

# AQUA AMMONIA INFORMATION MANUAL

## Forward

Ammonium hydroxide (commonly referred to as “aqua ammonia”) is a very useful chemical in today’s modern world. Its wide range of uses for air pollution control, potable water treatment, waste treatment and a multitude of chemical process industry applications make it a valuable and essential chemical.

The increasing use of this product and the rising safety consciousness of industry has prompted Airgas Inc. (Airgas) to prepare this booklet of information for its employees, customers and other interested personnel. Its readers will find answers to their most often asked questions regarding aqua ammonia’s production, transportation, storage and handling, safety, first aid, emergency response information and regulations pertaining to its use.

**INFORMATION REGARDING REGULATIONS IS NOT INTENDED TO BE COMPREHENSIVE AND IS PROVIDED FOR EXEMPLARY PURPOSES ONLY AND SHOULD NOT BE RELIED UPON. READERS ARE DIRECTED TO THE ACTUAL LAWS, RULES AND REGULATIONS GOVERNING AQUA AMMONIA AND ADVISED TO SEEK COUNSEL REGARDING THEIR COMPLIANCE.**

A collection of charts, tables and graphs of physical and chemical properties is also included.

Individuals seeking information not covered in this booklet are urged to contact Airgas directly.

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## Definition

Aqua ammonia, aqueous ammonia and ammonium hydroxide are synonymous terms referring to a solution of ammonia in water. The chemical formula for ammonium hydroxide is  $\text{NH}_4\text{OH}$ . Ammonia dissolved in water is present principally as the ion  $\text{NH}_4^+$ . Non-ionized molecular  $\text{NH}_4\text{OH}$ , sometimes referred to as an associated form of ammonium hydroxide, is also present. Ammonia dissolved in water is commonly referred to simply as ‘ammonia’. Understand that this usage of the word does not refer to anhydrous ammonia!

## Production

Aqua ammonia is produced commercially by reacting anhydrous ammonia with water to form a solution which is typically less than 30% ammonia by weight. Prior to reaction, the water is commonly purified by treatment with either:

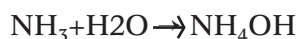
- 1) Softening: A conventional sodium-form water-softening ion exchange resin which replaces all cations present with sodium ions;
- 2) Ion Exchange: Both hydrogen-form cation resin and hydroxyl-form anion resin, resulting in total de-ionization of the water; or
- 3) Reverse Osmosis: This also results in total de-ionization of the water.

Some agricultural grades of aqua ammonia are manufactured without pre-treatment of the water. These are generally not recommended for industrial use.

The reaction of ammonia and water is exothermic and generates a substantial amount of heat, necessitating the use of heat exchangers to control the temperature.

The resulting aqua ammonia contains ammonium ions ( $\text{NH}_4^+$ ), hydroxyl ions ( $\text{OH}^-$ ), and non-ionized ammonium hydroxide molecules ( $\text{NH}_4\text{OH}$ ).

The chemical reactions are:



## Product Specifications

The most common aqua ammonia concentrations are 19.5% and 29.4% ammonia by weight. Certain Airgas plants produce additional concentrations based on specific customer requirements.

29.4% ammonia product is also frequently described as 26°Bé product. Degrees Baumé is a system for density measurement. Since the density of aqua ammonia is inversely proportional to concentration, degrees Baumé can be used as an indicator of concentration. Degrees Baumé is related to specific gravity by the formula:

$$^{\circ}\text{Bé} = (140/\text{sp.gr.}) - 130$$

“26° Baumé at 60°F”, “29.4% ammonia by weight” and “0.8974 specific gravity at 60°F” are all terms referring to an identical concentration of aqua ammonia. Specific gravity and degrees Baumé of aqua ammonia vary with temperature. See Appendix A, pages 15-28.

Aqua ammonia is available at several different purity levels. Airgas utilizes the following Product Specification Grades:

Reagent  
Technical  
N.F. (formerly U.S.P.)  
Commercial  
Neutralization  
FCC

NOTE: Not all of these grades are available at each Airgas production point. For specific availability information, contact Airgas Inc.

The American Chemical Society (ACS) defines reagent grade; Federal Specification OA-451-F defines technical grade aqua ammonia; the National Formulary Monograph defines N.F. grade; and the Food Chemical Codex defines FCC Grade which is also referred to as food grade.

## Analytical Methods

Information on analytical procedures required for the analysis of aqua ammonia (ammonium

hydroxide) to determine compliance with the requirements of various specifications are given in Appendix C, pages 41-50.

It is important to understand that an aqua ammonia sample exposed to the air in a sampling container will rapidly lose ammonia vapor and significantly decrease in concentration and increase in density.

This analytical error can be minimized by cooling the sampling container to 5-10°C (40-50°F) prior to collection of the sample. If the density/concentration analysis is not to be done immediately, cool the sample and container subsequent to collection and during storage. If the sample is to be retained, the container should be kept tightly closed except for sample withdrawal. Withdrawal of a sample and its density/concentration analysis should be accomplished as quickly as possible.

For the most accurate analysis, the sample should be analyzed immediately.

## Transportation

Aqua ammonia is transported by Airgas in cargo tanks constructed in accordance with Department of Transportation (DOT) Specification MC-307 or MC-407. The use of other cargo tanks, such as MC-312 or MC-412, is authorized provided they are in compliance with the requirements of 49 CFR 173.241.

Transportation of aqua ammonia with a concentration greater than 10% is regulated by the federal DOT, which classifies it as a "CORROSIVE" and assigns it the identification number UN2672. Applicable DOT regulations should be carefully followed when transporting aqua ammonia. The proper shipping name for aqua ammonia is "Ammonia Solutions". It is a Hazard Class 8, Packing Group III chemical.

Re-packagers of bulk aqua ammonia use many different types of shipping containers including 55-gallon steel drums, carboys and plastic containers. Effective October 1, 1996, all non-bulk containers must meet the performance-oriented standards as referenced in 49 CFR 172.101 and defined in 49 CFR 173.203. See Appendix C, pages 43-46 for DOT regulatory references.

For chemical transportation emergencies, contact CHEMTREC at (800) 424-9300.

Any release of this material, during the course of loading, transporting, unloading or temporary

storage, must be reported to the DOT as required by 49 CFR 171.15 and 171.16.

## Delivery

Deliveries into aqua ammonia storage tanks are made by cargo tank transports having a capacity of approximately 6500 gallons. Transfer of the product is usually effected by the use of a pump, however a compressor or a source of compressed air or nitrogen can be used in certain applications. The pump is typically mounted on the delivery cargo tank transport, however it may be a part of the receiving storage system. Refer to Appendix D, page 48.

If compressed air or nitrogen is used to effect delivery, the piping system shall include a backflow check valve to prevent ammonia vapor from entering the air or nitrogen line. Additionally, a scrubber or other similar system should be used to control the release of vapors from the tank when this type of delivery is employed.

Aqua Ammonia storage tanks should not be filled to more than 95% of their volume.

## Storage

Airgas recommends that bulk storage of aqua ammonia utilizes ASME Code pressure vessels constructed for a design pressure (Maximum Allowable Working Pressure) of at least 25psig. Any alterations or repairs to such storage tanks must be accomplished in accordance with ASME Code requirements.

Tanks shall be equipped with relief valve(s) in accordance with design pressure. Each tank should also be equipped with a vacuum breaker set at 2-4 ounces of vacuum. A conservative method for calculating the relief capacity for the tank utilizes the formula:  $CFMAIR = 22.11A0.82$ . Where A is the outer surface area of the vessel in square feet. (See Appendix B of the ANSI K61.1-1989 Standard for the Storage and Handling of Anhydrous Ammonia).

A typical bulk storage tank for aqua ammonia is shown in Airgas Technical Bulletin TB-8-8, which also describes the functions of the various tank openings and appurtenances. This bulletin is reproduced in Appendix D, pages 49-50.

Carbon steel, aluminum and stainless steel are acceptable metals for use in fabricating aqua ammonia storage vessels. Internal linings or

coatings are not commonly used.

Each tank or container should be clearly marked “AMMONIUM HYDROXIDE.”

Great care must be taken if it becomes necessary to introduce water into a storage tank containing ammonia vapors. Extremely rapid absorption of the ammonia vapors will occur with concurrent rapid reduction in pressure, possibly resulting in a collapse of the tank. The vacuum-breaker (with which most tanks are equipped) does not typically allow for a sufficiently rapid enough introduction of air into the vessel to prevent a tank collapse from occurring.

All tank openings except those for relief devices and float gauges shall be equipped with shut-off valves. Shut-off valves shall not be installed between relief devices and the tank.

Small containers of aqua such as drums and carboys should be stored in a cool place and out of direct sunlight.

The maintenance of aqua ammonia storage systems usually consists of 1) protection of any external steel surfaces from possible corrosion by inspection, re-priming and finish coat painting as necessary and 2) checking for leaks. The location of leaks tends to be obvious; however, sources can be pinpointed by using Airgas ammonia test paper (phenolphthalein paper).

Carbon steel tanks used for the storage of aqua ammonia are not subject to internal corrosion after passivation. However, residual rust formed during any hydrostatic pressure test conducted at the time of fabrication and rust from attached steel pipe and fittings may be present.

