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O-A-445B
February 25, 1975
SUPERSEDING
Fed. Spec. O-A-445A
January 31, 1961

FEDERAL SPECIFICATION

AMMONIA, TECHNICAL

This specification was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

1. SCOPE

1.1 Scope. This specification covers one grade of compressed anhydrous ammonia for refrigeration and nitriding purposes.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

Federal Standard:

Fed. Std. No. 123 - Marking for Domestic Shipment (Civil Agencies).

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(Single copies of this specification and other Federal Specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Office in Boston, New York, Washington, DC, Atlanta, Chicago, Kansas City, MO, Fort Worth, Denver, San Francisco, Los Angeles, and Seattle, WA.

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(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

Military Specifications:

MIL-V-2 - Valves, Cylinder, Gas (for Compressed or Liquefied Gases), General Specification for.
MIL-T-704 - Treatment and Painting of Materiel.
MIL-C-3250 - Cylinder, Gases and Liquids, One-Ton, Type D.
MIL-C-11733 - Cylinder, Compressed Gas: Ammonia, with Valve (ICC 4AA-480).
MIL-C-52752 - Cylinders, Compressed Gas, Packaging of.

Military Standards:

MIL-STD-101 - Color Code for Pipelines and for Compressed Gas Cylinders.
MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-129 - Marking for Shipment and Storage.

(Copies of Military Specifications and Standards required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

Laws and Regulations:

Department of Transportation (DOT)

49 CFR 171-190 - General Information and Regulations.

(The Code of Federal Regulations (CFR) and the Federal Register (FR) are for sale on a subscription basis by the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. When indicated, reprints of certain regulations may be obtained from the Federal Agency responsible for Issuance thereof.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

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National Bureau of Standard (NBS) Handbook:

H28 - Screw-Thread Standards for Federal Services.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, DC 20402).

Compressed Gas Association, Inc. (CGA):

Specification G-2. Commodity Specification for anhydrous ammonia.

(Application for copies should be addressed to the Compressed Gas Association, Inc., 500 Fifth Avenue, New York, NY 10036).

American Society for Testing and Materials (ASTM) Standard:

D96 - Water and Sediment in Crude Oils.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103).

3. REQUIREMENTS

3.1 Material. The ammonia shall conform to Table I.

TABLE I. Properties of Ammonia

Properties	Technical Grade Wt. percent
Purity (min) 1/	99.98
Oil (max)	.0005
Moisture (max)	.02

NOTE:

1/ Purity is the weight percent of ammonia by difference, when the weight percent of the residual evaporated at not greater than 86° F (30° C) is adjusted for absorbed NH_3 and subtracted from 100 percent.

3.1.1 The ammonia shall be filtered by the use of a 10-micron nominal filter between the suppliers source and the container of delivery.

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3.1.2 When the ammonia is produced with hydrogen from a source other than natural gas, it shall be tested for pyridine, naphthalene, and hydrogen sulfide. There shall be no evidence of the presence of the above compounds when tested in accordance with 4.4.2.6.1, 4.4.2.6.2 and 4.4.2.6.3.

3.2 Containers and valves. Unless otherwise specified herein, the ammonia shall be contained in Government-furnished containers, capacity as specified (see 6.2 and 6.3), in accordance with 49 CFR 171-190 and equipped with valves with outlet connection No. 241 in accordance with Handbook H28. When specified (see 6.2), new containers shall be furnished by the supplier, and shall be in accordance with either MIL-C-11733 or MIL-C-3250, capacity as specified (see 6.2), and shall be equipped with valves in accordance with MIL-V-2. When specified (see 6.2), the ammonia shall be furnished in supplier-owned Department of Transportation approved containers, capacity as specified (see 6.2).

3.3 Container maintenance. Government- and supplier-furnished containers that require maintenance shall be processed by the supplier for serviceability to meet the requirements of this specification, Title 49 CFR 171-190, and CGA pamphlet G-2, (see 6.6).

