P E N D I X   C
(*Clause* 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.
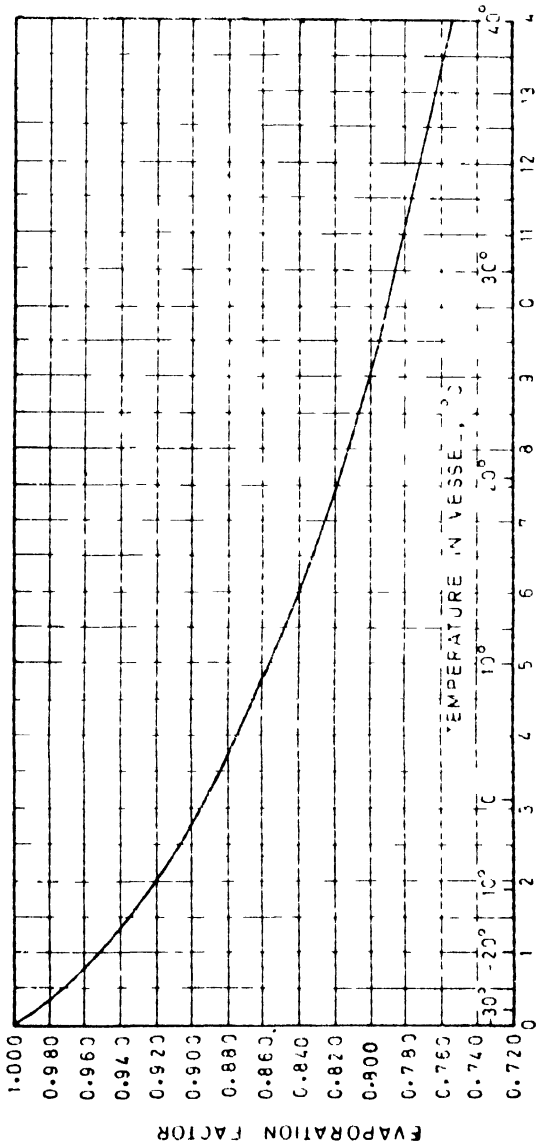
FIG. 6 EVAPORATION FACTOR

BUREAU OF INDIAN STANDARDS

Headquarters:
Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402
Fax: 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams Manaksanstha
(Common to all Offices)

	Telephone
Central Laboratory.	
Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010	8 77 00 32
Regional Offices:	
Central: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34 A, CHANDIGARH 160022	60 38 48
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East) MUMBAI 400093	032 92 95
Branch Offices::	
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‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T T Nagar, B H O P A L 462005	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	6 28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	6 71 19 96
53/5 Ward No. 29, R.G. Barua Road, 5th By lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT BUilding, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
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