Aluminum and stainless steel tanks are not subject to internal corrosion. Painting of exterior surfaces of such tanks is optional.

Maintenance procedures requiring the removal of any valve or fitting directly attached to the tank should be performed only after all aqua ammonia has been removed from the tank and ammonia vapor fumes purged from it.

## Materials Compatibility

### Metals

#### Acceptable

Carbon Steel  
Stainless Steel  
Aluminum  
Cast Iron  
Tin  
Lead  
Hastelloy B, C, D

#### Not Acceptable

Copper  
Copper Alloys (such as brass and bronze)  
Zinc (galvanized surfaces)  
Cadmium  
Silver (e.g., silver brazed two-part “all steel” valves)  
Nickel  
Monel

### Elastomers

#### Acceptable

Neoprene  
Ethylene Propylene  
Butyl  
Buna  
Teflon  
Haveg 41, 60  
Hastaflor  
Flourthene

#### Not Recommended

Natural Rubber  
Nitrile  
Polyurethane  
Hypalon  
Silicone  
Viton



## Emergency Response

Personnel responding to the accidental release of a hazardous material must be trained in accordance with HAZWOPER regulation 29 CFR 1910.120. The following are suggested steps to be followed in the event of an accidental release of aqua ammonia:

1. Report the release immediately to the National Response Center (1,000 lbs. Or more in any 24-hour period). Report to state and local authorities as required.
2. Alert all on-site personnel and shut down all aqua ammonia operations.
3. Account for all on-site personnel.
4. Administer first aid to injured personnel.
5. Evaluate the emergency and assess the possible need for an evacuation.
6. Contact off-site emergency responders as necessary.
7. Coordinate with off-site responders.
8. Direct site activities.
9. Identify the source of the aqua ammonia leak.
10. Isolate and control the source of leak.
11. Record site events (ongoing).
12. Monitor the leak (ongoing).

The application of a water fog to absorb ammonia vapors immediately downwind of a spill is an effective mitigation tool. Determine the potential flow pattern of the ammonia-water solution beforehand. If necessary, create proper pathways for retaining the dilute ammonia-water solution.

## Environmental

Properly designed containment systems must be used for confining aqua ammonia (ammonium hydroxide) in the event of a large spill. Spills should not be washed into ground water. Release into sewers is not permitted without appropriate approvals and dilution. For information, contact your local, state and federal regulatory agencies. Secure regulatory and/or sanitary district approval prior to disposal into a sewer. For hazardous waste regulations, contact the federal RCRA Hotline at (800) 424-9346.

Aqua ammonia in concentrations as low as 5 ppm can be harmful to aquatic life. Aqua ammonia is a regulated material and reporting of any release may be required.

## Safety

Refer to the Material Safety Data Sheet for Airgas aqua ammonia for more detailed safety information.

### Primary Hazards

Aqua ammonia is an alkaline material and reacts corrosively with human tissue in varying degrees depending on concentration and the time duration of exposure. Ammonia vapors from aqua ammonia can be suffocating and irritating to mucous membranes and lung tissue. Skin contact can cause severe irritation and burns. Eye contact with aqua ammonia may be severely irritating; ammonia vapor contact with eyes may be mildly irritating. Ingestion can cause vomiting, nausea and corrosive burns to the esophagus and stomach. Ammonia is not listed as a carcinogen by IARC, NPT or OSHA.

The easily recognized odor of aqua ammonia provides adequate warning of its presence.

Aqua ammonia is not flammable. However, ammonia vapors present in aqua ammonia storage and handling equipment can ignite in the presence of a flame or spark at about 1200°F. The flammability range of ammonia vapor is approximately 16-25% of ammonia in air by volume. The NFPA hazard designation for anhydrous ammonia is 3-1-0 (Health =3; Flammability = 1; Reactivity =1).

Before welding or cutting, aqua ammonia tanks and/or piping must be completely purged of all ammonia. Purge until no odor can be detected and continue the purging during the welding or cutting maintenance procedure.

### Other Hazards

Aqua ammonia may react with halogens such as bromine and chlorine, with silver or with hypochlorites to form explosive and/or toxic compounds.

Ammonia vapor begins to dissociate into nitrogen and highly flammable hydrogen at about 840°F if a suitable catalyst is present. Iron pipe is one such catalyst.

Aqua ammonia will react with many organic and inorganic acids to form salts. These reactions are usually exothermic, i.e., heat is generated.

# AQUA AMMONIA

## Human Physiological Effects

Aqua ammonia is not a cumulative metabolic poison. Ammonia is actually an important compound in life processes. Human blood contains about 1 ppm ammonia; humans produce about 4 grams of ammonia per day.

Depending on concentration and time, the effects of exposure to ammonia vapor vary from none or only mild irritation, to obstruction of breathing from laryngeal and bronchial spasm, to edema and severe damage of the mucosa membranes of the respiratory tract with possible fatal results.

Contact of the skin with aqua ammonia can result in a caustic burn.

Exposure levels of ammonia vapor which are tolerated by some persons may produce adverse reactions in others. People with chronic respiratory disease or undue sensitivity to ammonia should not be exposed to it. The table below indicates human physiological response to the inhalation of various concentrations of ammonia vapor in air.

### *Physiological Effects Of Ammonia Vapor*

<b><i>Effect</i></b>	<b><i>PPM Ammonia in Air by Volume</i></b>
Least perceptible odor	5 ppm
Readily detectable odor	20-25 ppm
No discomfort or impairment of health for prolonged exposure	50-100 ppm
General discomfort and eye-tearing; No lasting effect on short exposure	150-200 ppm
Severe irritation of eyes; ears, nose and throat; No lasting effect on short exposure	400-700 ppm
Coughing, bronchial spasms	1,700 ppm
Dangerous, less than _-hour exposure may be fatal	2,000-3,000 ppm
Serious edema, strangulation, asphyxia, rapidly fatal	5,000-10,000 ppm
Immediately fatal	>10,000 ppm

(From Anhydrous Ammonia G-2-1995, Eighth Edition, 1995, Compressed Gas Association, Inc.)

The odor threshold of ammonia is from about 5

ppm to 50 ppm. This low threshold level typically provides more than ample warning of its presence.

## Exposure Limits For Vapor Ammonia

OSHA regulations have established a Permissible Exposure Limit (PEL) of 50 ppm of ammonia in air by volume as a time-weighted average (TWA), the definition for which is the average exposure in any 8-hour work shift of a 40-hour work week,

The American Conference of Government and Industrial Hygienists (ACGIH) established an exposure limit of 25 ppm ammonia in air by volume as a time-weighted average (TWA).

ACGIH also established a 15-minute short term exposure limit (STEL) of 35 ppm. An STEL is a 15-minute time-weighted average which cannot be exceeded at any time during a work day. NIOSH has set 300 ppm of ammonia as it immediately dangerous to life and health (IDLH) value.

Ammonia vapor is substantially lighter than air. This property makes ventilation an important tool in preventing accumulations which may exceed these limits.

## Personal Protective Equipment

Persons working with aqua ammonia under routine circumstances of operation and maintenance should wear chemical splash-proof goggles and rubber gauntlet gloves with rolled-up cuffs. A full face shield may be worn over goggles for additional protection, but not as a substitute for the goggles. DO NOT wear contact lenses.

Additional personal protective equipment includes:

- \* Long-sleeve shirt and full-length pants
- \* Hard hat with full face shield
- \* Waterproof boots made with special grip soles
- \* Raincoat

## Emergency Protective and Safety Equipment

Every location storing or handling aqua ammonia should have readily-available and freely-accessible emergency protective and safety equipment as required by federal, state and local government regulations. The location of this equipment should be well identified by appropriate signs.

Depending on the size and nature of the aqua ammonia use, emergency protective and safety equipment may include one or more of the following:



### **Safety Shower:**

Parts of the body contacted by aqua ammonia must be flooded immediately with large quantities of water. An emergency safety shower, eye wash fountain or other source of clean water can be used for this purpose. Such a source should be protected from freezing in cold weather.

### **Respiratory Devices:**

1. Two full-face gas masks with industrial-size ammonia canisters approved by MSHA/NIOSH and a spare canister for each. Refer to the manufacturer's label to determine the time allowed at specific breathing rates and various ammonia concentrations. Use of a gas mask for purposes other than escape is not recommended in ammonia vapor concentrations exceeding the IDHL level of 300 ppm. Canisters are stamped with expiration dates by the manufacturer. Do not use after the expiration date. The use of gas mask requires a written training and maintenance program.

2. A self-contained air breathing apparatus of a pressure demand type should be used for protection in emergency situations where ammonia concentrations are unknown or may exceed the concentration for which a gas mask is rated for entry purposes. The use of a self-contained breathing apparatus requires a written training and maintenance program.

A fire hose with fog nozzle is useful for controlling ammonia vapors originating from an accidental release of aqua ammonia. Control the vapors downwind as they leave the area.

## **Regulations**

### **OSHA**

The OSHA Hazard Communication Rule, 29 CFR 1910.1200 applies to aqua ammonia. A hazard communication label should be placed on any container of aqua ammonia.