3.3.1 Residual pressure. Before refilling, each container shall be checked for residual pressure. A container with an open valve or with insufficient pressure to cause an audible hiss, when the valve is slightly opened, shall be devalved, cleaned, dried, and revalved to assure that the internal condition of the cylinder will preserve the properties of the ammonia. Containers with residual pressure shall be bled to zero pressure in the horizontal position with the diptube turned up and the base slightly elevated. Any cylinder that displays condensation or freezing of water vapor at the valve or within the valve outlet, shall be devalved, cleaned, dried, and revalved before filling. When the cylinder bled to zero pressure is free of water, the valve shall be closed and the cylinder is acceptable for refilling with anhydrous ammonia.

3.3.2 Cylinder drying. Containers subjected to hydrostatic testing, aqueous cleaning, or to water from any source in usage, shall be dried until effluent gas has a dewpoint lower than 40° F. Drying may be accomplished by use of heated inert gas, evacuation of ambient air from the bottom of the container submerged in a water bath at 180° F or by forcing heated or dry air to the bottom of the cylinder. The cylinder shall be valved or plugged to retain the dry condition for refilling with ammonia.

3.3.3 Treatment and painting. Government-furnished containers for ammonia requiring repainting, shall be stripped, treated, and painted as required in accordance with MIL-T-704. Each cylinder shall be color coded and marked in accordance with MIL-STD-101.

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3.3.4 Valves. Unless otherwise specified (see 6.2), replacement valves for defective valves in Government-furnished containers shall be supplier furnished and shall be in accordance with MIL-V-2.

3.3.5 Container filling. Containers with residual pressure approved for refill, shall be charged with anhydrous ammonia to their nominal weight. Cylinders that have been devalved, cleaned, dried, and revalved, shall be filled to nominal capacity and shall be immediately bled-free of approximately 5 cu. ft. of gases, purging the cylinder of the trapped atmospheric air. For anhydrous ammonia, cylinder filling density see 6.5.

3.4 Capacity. Containers shall be filled to rated capacity. The weight of ammonia supplied in each container shall be the difference between the filled weight and the unfilled tare weight of the container.

3.5 Leakage. Containers and valves shall not leak after being filled and examined by submersion or with leak solution.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Component and material inspection. The supplier is responsible for insuring that components and material used are manufactured, examined, and tested in accordance with referenced specifications and standards. The supplier shall verify that the production facility will produce anhydrous ammonia that complies with the percent by volume anhydrous ammonia purity requirements of 3.1 for the grade supplied (see 6.4). This verification may be by the current analytical records of anhydrous ammonia from the facility. The analytical records shall be considered current if taken within 180 days preceding the date of contract or purchase order. When current analytical reports are not available, or in case of process upset or equipment malfunction which effects the product purity, anhydrous ammonia from the facility shall be tested as specified in 4.4.2 prior to starting or continuing production.

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4.2 Classification of inspections. Inspections shall be classified as follows:

(a) Quality conformance inspection (see 4.3).
(b) Inspection of preparation for delivery (see 4.5).

4.3 Quality conformance inspection.

4.3.1 Inspection lot. All cylinders, 15 or less 1-ton containers filled from the same source on the same day, or each tank car or trailer shall be considered a lot.

4.3.2 Sampling. Sampling for testing shall be from filled containers and, unless otherwise specified herein, shall be as follows:

(a) Cylinders and 1-ton containers selected in accordance with MIL-STD-105, Inspection Level S-2.
(b) Each tank car.
(c) Bulk storage tank. When specified (see 6.2), bulk liquid ammonia used exclusively for filling an entire order of small containers may be sampled and tested prior to the actual transfer.

4.3.3 Examination. All containers shall be examined as specified in 4.4.1. Presence of one or more defects shall be cause for rejection.