Aqua ammonia is subject to OSHA Process Safety Management regulations if the concentration is 20% or greater and the quantity maintained on site is 20,000 lbs. or greater.

### **DOT**

See Appendix C, pages 43-46.

### **EPA**

Aqua ammonia is subject to the reporting requirements of the Superfund Amendments and Reauthorization Act (SARA), Section 313 of Title

III and 40 CFR Part 370. The Emergency Planning and Community Right-To-Know Act (EPCRA) Section 302 does not list aqua ammonia as an extremely hazardous substance. Section 311/312 indicates a category of Immediate (Acute) Health Hazard. Section 313 does identify it as a toxic chemical.

Releases of 1,000 lbs. or more of aqua ammonia in any 24-hour period must be reported immediately to the National Response Center in Washington at (800) 424-8802 (CERCLA/Superfund, 40 CFR Parts 117 and 302). State and local agencies may also require reporting.

Aqua ammonia is listed in the toxic Substances Control Act (TSCA).

Aqua ammonia is not listed under California Proposition 65 as either a reproductive hazard or carcinogen.

Aqua ammonia is subject to EPA's Risk Management Plan (40 CFR Part 68) if the concentration is 20% or greater and the quantity maintained on-site is 20,000 lbs. or greater.

The Consumer Product Safety Commission has required in 16 CFR 1500.129 that any ammonium hydroxide consumer product containing 5% or more ammonia bear a POISON label.

# AQUA AMMONIA

## Chemical Properties

### pH

Aqua ammonia (ammonium hydroxide) is classified as a weak base. It ionizes much less completely in water than does a strong base such as sodium hydroxide. This is reflected in the pH's normally encountered with solutions of ammonia. Typically, the pH of an ammonia solution will be between 11 and 12, compared with a pH of about 14 for sodium hydroxide solutions. The theoretical pH's below are for ammonia in pure water at 77°F.

<u>Wt. %</u>			
<u>NH<sub>3</sub></u>	<u>Normality</u>	<u>pH</u>	<u>% Ionized</u>
17.0	10.0	12.1	
1.7	1.0	11.6	0.42
0.17	0.1	11.1	1.33
0.017	0.01	10.6	4.15
0.0017	0.001	10.1	12.52

### Dissociation

#### Dissociation Constants (K<sub>b</sub>'s) of Aqua Ammonia From 0°C to 50°C

<u>Temperature °C</u>	<u>pK<sub>b</sub></u>	<u>K<sub>b</sub></u>
0	4.862	1.374 x 10 <sup>-5</sup>
5	4.830	1.479 x 10 <sup>-5</sup>
10	4.804	1.570 x 10 <sup>-5</sup>
15	4.782	1.652 x 10 <sup>-5</sup>
20	4.767	1.710 x 10 <sup>-5</sup>
25	4.751	1.774 x 10 <sup>-5</sup>
30	4.740	1.820 x 10 <sup>-5</sup>
35	4.733	1.849 x 10 <sup>-5</sup>
40	4.730	1.862 x 10 <sup>-5</sup>
45	4.726	1.879 x 10 <sup>-5</sup>
50	4.723	1.892 x 10 <sup>-5</sup>

±0.005, determined by emf method by R.G. Bates and G.D. Pinching

Note: pK<sub>b</sub> = pK<sub>w</sub> - pK<sub>a</sub>

where pK<sub>w</sub> = 14 and pK<sub>a</sub> = [H<sup>+</sup>]{NH<sub>3</sub>}/[NH<sub>4</sub><sup>+</sup>]

### Heat of Solution

When liquid anhydrous ammonia is dissolved in water, heat is liberated which varies with the final

concentration of aqua ammonia produced.

<u>Final Wt % NH<sub>3</sub></u>	<u>BTU/lb. NH<sub>3</sub></u>
10.0	343.8
20.0	328.5
30.0	308.2
40.0	270.0
50.0	218.8

### Reactivity

Aqua ammonia will react with many organic and inorganic acids to form ammonium salts and compounds; with certain metals to form complex-ion salts; with halogens to form haloamines (such as its reaction with sodium hypochlorite [bleach] to form toxic chloramines); and under extreme circumstances with silver and mercury to form explosive azides.

Aqua ammonia corrodes copper (and copper-containing alloys such as brass), zinc, cadmium and silver.

For chemical corrosivity information, see the Materials Compatibility section on page 6.

## Physical Properties

Aqua ammonia is a clear, colorless liquid having a strong pungent ammonia odor.

### Specific Gravity

The specific gravity of aqua ammonia is customarily expressed as its density at 60°F compared to the density of water at 60°F. Comprehensive tables of specific gravity, as well as corrections to use for temperature variations, are presented on pages 16-20 in Appendix A.

<u>Weight % Ammonia</u>	<u>Specific Gravity at 60°F/60°F</u>	<u>Degrees Baumé</u>	<u>Weight % Ammonia</u>	<u>Specific Gravity at 60°F/60°F</u>	<u>Degrees Baumé</u>
0.00	1.0000	10.00	20.64	0.9241	21.50
0.40	0.9982	10.25	21.12	0.9226	21.75
0.80	0.9964	10.50	21.60	0.9211	22.00
1.21	0.9947	10.75	22.08	0.9195	22.25
1.62	0.9929	11.00	22.56	0.9180	22.50
2.04	0.9912	11.25	23.04	0.9165	22.75
2.46	0.9894	11.50	23.52	0.9150	23.00
2.88	0.9876	11.75	24.01	0.9135	23.25
3.30	0.9859	12.00	24.50	0.9121	23.50
3.73	0.9842	12.25	24.99	0.9106	23.75
4.16	0.9825	12.50	25.48	0.9091	24.00
4.59	0.9807	12.75	25.97	0.9076	24.25
5.02	0.9790	13.00	26.46	0.9061	24.50
5.45	0.9773	13.25	26.95	0.9047	24.75
5.88	0.9756	13.50	27.44	0.9032	25.00
6.31	0.9739	13.75	27.93	0.9018	25.25
6.74	0.9722	14.00	28.42	0.9003	25.50
7.17	0.9705	14.25	28.91	0.8989	25.75
7.61	0.9689	14.50	29.40	0.8974	26.00
8.05	0.9672	14.75	29.89	0.8960	26.25
8.49	0.9655	15.00	30.38	0.8946	26.50
8.93	0.9639	15.25	30.87	0.8931	26.75
9.38	0.9622	15.50	31.36	0.8917	27.00
9.83	0.9605	15.75	31.85	0.8903	27.25
10.28	0.9589	16.00	32.34	0.8889	27.50
10.73	0.9573	16.25	32.83	0.8875	27.75
11.18	0.9556	16.50	33.32	0.8861	28.00
11.64	0.9540	16.75	33.81	0.8847	28.25
12.10	0.9524	17.00	34.30	0.8833	28.50
12.56	0.9508	17.25	34.79	0.8819	28.75
13.02	0.9492	17.50	35.28	0.8805	29.00
13.49	0.9475	17.75			
13.96	0.9459	18.00			
14.43	0.9444	18.25			
14.90	0.9428	18.50			
15.37	0.9412	18.75			
15.84	0.9396	19.00			
16.32	0.9380	19.25			
16.80	0.9365	19.50			
17.28	0.9349	19.75			
17.76	0.9333	20.00			
18.24	0.9318	20.25			
18.72	0.9302	20.50			
19.20	0.9287	20.75			
19.68	0.9272	21.00			
20.16	0.9256	21.25			

Data of Ferguson from Lange's Handbook of Chemistry (5th Edition).

### Pressure

The total vapor pressure of an aqua ammonia solution is comprised of the partial vapor pressure due to NH<sub>3</sub> plus the partial vapor pressure due to H<sub>2</sub>O. Information on these pressures as a function of temperature is provided in Airgas Technical Bulletin TB-9-1, which is reproduced in Appendix A, page 21. More comprehensive tables of this relationship are presented in Appendix A, pages 22-25. Note that any pressure due to the presence of air in a storage tank or system is additional to the aqua ammonia vapor pressure.

# AQUA AMMONIA

## Solubility

Water and ammonia are miscible in all proportions. When one refers to the solubility of ammonia in water, it is usually meant to be the solubility at a given temperature for which the vapor pressure is equal to atmospheric pressure.

**Temperature °F**                      **Wt. % Ammonia Solubility**

32	47.3
50	40.6
68	34.1
86	29.0
104	25.3
122	22.1
140	19.2
158	16.2
176	13.3
194	10.2
212	6.9

For a graph of solubility vs. temperature, see Appendix A, page 26

## Freezing Point

The freezing point of 29.4% ammonia is about -111°F

**Weight Percent of Ammonia in Aqua Ammonia**

**Freezing Point °F**

100	-107.7
94.4	-113.6
89.9	-118.7
85.8	-123.7
80.7	-134.5
77.5	-127.7
72.4	-116.0
69.9	-112.0
68.7	-111.5
65.4	-109.8
64.5	-110.0
63.3	-110.6

**Weight Percent of Ammonia in Aqua Ammonia**

**Freezing Point °F**

60.6	-113.8
59.9	-115.1
59.3	-116.1
58.9	-117.2
57.6	-121.4
55.6	-119.4
51.6	-112.4
48.8	-110.4
48.7	-110.2
47.9	-110.4
42.3	-117.4
40.8	-122.8
39.2	-126.8
38.4	-131.8
37.7	-133.1
34.4	-142.8
32.7	-142.1
31.4	-128.5
28.5	-101.6
27.5	-91.8
26.4	-82.7
25.4	-74.9
22.0	-46.3
19.3	-30.8
17.1	-19.5
4.22	23.4
0.00	32.0
Eutectic I: 33.4%	-148.5
Eutectic II: 57.1%	-122.8
Eutectic III: 80.5%	-134.5

Data: S. Postma Recveil des Travaux Chimigues des Pays-Bas 39,515 (1920)

For a graph of freezing point versus temperature, see Appendix A, page 27.