4.3.4 Tests. Samples selected in accordance with 4.3.2 shall be tested as specified in 4.4.2. AQL shall be 2.5 percent defective.

4.4 Inspection procedure.

4.4.1 Examination. The filled ammonia containers shall be examined as specified herein for the following defects:

101 Container and valve not as specified.
102. Container not processed and dried as specified.
103. Painting, color coding, and marking not as specified.
104. Capacity at 70° F not as specified.
105. Container or valve leakage.

4.4.2 Tests.

4.4.2.1 Sampling.

(a) The flasks, tubes, and assemblies shall be cleaned with chlorinated solvent and dried. Boiling chips of known volume shall be placed

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in the sample containers and the following samples shall be taken from each inspection lot. For moisture residual and purity tests, use standard ASTM 100 ml centrifuge tubes calibrated in 0.05 ml increments to 1.0 ml. Prepare an adapter from a 1/2 inch pyrex tube with a 1/4 inch lateral vent, fit one end to receive the sampling hose and the other end with a one-hole stopper that will mate with the mouth of the centrifuge tube for a snug fit. Assemble the adapter, hose, and centrifuge tube into a unit and provide a vented stopper for each flask to be used. If possible chill this equipment before taking samples.

(b) Attach the hose of the 100 ml tube assembly to the container of ammonia to be tested and as quickly and accurately as possible draw 100 ml into the centrifuge tube with the container shut-off valve. With the apparatus intact, allow the ammonia to evaporate to less than 0.5 ml. Repeat this sampling procedure four more times for a total sample size of 500 ml. With the final residue in the centrifuge tube and with assembly intact loosen the hose from the adapter and allow the ammonia to continue to evaporate at room temperature.

(c) For oil determination draw a 1000 ml sample into a round bottom calibrated flask. Immediately stopper the flask with a vented stopper and allow to evaporate at room temperature. Read the pressure or temperature of the storage container to determine the evaporation factor from Figure 1.

4.4.2.2 Sample preparation. Place the tubes and the round bottom flasks in a circulating water bath until the ammonia evaporation is complete. The temperature of the bath shall not exceed 86° F (30° C). The remaining residue is a mixture of water, oil, nonvolatile impurities, and ammonia.

4.4.2.3 Moisture determination. The ammonia shall be tested for moisture by using the routine analysis method specified in 4.4.2.3.1. In case of dispute the referee method specified in 4.4.2.4 shall be used as the determining criteria.

4.4.2.3.1 Routine analysis method. After evaporation is complete (see 4.4.2.2), determine the volume of the residue remaining in the calibrated centrifuge tubes to the nearest 0.05 ml (corrected to eliminate the volume of the small piece of boiling chip). The composition of the residue will average 28 percent ammonia. The percent by weight of water residue in the sample shall be calculated as follows:

Weight percent moisture = 0.264 1/ (0.72A) F.

Where:

$$0.264 = \frac{100 \times 0.899 \text{ Sp. Gr. Residue}}{(500 \text{ ml}) \quad (0.582 \text{ ml } NH_3}$$

0.72 = 100 - 28 percent NH_3 in the residue.

A = Ml residue in 500 ml sample.

F = Evaporation factor from Figure 1.

1/ = 0.134 ml - 0.20[illegible] wt %

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4.4.2.3.2 Purity. The purity of the ammonia shall be calculated as follows:

100 - weight percent moisture (total residue) = weight percent purity.

4.4.2.3.3 Failure criteria. Nonconformance to 3.1 shall constitute failure of this test.

4.4.2.4 Referee (Karl Fischer) Method. By means of gentle pressuring with dry nitrogen or air, transfer about 25 ml of anhydrous methanol, previously titrated to the dead-stop endpoint, from the aquameter reaction vessel to the tube containing residue, (see Figure 2). Mix by gentle swirling, and then transfer the contents back into the aquameter vessel, by inverting sample tube. Titrate to the dead-stop endpoint. Repeat the transfer and titration once more, and record the total volume of Karl Fischer reagent used. Throughout the transfers and titrations avoid exposing the methanol or the glassware to the atmosphere to avoid absorption of atmospheric moisture.