## Boiling Point

The boiling point of aqua ammonia is defined as the temperature at which the partial vapor pressure of the ammonia vapor over the aqua ammonia equals atmospheric pressure.

<u>Degrees Be'</u> <u>at 60°</u>	<u>Weight % NH<sub>3</sub></u> <u>Concentration</u>	<u>Boiling Point</u> <u>°F</u>
10	0.00	212
11	1.62	195
12	3.30	186
13	5.02	177
14	6.74	171
15	8.49	163
16	10.28	156
17	12.10	149
18	13.96	142
19	15.84	134
20	17.76	127
21	19.68	120
22	21.60	111
23	23.52	103
24	25.48	95
25	27.44	88
26	29.40	81
27	31.36	73
28	33.32	66
29	35.28	59

## Viscosity

Aqua ammonia viscosity is higher than that for liquid anhydrous ammonia. The viscosities shown below are for 26% concentration aqua ammonia.

<u>Temperature °F</u>	<u>Centipoise</u>
-40	5.0
0	2.8
40	1.7
80	1.1
120	0.7

from Perry's *Chemical Engineer's Handbook* (1984)

## Surface Tension

The surface tension of aqua ammonia at 67°F for various concentrations is shown below:

<u>% NH<sub>3</sub></u>	<u>Surface Tension</u> <u>(dynes/cm)</u>
1.72	71.65
3.39	70.65
4.99	69.95
9.51	67.85
17.37	65.25
34.47	61.05
54.37	57.05

from Perry's *Chemical Engineer's Handbook* (1984)

## Conversions

### Dilutions

The calculations required to determine the volume of anhydrous ammonia or aqua ammonia of an initial concentration to mix with water to create a specific concentration aqua ammonia do not follow normal dilution rules since the anhydrous ammonia and aqua ammonia volumes are not additive with water volumes, i.e., one gallon of anhydrous ammonia added to nine gallons of water does not result in 10 gallons of solution. The final volume would be less than 10 gallons. For many aqua dilutions, the non-additive effects are minimal. For anhydrous additions, they are significant.

The steps to calculate dilutions are as follows:

- Let  $V_o$  = volume in gallons of original concentration aqua ammonia or anhydrous ammonia  
 $C_o$  = concentration in wt. % NH<sub>3</sub> of anhydrous ammonia or original aqua ammonia solution used  
 $V_f$  = volume in gallons of final solution desired  
 $C_f$  = concentration in wt. % NH<sub>3</sub> of final aqua ammonia solution desired  
 $V_w$  = volume in gallons of water to be added
- Determine specific gravities at 60°F/60°F of both original and final concentrations of aqua ammonias by referring to tables in "Physical Properties" on page 11. Interpolation is used to

# AQUA AMMONIA

calculate intermediate values. For anhydrous ammonia, use: specific gravity = 0.6182

3. Let  $Sg_o$  = specific gravity of anhydrous ammonia or original concentration aqua ammonia

$Sg_f$  = specific gravity of final concentration aqua ammonia

$Sg_w$  = 1.0000 specific gravity of water

4. Two facts are known. First, the weight of the original anhydrous ammonia or aqua solution plus the weight of the water added must equal the weight of the final solution. Second, the weight of the ammonia ( $NH_3$ ) present originally (either as anhydrous ammonia or in the original aqua ammonia) must equal the weight of the ammonia ( $NH_3$ ) in the final solution. Therefore, two equations with two unknowns are generated from which desired values can be calculated. The “ammonia equation” becomes  $(V_o)(SG_o)(C_o) = (V_f)(SG_f)(C_f)$  and the “weight equation”  $(V_o)(SG_o) + V_w = (V_f)(SG_f)$ .

Example: What volume anhydrous ammonia ( $NH_3$ ) would you add to what volume of water to obtain 1,000 gallons of 29.4% aqua ammonia?

The “ammonia equation” becomes:

$$V_o = V_f(SG_f)(C_f)/(SG_o)(C_o)$$

$$\text{or } V_o = 1,000(0.8974)(0.294)/(0.6182)(1.00)$$

$$\text{or } V_o = 426.7 \text{ gallons}$$

The “weight equation” becomes:

$$V_w = (V_f)(SG_f) - (V_o)(SG_o)$$

$$\text{or } V_w = (1,000)(0.8974) - (426.7)(0.6182)$$

$$\text{or } V_w = 633.6 \text{ gallons}$$

Note that  $426.7 + 633.6$  does not equal 1,000. There has been a decrease of about 6% in volume in the mixing process.

## References

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2. Reagent Chemicals, 8th Edition, 1993 American Chemical Society, Washington, D.C.
3. United States Pharmacopoeia 21 - National Formulary 16, 1985 Edition, United States Pharmacopoeial Convention, Inc.
4. Food Chemicals Codex, 4th Edition, 1996, National Academy Press, Washington, D.C.

5. Anhydrous Ammonia G-2-1995, 8th Edition, 1995, Compressed Gas Association, Inc.

6. Anhydrous Ammonia Safety, 1989, Airgas Industries, Inc.

7. Perry's Chemical engineers Handbook, 6th Edition, 1984, McGraw Hill Book Company



# Appendix

Charts

Graphs

Tables



## °Baumé/Specific Gravity/Temperature/Concentration Table For Ammonium Hydroxide

°Bé °F/60°F	Sp. Gr. °F/60°F	Percent Ammonia At						
		40°F	50°F	60°F	70°F	80°F	90°F	100°F
12.6	0.9818	4.8	4.6	4.4	4.1	3.8	3.4	2.9
12.8	0.9804	5.1	4.9	4.7	4.4	4.1	3.7	3.2
13.0	0.9790	5.5	5.3	5.0	4.7	4.4	4.0	3.5
13.2	0.9777	5.8	5.6	5.4	5.0	4.7	4.3	3.8
13.4	0.9763	6.2	6.0	5.7	5.4	5.0	4.6	4.1
13.6	0.9749	6.5	6.3	6.0	5.7	5.4	4.9	4.4
13.8	0.9736	6.9	6.7	6.4	6.1	5.7	5.2	4.7
14.0	0.9722	7.3	7.0	6.7	6.4	6.0	5.5	5.0
14.2	0.9709	7.6	7.4	7.0	6.7	6.3	5.8	5.2
14.4	0.9695	8.0	7.8	7.4	7.1	6.7	6.2	5.5
14.6	0.9682	8.4	8.1	7.8	7.4	7.0	6.5	5.8
14.8	0.9669	8.8	8.5	8.1	7.7	7.3	6.8	6.2
15.0	0.9655	9.1	8.8	8.5	8.1	7.6	7.1	6.5
15.2	0.9642	9.5	9.2	8.8	8.4	8.0	7.4	6.8
15.4	0.9629	9.9	9.5	9.2	8.8	8.3	7.7	7.1
15.6	0.9615	10.3	9.9	9.5	9.1	8.6	8.0	7.4
15.8	0.9602	10.7	10.3	9.9	9.5	8.9	8.4	7.7
16.0	0.9589	11.0	10.7	10.3	9.8	9.3	8.7	8.0
16.2	0.9576	11.4	11.0	10.6	10.2	9.6	9.0	8.3
16.4	0.9563	11.8	11.4	11.0	10.5	9.9	9.3	8.7
16.6	0.9550	12.2	11.8	11.4	10.9	10.3	9.7	9.0
16.8	0.9537	12.6	12.2	11.8	11.2	10.7	10.0	9.3
17.0	0.9524	13.0	12.6	12.1	11.6	11.0	10.4	9.6
17.2	0.9511	13.4	13.0	12.5	12.0	11.4	10.7	10.0
17.4	0.9498	13.8	13.4	12.9	12.3	11.7	11.0	10.3
17.6	0.9485	14.2	13.7	13.2	12.7	12.0	11.3	10.6
17.8	0.9472	14.6	14.1	13.6	13.0	12.4	11.7	10.9
18.0	0.9459	15.0	14.5	14.0	13.4	12.7	12.0	11.2
18.2	0.9447	15.3	14.9	14.3	13.7	13.1	12.3	11.5
18.4	0.9434	15.8	15.3	14.7	14.1	13.5	12.7	11.9
18.6	0.9421	16.2	15.6	15.0	14.4	13.8	13.0	12.2
18.8	0.9409	16.6	16.0	15.4	14.8	14.1	13.4	12.5
19.0	0.9396	17.0	16.5	15.9	15.2	14.5	13.8	12.9
19.2	0.9383	17.4	16.8	16.2	15.5	14.9	14.1	13.2
19.4	0.9371	17.8	17.2	16.6	15.9	15.2	14.4	13.6
19.6	0.9358	18.2	17.6	17.0	16.3	15.6	14.8	13.9
19.8	0.9346	18.6	18.0	17.4	16.7	16.0	15.1	14.2