4.4.2.4.1 Reagents and equipment.

(a) Reagents

(1) Karl Fischer, for aquameter. Fisher Scientific Company. No. SO-K-3 or equivalent.
(2) Methanol, anhydrous, ACS reagent.
(3) Silica-gel for use as desiccant.

(b) Equipment.

(1) Centrifuge tube, oil, ASTM Method D96, 100-ml graduated.
(2) Stoppers, rubber, one-hole, fitted with vent tubes, (Bunsen valves).
(3) Aquameter, Beckman KF-2 or equivalent.
(4) Analytical balance.
(5) Stopper, one hole, for aquameter reaction vessel with rubber seal and two hole stopper for centrifuge tube.
(6) 3/16" Tygon tubing or equivalent.
(7) Two 6" desiccant tubes.
(8) Two syringe bulbs.

4.4.2.4.1.2 Calculations. Calculate the moisture content of the ammonia as follows:

$$\text{Percent water} = \frac{1.47\ (C)\ \ (E)\ \ (F)}{1,000}$$

Where:

C = volume of Karl Fischer reagent consumed, in milliliters.
E = water equivalence factor of Karl Fischer reagent, milligrams H_2O per milliliter of reagent.
F = evaporation factor (see Figure 1).

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4.4.2.4.1.3 Failure criteria. Nonconformance to 3.1 shall constitute failure of this test.

4.4.2.5 Oil determination. Extract the residue in the round bottom flask with four 10 ml portions of carbon tetrachloride. Add the solvent from a 10-ml pipet and while adding each portion, run the tip of the pipet around the inside neck of the flask as the latter is rotated, to completely wash the inside wall of the flask. Carefully transfer each 10 ml portion of carbon tetrachloride (and any water present in the residue) to a separatory funnel, combining the extracts in the same funnel. The funnel should have a dry stopcock (no lubricant) and should be previously rinsed with carbon tetrachloride. After shaking, allow the water phase of the combined extract to separate and drew off the organic bottom layer through a dry filter paper into a weighed evaporing dish. Rinse the filter paper with two 10 ml portions of carbon tetrachloride. The filter paper should be washed previously with carbon tetrachloride and dried. Place the evaporating dish on a steam bath and evaporate the carbon tetrachloride. As the last of the solvent is evaporated, wipe the outside of the dish with a piece of absorbent tissue and cool the dish in a desiccator. Finally, weigh the dish and residue to the nearest 0.1 milligram. Make a blank evaporation of 60 ml of carbon tetrachloride for each lot of the solvent used.

4.4.2.5.1 Reagents and equipment.

(a) Reagents.

(1) Carbon tetrachloride, ACS reagent.
(2) Calcium chloride, anhydrous, for use as desiccant.

(b) Equipment.

(1) Evaporation flask, 1,000 ml capacity, round bottom.
(2) Evaporating dish, 100 ml capacity, glass flat bottom.
(3) Pipet, 10 ml capacity.
(4) Funnel, separatory, 125 ml capacity.
(5) Paper, filter, Whatman No. 5 or No. 41 or equivalent.
(6) Desiccator.
(7) Analytical balance.

4.4.2.5.2 Calculations. Calculate the oil content of the sample by means of the following equations:

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Oil content, ppm = 1.47 (A-B) F

Where:

A = Weight of oil residue, milligrams.
B = Weight of carbon tetrachloride blank, milligrams.
F = Evaporation factor derived from the chart (Figure 1) relating evaporation factor to the pressure (or temperature) of ammonia in the tank from which the sample was drawn.

$$1.47 = \frac{1000}{(1{,}000 \text{ ml})\ (0.682 \text{ gram/ml})}$$

where 0.682 is the density of liquid ammonia at atmospheric pressure (14.7 psig).