## °Baumé/Specific Gravity/Temperature/Concentration Table For Ammonium Hydroxide, con't.

°Bé	Sp. Gr	Percent Ammonia At						
		°F/60°F	40°F	50°F	60°F	70°F	80°F	90°F
20.0	0.9333	19.1	18.5	17.8	17.1	16.3	15.5	14.6
20.2	0.9321	19.4	18.8	18.1	17.4	16.7	15.8	14.9
20.4	0.9309	19.9	19.2	18.5	17.8	17.1	16.2	15.3
20.6	0.9296	20.4	19.6	18.9	18.2	17.4	16.5	15.6
20.8	0.9284	20.7	20.0	19.3	18.6	17.8	16.9	16.0
21.0	0.9272	21.1	20.4	19.7	19.0	18.1	17.2	16.3
21.2	0.9259	21.5	20.8	20.1	19.3	18.5	17.5	16.7
21.4	0.9247	21.8	21.2	20.5	19.7	18.9	17.9	17.0
21.6	0.9235	22.3	21.6	20.8	20.0	19.2	18.2	17.3
21.8	0.9223	22.7	22.0	21.3	20.4	19.6	18.6	17.7
22.0	0.9211	23.1	22.4	21.6	20.8	19.9	18.9	18.0
22.2	0.9198	23.5	22.8	22.0	21.1	20.3	19.2	18.3
22.4	0.9186	23.9	23.2	22.4	21.5	20.6	19.6	18.7
22.6	0.9174	24.4	23.6	22.7	21.9	21.0	19.9	19.0
22.8	0.9162	24.8	24.0	23.1	22.3	21.3	20.3	19.3
23.0	0.9150	25.2	24.4	23.5	22.7	21.7	20.6	19.7
23.2	0.9138	25.6	24.9	23.9	23.0	22.0	20.9	20.0
23.4	0.9126	26.0	25.2	24.3	23.4	22.4	21.3	20.4
23.6	0.9111	26.4	25.6	24.7	23.8	22.8	21.6	20.7
23.8	0.9103	26.8	26.0	25.1	24.2	23.2	22.0	21.0
24.0	0.9091	27.3	26.4	25.5	24.5	23.5	22.3	21.3
24.2	0.9079	27.7	26.8	25.8	24.9	23.8	22.6	21.7
24.4	0.9067	28.2	27.2	26.2	25.3	24.2	23.0	22.0
24.6	0.9056	28.5	27.6	26.6	25.6	24.6	23.3	22.3
24.8	0.9044	28.9	28.0	27.0	26.0	24.9	23.6	22.6
25.0	0.9032	29.4	28.5	27.4	26.4	25.3	24.0	23.0
25.2	0.9021	29.8	28.9	27.8	26.7	25.6	24.3	23.3
25.4	0.9009	30.2	29.3	28.2	27.1	26.0	24.6	23.7
25.6	0.8997	30.6	29.6	28.6	27.5	26.4	24.9	24.0
25.8	0.8986	31.0	30.1	29.0	27.9	26.8	25.3	24.4
26.0	0.8974	31.5	30.5	29.4	28.3	27.1	25.7	
26.2	0.8963	31.9	30.9	29.8	28.7	27.5	26.0	
26.4	0.8951	32.4	31.3	30.2	29.0	27.9	26.4	
26.6	0.8940	32.7	31.7	30.5	29.4	28.2	26.7	
26.8	0.8929	33.2	32.1	30.9	29.7	28.6		
27.0	0.8917	33.6	32.5	31.3	30.1	28.9		
27.2	0.8906	34.0	32.9	31.7	30.5	29.3		
27.4	0.8895		33.3	32.1	30.8			
27.6	0.8883		33.7	32.5	31.2			
27.8	0.8872		34.1	32.9	31.6			
28.0	0.8861			33.3	32.0			

## °Baumé/Specific Gravity/Temperature/Concentration Table For Ammonium Hydroxide

°Bé °F/60°F	Sp. Gr. °F/60°F	Percent Ammonia At						
		40°F	50°F	60°F	70°F	80°F	90°F	100°F
12.6	0.9818	4.8	4.6	4.4	4.1	3.8	3.4	2.9
12.8	0.9804	5.1	4.9	4.7	4.4	4.1	3.7	3.2
13.0	0.9790	5.5	5.3	5.0	4.7	4.4	4.0	3.5
13.2	0.9777	5.8	5.6	5.4	5.0	4.7	4.3	3.8
13.4	0.9763	6.2	6.0	5.7	5.4	5.0	4.6	4.1
13.6	0.9749	6.5	6.3	6.0	5.7	5.4	4.9	4.4
13.8	0.9736	6.9	6.7	6.4	6.1	5.7	5.2	4.7
14.0	0.9722	7.3	7.0	6.7	6.4	6.0	5.5	5.0
14.2	0.9709	7.6	7.4	7.0	6.7	6.3	5.8	5.2
14.4	0.9695	8.0	7.8	7.4	7.1	6.7	6.2	5.5
14.6	0.9682	8.4	8.1	7.8	7.4	7.0	6.5	5.8
14.8	0.9669	8.8	8.5	8.1	7.7	7.3	6.8	6.2
15.0	0.9655	9.1	8.8	8.5	8.1	7.6	7.1	6.5
15.2	0.9642	9.5	9.2	8.8	8.4	8.0	7.4	6.8
15.4	0.9629	9.9	9.5	9.2	8.8	8.3	7.7	7.1
15.6	0.9615	10.3	9.9	9.5	9.1	8.6	8.0	7.4
15.8	0.9602	10.7	10.3	9.9	9.5	8.9	8.4	7.7
16.0	0.9589	11.0	10.7	10.3	9.8	9.3	8.7	8.0
16.2	0.9576	11.4	11.0	10.6	10.2	9.6	9.0	8.3
16.4	0.9563	11.8	11.4	11.0	10.5	9.9	9.3	8.7
16.6	0.9550	12.2	11.8	11.4	10.9	10.3	9.7	9.0
16.8	0.9537	12.6	12.2	11.8	11.2	10.7	10.0	9.3
17.0	0.9524	13.0	12.6	12.1	11.6	11.0	10.4	9.6
17.2	0.9511	13.4	13.0	12.5	12.0	11.4	10.7	10.0
17.4	0.9498	13.8	13.4	12.9	12.3	11.7	11.0	10.3
17.6	0.9485	14.2	13.7	13.2	12.7	12.0	11.3	10.6
17.8	0.9472	14.6	14.1	13.6	13.0	12.4	11.7	10.9
18.0	0.9459	15.0	14.5	14.0	13.4	12.7	12.0	11.2
18.2	0.9447	15.3	14.9	14.3	13.7	13.1	12.3	11.5
18.4	0.9434	15.8	15.3	14.7	14.1	13.5	12.7	11.9
18.6	0.9421	16.2	15.6	15.0	14.4	13.8	13.0	12.2
18.8	0.9409	16.6	16.0	15.4	14.8	14.1	13.4	12.5
19.0	0.9396	17.0	16.5	15.9	15.2	14.5	13.8	12.9
19.2	0.9383	17.4	16.8	16.2	15.5	14.9	14.1	13.2
19.4	0.9371	17.8	17.2	16.6	15.9	15.2	14.4	13.6
19.6	0.9358	18.2	17.6	17.0	16.3	15.6	14.8	13.9
19.8	0.9346	18.6	18.0	17.4	16.7	16.0	15.1	14.2

## Table of Corrections To Aqua Ammonia Specific Gravity Readings To Compensate For Sampling Temperature Variations

Correction To Specific Gravity Reading For Each  
Fahrenheit Degree Variation From The 60°F Standard

Observed Specific Gravity	Observed Temperature						
	40°F	50°F	60°F	70°F	80°F	90°F	100°F
0.9524	-0.00016	-0.00017	0.00000	+0.00018	+0.00019	+0.00021	+0.00022
0.9459	-0.00017	-0.00019	0.00000	+0.00020	+0.00021	+0.00022	+0.00024
0.9396	-0.00019	-0.00020	0.00000	+0.00021	+0.00022	+0.00023	+0.00025
0.9333	-0.00021	-0.00022	0.00000	+0.00023	+0.00024	+0.00025	+0.00026
0.9272	-0.00022	-0.00024	0.00000	+0.00025	+0.00026	+0.00026	+0.00028
0.9211	-0.00024	-0.00025	0.00000	+0.00027	+0.00027	+0.00029	+0.00029
0.9150	-0.00025	-0.00027	0.00000	+0.00028	+0.00029	+0.00030	-----
0.9091	-0.00027	-0.00029	0.00000	+0.00029	+0.00031	+0.00032	-----
0.9032	-0.00029	-0.00031	0.00000	+0.00031	+0.00032	-----	-----
0.8974	-0.00030	-0.00033	0.00000	+0.00033	+0.00034	-----	-----