4.4.2.5.3 Failure criteria. Nonconformance to 3.1 shall constitute failure of this test.

4.4.2.6 Pyridine, naphthalene, and hydrogen sulfide determination. Unless the ammonia is made from natural gas the following tests for pyridine, naphthalene, and hydrogen sulfide shall be made. Samples 100 milliliters in size, shall be taken as specified in 4.4.2.1 instead of 500 ml samples when required.

4.4.2.6.1 Pyridine. About 100-milliliter sample of ammonia, taken as specified in 4.4.2.1 shall be converted to nitrate or sulfate, using as little excess of acid as possible. Evaporate the solution until crystals begin to separate and transfer the concentrated solution to a separatory funnel. Make alkaline with a saturated solution of reagent-grade sodium bicarbonate and shake with ethyl ether. The sodium bicarbonate has no action on the ammonia salt but liberates the pyridine which dissolves in the ethyl ether. Shake the solution two additional times with ethyl ether and treat the combined ether extract with distilled water and sufficient dilute nitric acid (1:3) to render it slightly acid. Evaporate the mixture to a small volume, transfer to a small distilling flask, together with a small piece of zinc to prevent bumping, and render alkaline with sodium hydroxide solution. Distill over two-thirds of the liquid into a small test tube containing 1 milliliter of saturated solution of mercuric chloride. Heat the distillate almost to boiling and filter into a small test tube. Cool to about 25° C. The presence of any cluster of needles shall indicate the presence of pyridine.

4.4.2.6.2 Naphthalene. About 100 milliliters of the sample of ammonia, taken as specified in 4.4.2.1 shall be evaporated with 0.20 gram of picric acid. Dissolve the residue in 5 milliliters of boiling 95 percent ethyl alcohol and gradually cool to about 25° C. Note whether any crystals have separated. Naphthalene shall be considered absent if no crystals separate out.

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Should crystals separate out, filter off the separated materials and recrystallize from a small amount of 95 percent ethyl alcohol. Dry the crystals on a clay plate and determine the melting point. A melting point of 150° plus or minus 1° C indicates the presence of naphthalene.

4.4.2.6.3 Hydrogen sulfide. Place a strip of lead acetate paper in a small quantity of liquid ammonia drawn from the cylinder. Any black spots on the lead acetate paper indicate the presence of hydrogen sulfide.

4.4.2.6.4 Failure criteria. Evidence of the presence of any pyridine, naphthalene, or hydrogen sulfide shall constitute failure of this test.

4.5 Inspection of preparation for delivery. The packing shall be examined to determine compliance with MIL-C-52752.

5. PREPARATION FOR DELIVERY

5.1 Packing. The cylinders of anhydrous ammonia shall be packed in accordance with MIL-C-52752. The level of packing shall be Level A, B, or C as specified (see 6.2).

5.2 Marking.

5.2.1 Civil agencies. In addition to any special marking required by the contract or order, loose, boxed or palletized cylinders shall be marked in accordance with FED. STD. No. 123.

5.2.2 Military agencies. In addition to any special marking required by the contract or order, loose, boxed or palletized cylinders shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. The anhydrous ammonia is intended for use as a coolant in missiles, a refrigerant, and as an agent for metal nitriding.

6.2 Ordering data. Purchasers should select the preferred options permitted herein, and procurement documents should specify the following:

(a) Title, number, and date of this specification.
(b) Capacity of Government-furnished container required, when applicable (see 3.2).
(c) When new containers will be furnished to the Government by the supplier, or when the ammonia will be contained in supplier-owned containers, and capacity of container required (see 3.2).

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(d) When replacement valves for Government-furnished cylinders are to be furnished by the Government (see 3.3.4).
(e) When bulk liquid ammonia may be tested in lieu of sampling small liquid containers filled from the bulk source (see 4.3.2).
(f) Level of packing required (see 5.1).

6.3 Government-furnished containers. When applicable, the contracting officer should arrange to furnish the cylinders specified in 3.2.

6.4 Certification. When specified by the procurement agency, certification of the analysis by the manufacturer of the anhydrous ammonia may be accepted in lieu of an analysis of lot inspection, provided the ammonia is transferred directly from the manufacturer's tested container to acceptable Government-furnished containers.

6.5 Filling density. Anhydrous ammonia containers shall be filled to a density not greater than 54 percent in weight ratio to the weight of the container's water capacity.

6.6 Maintenance of Government-furnished containers. Purchasers should specify the extent to which Government-furnished containers that require maintenance should be processed by the gas supplier.

6.6.1 Gas supplier. The gas supplier should furnish at no additional cost, all services which are required at each and every filling of a container to comply with applicable regulations and normal good practice. Such services would include, but not be limited to, all inspection, testing, evacuation, and handling services required for the gas supplied.

6.6.2 Allowable fees. A schedule of allowable fees should be specified by the purchaser for the gas supplier's performance of services such as the replacement of valves, valve parts, and cylinder caps, hydrostatic testing, cleaning, painting, color coding, marking, and handling of unserviceable containers as required. All materials and components for these services should be furnished by the gas supplier.

6.7 Ammonia should be purchased by weight.

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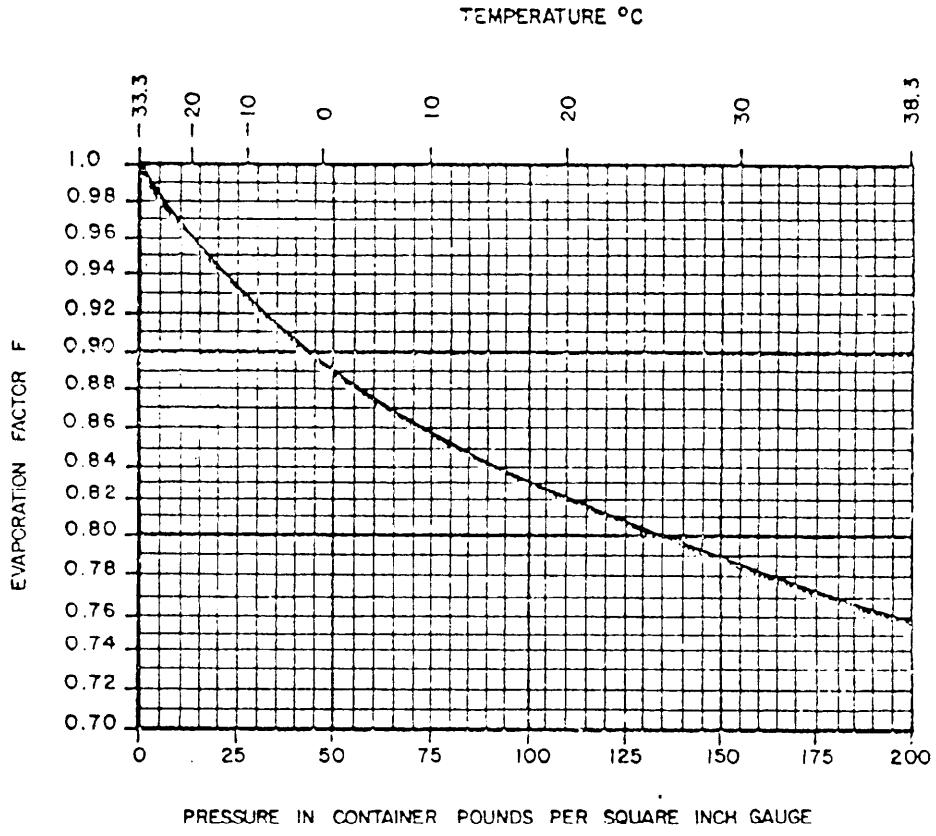

THE EVAPORATION FACTOR F IS A CORRECTION FOR THE EVAPORATIVE COOLING OF LIQUID AMMONIA FROM THE TEMPERATURE CORRESPONDING TO THE PRESSURE IN THE STORAGE VESSEL TO THE BOILING POINT AT ATMOSPHERIC PRESSURE (-33.3 °C).