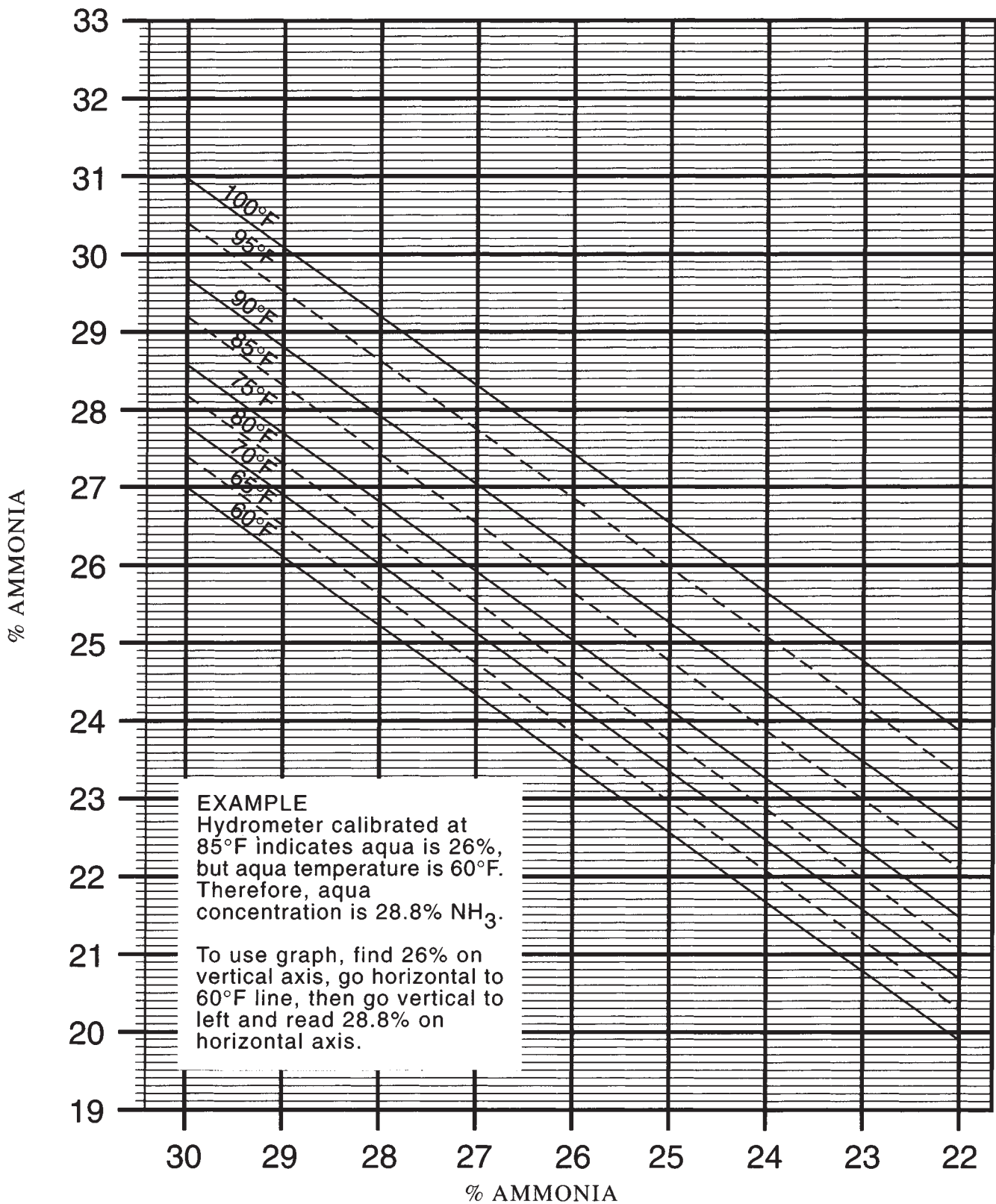
Calculating Specific Gravity Adjusted To 60°F Standard Temperature:

It is satisfactory for most situations simply to find the values for specific gravity and temperature on the chart which are closest to the observed values and use the indicated correction factor.

Example: Find the specific gravity adjusted to 60°F for an observed 0.9163 specific gravity measured at 78°F. The closest specific gravity in the table is 0.9150. Round off 78°F to 80°F. The correction value found in the table at the intersection of 0.9150 specific gravity and 80°F is +0.00029 per Fahrenheit degree. The difference between the observed temperature and the standard 60°F is 18 F°. The adjusted specific gravity reading is  $0.9163 + (+0.00029)(18) = 0.9163 + 0.0052 = 0.9215$ . Properly stated, the specific gravity adjusted to 60°F is 0.9215.

It is possible to apply a mathematical estimating tool called interpolation to increase the accuracy in the determination of the correction factor. However, the difference in the final calculated adjusted specific gravity is usually negligible. For instance, in the above example, interpolation results in a correction factor of +0.000284 and an end result of 0.9214 specific gravity.

## AQUA AMMONIA CONCENTRATION CONVERSIONS





## ABSOLUTE PRESSURE OF AMMONIA-WATER SOLUTIONS

Pressures at Temperatures Shown, psia

Ammonia Concentration														
%	32°F	40°F	50°F	60°F	70°F	80°F	90°F	100°F	110°F	120°F	130°F	140°F		
2	0.2	0.3	0.3	0.5	0.7	1.0	1.3	1.7	2.2	2.8	4.5	7.3		
4	0.3	0.4	0.5	0.7	1.0	1.3	1.8	2.4	3.1	3.9	6.1	9.3		
6	0.4	0.6	0.7	0.9	1.2	1.8	2.3	3.0	3.8	4.8	7.6	11.2		
8	0.6	0.7	0.8	1.2	1.5	2.2	2.8	3.6	4.5	5.8	8.9	13.2		
10	0.7	0.8	1.1	1.5	1.9	2.5	3.3	4.2	5.3	6.8	10.3	15.5		
12	0.8	1.0	1.2	1.7	2.3	3.1	4.0	5.1	6.4	7.9	12.1	18.1		
14	1.0	1.2	1.6	2.2	2.8	3.7	4.8	6.1	7.6	9.4	14.1	21.2		
16	1.2	1.5	1.9	2.7	3.5	4.4	5.6	7.2	9.0	11.1	16.8	24.5		
18	1.4	1.8	2.3	3.2	4.2	5.3	6.7	8.5	10.6	13.2	19.7	28.5		
20	1.8	2.2	2.9	3.8	5.0	6.4	8.1	10.1	12.5	15.6	23.1			
22	2.2	2.7	3.6	4.6	6.0	7.7	9.6	12.1	14.9	18.3	27.4			
24	2.6	3.3	4.3	5.6	7.1	9.1	11.4	14.2	17.6	21.5	31.5			
26	3.2	4.0	5.2	6.7	8.7	10.8	13.8	17.0	20.8	25.6				
27.4	3.7	4.6	5.9	7.8	9.8	12.2	15.5	19.2	23.5	28.8				
28	3.9	4.8	6.3	8.1	10.3	12.8	16.2	20.2	24.7	30.1				
29.4	4.4	5.5	7.2	9.1	11.7	14.6	18.2	22.8	27.8					
30	4.7	5.8	7.7	9.7	12.3	15.3	19.1	24.0	29.3					
32	5.7	7.0	9.1	11.6	14.6	18.1	22.4	28.1						
34	6.8	8.3	10.7	13.6	17.0	21.3	26.3							
36	8.1	9.8	12.7	15.8	19.7	24.9	30.2							
38	9.5	11.5	14.8	18.6	22.9	28.6								
40	11.0	13.3	17.2	21.6	27.1	32.3								
42	12.7	15.4	19.6	24.7	31.2									
44	14.6	17.7	22.1	27.7										
46	16.7	20.2	24.8	30.8										
48	18.8	22.8	28.1											
50	21.0	25.3												

TB-9-1  
9/92

The technical data in this bulletin is based on information which we believe to be accurate. However, nothing in this bulletin is to be construed as an expressed or implied warranty regarding the accuracy of the data or the use of any of the products herein described. Nothing contained herein should be considered as a recommendation for the use of any product or method in violation of any patent now effective or which may issue hereafter. All risks of liability rest solely with the user of the data or products herein described.