FIGURE 1. EVAPORATION FACTOR

X-2685

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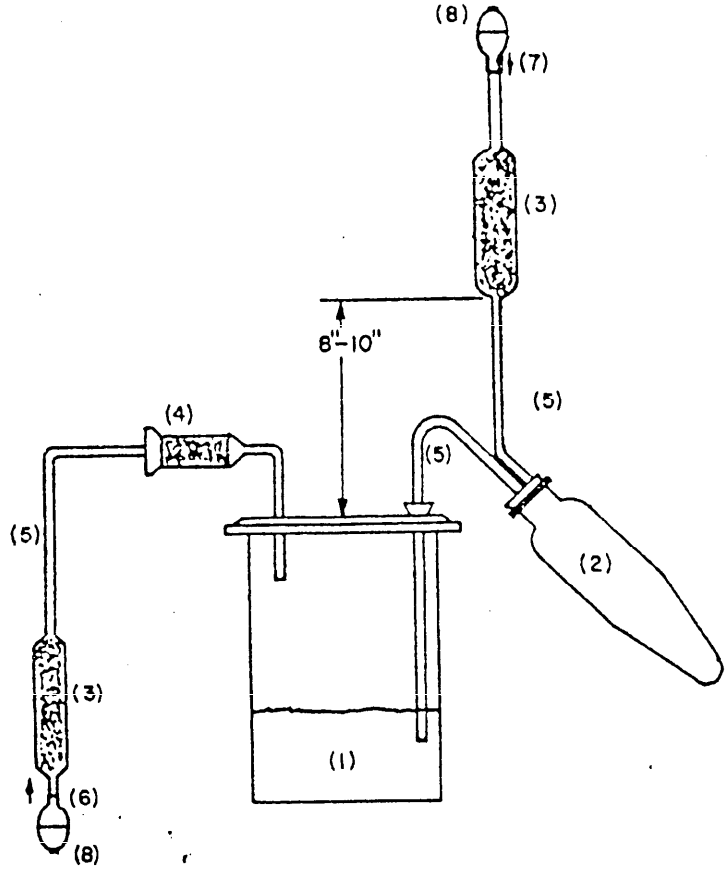

(1) AQUAMETER REACTION VESSEL
(2) SAMPLE TUBE (CENTRIFUGE)
(3) DESICCANT TUBE 6"
(4) AQUAMETER DESICCANT TUBE
(5) 3/16" PLASTIC TUBING (TYGON OR EQUIVALENT)
(6) PRESSURIZE TO MIX DRY METHANOL WITH SAMPLE
(7) PRESSURIZE TO RETURN SAMPLE TO REACTION VESSEL
(8) SYRINGE BULBS OR NITROGEN SOURCE

FIGURE 2. ADAPTATION TO AQUAMETER REACTION VESSEL

X-2686

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MILITARY INTERESTS:

Custodians:

Army - ME
Navy - AS
Air Force - 68

User activity:

Navy - AS

Civil Agency Coordinating Activities:

GSA - FSS

Preparing activity:

Army - ME

DOD Project No. 6830-0076

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STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER	2. DOCUMENT TITLE

3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION *(Mark one)*
	☐ VENDOR
b. ADDRESS *(Street, City, State, ZIP Code)*	☐ USER
	☐ MANUFACTURER
	☐ OTHER *(Specify)*: _____

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional	b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional
c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional	8. DATE OF SUBMISSION *(YYMMDD)*

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