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6340 Sugarloaf Parkway  
Duluth, GA 30097  
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## Partial Vapor Pressure Of Ammonia Over Aqua Ammonia (Pressure indicated in psi)

Temp.°F	4.74	9.5	14.29	19.1	23.94	28.81	33.71	38.64	43.59	48.57	53.58	58.62	63.69	68.79	73.91	79.07	84.26	89.47	94.72
32	0.3	0.5	0.9	1.5	2.7	4.3	6.5	8.9	14.1	19.4	25.1	31.1	36.7	42.7	45.9	49.3	52.1	54.9	58.0
40	0.3	0.7	1.1	1.9	3.2	5.1	8.0	12.0	17.1	23.3	30.2	37.2	43.7	49.6	54.4	58.3	61.6	64.8	68.3
50	0.5	0.9	1.5	2.5	4.2	6.6	10.2	15.2	21.6	29.2	37.5	45.9	53.8	60.8	66.6	71.3	75.2	79.1	83.4
50	0.6	1.2	2.0	3.2	5.4	8.5	13.1	19.2	26.9	36.1	46.1	56.2	65.8	74.0	80.9	86.4	91.0	95.7	100.7
70	0.8	1.5	2.6	4.3	6.9	10.8	16.3	23.8	33.2	44.3	56.3	58.3	79.4	89.3	97.4	104.0	109.6	114.8	120.6
50	1.0	2.0	3.3	6.5	8.7	13.5	20.3	29.4	40.7	53.8	68.0	82.4	95.5	107.1	116.4	124.2	130.6	136.4	143.7
90	1.4	2.5	4.3	6.9	10.9	16.8	25.0	35.9	49.5	65.0	81.6	98.4	113.8	127.2	138.2	147.0	154.5	161.7	169.7
100	1.7	3.2	5.3	8.6	13.5	20.7	30.6	43.6	59.5	77.9	97.3	116.8	134.7	150.2	162.9	173.2	182.0	190.1	199.2
110	2.1	4.0	6.7	10.6	16.7	25.2	37.0	52.4	71.2	92.6	115.2	137.6	158.4	176.2	190.9	203.0	212.7	222.2	232.8
120	2.7	5.0	8.2	13.1	20.3	30.5	44.6	62.6	84.4	109.4	135.5	161.4	185.1	206.8	222.3	236.1	247.1	258.2	270.0
130	3.3	6.1	10.1	15.9	24.6	36.7	53.2	74.3	99.7	128.5	158.5	188.2	215.1	238.7	257.9	272.9	286.1	298.5	311.8
140	4.0	7.4	12.2	19.2	29.4	43.8	63.0	87.6	116.7	149.9	184.2	218.2	248.7	275.3	297.1	314.6	329.0	342.9	358.5
150	4.8	8.9	14.7	23.1	35.1	51.9	74.3	102.5	136.2	173.6	212.9	261.2	286.0	316.2	340.8	360.4	376.6	392.6	409.6
160	5.7	10.7	17.6	27.5	41.6	61.0	86.9	119.4	157.7	200.5	246.0	288.4	327.8	361.6	389.1	411.3	429.7	447.4	466.4
170	6.8	12.7	20.9	32.4	48.9	71.5	101.1	138.3	182.0	230.4	280.5	329.4	373.6	411.6	442.3	466.7	487.9	507.6	528.5
180	7.9	15.0	24.6	36.1	57.2	83.1	117.0	159.4	208.7	263.4	319.9	374.3	424.1	466.3	500.6	528.1	551.2		
150	9.2	17.6	28.8	44.5	66.5	96.2	134.9	182.7	238.4	299.9	363.1	424.2	479.4	526.2					
200	10.7	20.5	33.5	51.6	76.9	110.9	154.6	208.5	270.9	340.0	410.2	478.6	\$39.8						
210	12.3	23.7	38.8	59.7	88.5	126.8	176.2	237.0	307.1	384.0	462.4	537.8							
220	14.0	27.2	44.6	68.4	101.2	144.7	200.5	268.3	346.1	431.4	518.2								
230	16.0	31.1	51.1	78.1	116.5	164.2	226.7	302.5	389.3	483.5									
240	17.9	35.4	58.0	89.0	130.9	185.8	255.3	339.7	435.8	540.4									
250	20.1	40.1	65.7	100.7	147.7	209.4	286.9	380.4	486.7										

Adapted from Wilson, University of Illinois, Eng. Expt. Sta. Bull. 146

## Partial Vapor Pressure Of Ammonia Over Aqua Ammonia (Pressure indicated in psi)

Temp. °F	Weight % Concentration Of Ammonia In Aqua Ammonia																		
	4.74	9.50	14.29	19.10	23.94	28.81	33.71	38.64	43.59	48.57	53.58	58.62	63.69	68.79	73.91	79.07	84.26	89.47	94.72
32	0.08	0.08	0.07	0.07	0.06	0.06	0.06	0.05	0.05	0.04	0.04	0.03	0.03	0.02	0.02	0.02	0.01	0.01	0.00
40	0.12	0.11	0.10	0.10	0.09	0.08	0.08	0.07	0.06	0.06	0.05	0.05	0.04	0.04	0.03	0.02	0.02	0.01	0.01
50	0.17	0.16	0.15	0.14	0.13	0.12	0.11	0.10	0.09	0.08	0.08	0.07	0.06	0.05	0.04	0.03	0.02	0.02	0.01
60	0.24	0.23	0.21	0.20	0.19	0.17	0.16	0.15	0.13	0.12	0.11	0.10	0.08	0.07	0.06	0.05	0.04	0.02	0.01
70	0.34	0.32	0.30	0.28	0.26	0.25	0.23	0.21	0.19	0.17	0.15	0.14	0.12	0.10	0.09	0.07	0.05	0.03	0.02
50	0.48	0.45	0.42	0.40	0.37	0.34	0.32	0.29	0.27	0.24	0.22	0.19	0.17	0.14	0.12	0.10	0.07	0.05	0.02
90	0.66	0.63	0.58	0.55	0.51	0.47	0.44	0.40	0.37	0.33	0.30	0.26	0.23	0.20	0.16	0.13	0.10	0.07	0.03
100	0.90	0.85	0.79	0.74	0.69	0.64	0.59	0.55	0.50	0.45	0.41	0.36	0.31	0.27	0.22	0.18	0.13	0.09	0.04
110	1.20	1.14	1.07	1.00	0.93	0.86	0.80	0.73	0.67	0.60	0.54	0.48	0.42	0.36	0.30	0.24	0.18	0.12	0.06
120	1.60	1.51	1.42	1.33	1.24	1.15	1.06	0.97	0.89	0.80	0.72	0.64	0.56	0.48	0.40	0.32	0.24	0.16	0.08
130	2.10	1.98	1.86	1.74	1.62	1.51	1.39	1.28	1.17	1.05	0.95	0.84	0.74	0.63	0.53	0.42	0.32	0.21	0.10
140	2.73	2.57	2.42	2.26	2.11	1.96	1.81	1.66	1.52	1.37	1.23	1.10	0.96	0.82	0.69	0.55	0.41	0.27	0.14
150	3.51	3.31	3.11	2.91	2.72	2.52	2.33	2.14	1.95	1.76	1.59	1.41	1.24	1.06	0.88	0.71	0.53	0.35	0.18
150	4.48	4.22	3.97	3.71	3.46	3.22	2.97	2.73	2.49	2.25	2.02	1.80	1.58	1.35	1.12	0.90	0.67	0.45	0.22
170	5.66	5.34	5.02	4.70	4.38	4.07	3.75	3.45	3.15	2.84	2.56	2.28	1.99	1.71	1.42	1.13	0.85	0.58	0.30
150	7.10	6.69	6.30	5.89	5.49	5.10	4.71	4.33	3.94,	3.57	3.21	2.85	2.50	2.14	1.77	1.42	1.06		
190	8.83	8.32	7.82	7.32	6.83	6.34	5.86	5.38	4.91	4.44	3.99	3.55	3.10	2.65					
200	10.90	10.27	9.65	9.04	8.43	7.83	7.23	6.64	6.06	5.48	4.93	4.38	3.81						
210	13.35	12.58	11.82	11.07	10.32	9.59	8.86	8.13	7.42	6.71	6.04	5.34							
220	16.25	15.32	14.39	13.48	12.57	11.67	10.78	9.90	9.03	8.17	7.31								
230	19.64	18.51	17.40	16.29	15.19	14.11	13.03	11.97	10.91	9.87									
240	23.60	22.25	20.91	19.53	18.26	16.95	15.66	14.38	13.12	11.86									
250	28.20	26.58	25.00	23.39	21.82	20.25	18.71	17.18	15.67										

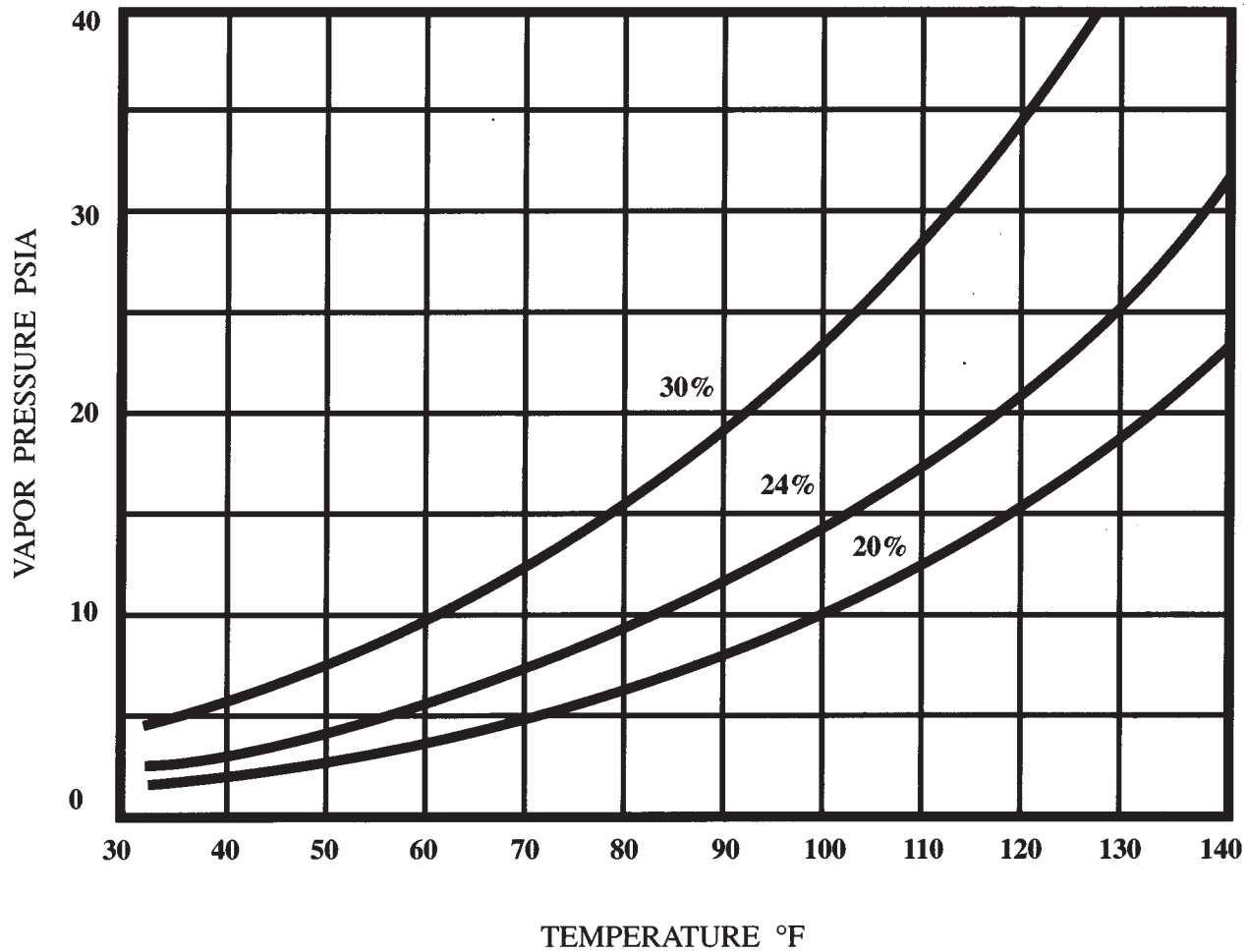
Adapted from Wilson, University of Illinois, Eng. Expt. Sta. Bull. 146

## Total Vapor Pressure (Ammonia + Water) Over Aqua Ammonia (Pressure indicated in psi)

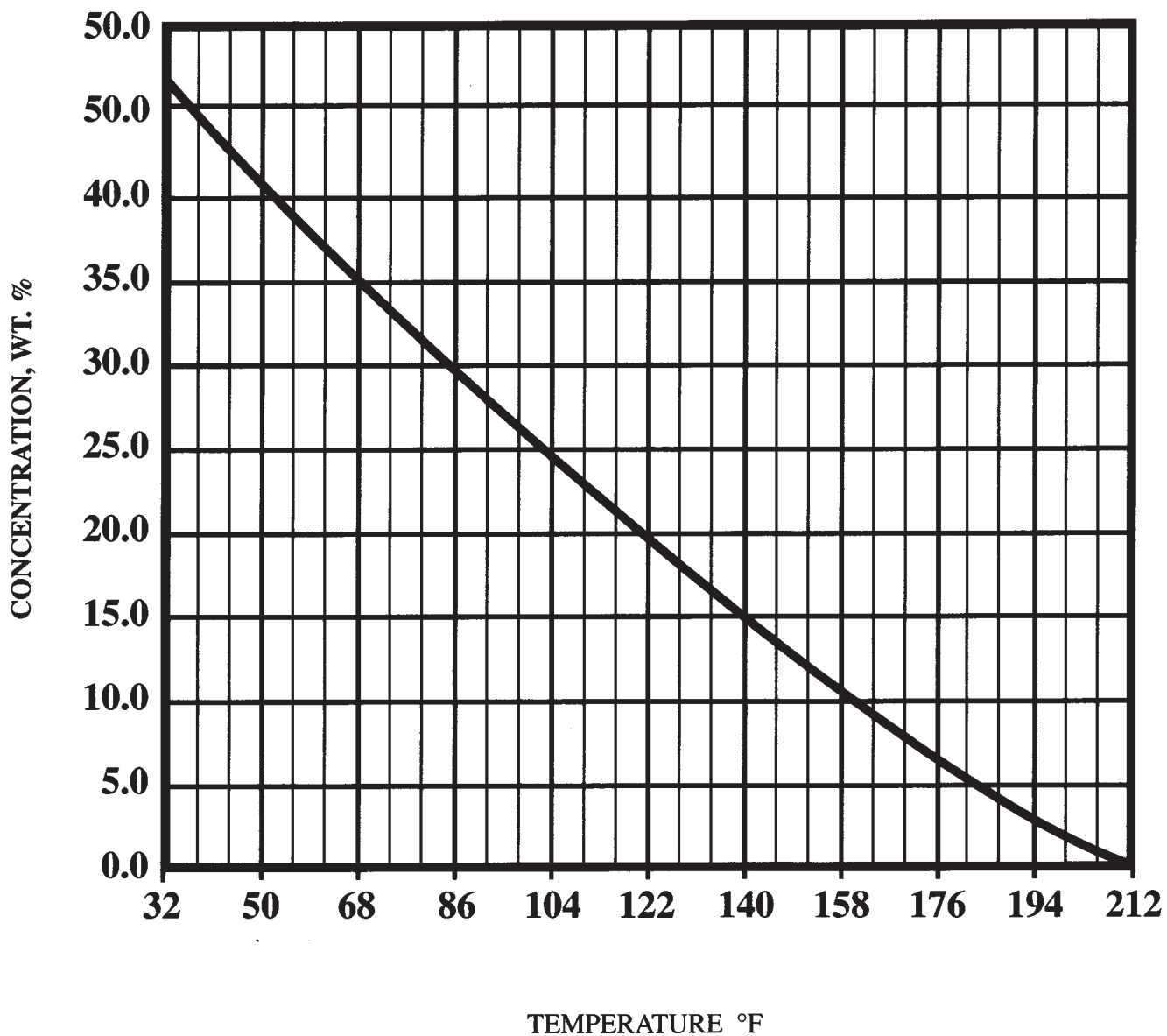
Temp. °F	Weight % Concentration Of Ammonia In Aqua Ammonia																		
	4.74	9.50	14.29	19.10	23.94	28.81	33.71	38.64	43.59	48.57	53.58	58.62	63.69	68.79	73.91	79.07	84.26	89.47	94.72
32	0.3	0.6	1.0	1.6	2.6	4.2	6.5	9.9	14.2	19.4	25.2	31.2	36.8	42.7	45.9	49.3	52.1	54.9	58.0
40	0.4	0.8	1.2	2.0	3.2	5.2	8.1	12.0	17.2	23.4	30.2	37.2	43.7	49.6	54.4	58.3	61.6	64.8	68.3
50	0.6	1.0	1.6	2.7	4.3	6.8	10.4	15.3	21.6	29.3	37.5	45.9	53.8	60.9	66.7	71.3	75.2	79.1	83.4
60	0.9	1.4	2.2	3.5	5.6	8.6	13.2	19.3	27.0	36.3	46.2	56.3	65.9	74.1	81.0	86.5	91.1	95.7	100.7
70	1.2	1.8	2.9	4.6	7.1	11.0	16.6	24.0	33.4	44.4	56.4	68.5	79.5	89.4	97.5	104.1	109.6	114.9	120.6
50	1.5	2.4	3.8	5.8	9.1	13.9	20.6	29.7	41.0	54.1	66.2	82.6	95.7	107.2	116.5	124.3	130.6	136.4	143.7
90	2.0	3.2	4.8	7.4	11.4	17.2	25.5	36.3	49.8	65.3	81.9	98.6	114.0	127.4	138.3	147.2	154.6	161.8	169.8
100	2.6	4.0	6.1	9.3	14.2	21.3	31.2	44.1	60.0	78.3	97.7	117.2	135.0	150.5	163.2	173.4	182.1	190.2	199.2
110	3.3	5.1	7.7	11.6	17.6	26.1	37.8	53.2	71.9	93.2	115.7	138.1	158.8	176.5	191.2	203.3	212.9	222.3	232.8
120	4.3	6.5	9.6	14.4	21.5	31.7	45.6	63.6	85.3	110.2	136.2	162.1	185.7	206.3	222.7	236.4	247.4	258.4	270.1
130	5.4	8.1	11.9	17.7	26.2	38.2	54.6	75.6	100.9	129.6	159.0	189.0	215.9	239.3	258.4	273.3	286.4	298.7	311.9
140	6.7	10.0	14.6	21.5	31.5	45.7	64.8	89.2	118.2	151.3	185.4	219.3	249.7	276.2	297.8	315.0	329.4	343.2	358.5
150	8.3	12.2	17.8	26.0	37.8	54.4	76.6	104.6	138.1	175.4	214.5	252.6	287.2	317.3	341.7	361.1	377.1	392.8	409.8
150	10.2	14.9	21.5	31.2	45.0	64.2	89.9	122.1	160.2	202.7	247.0	290.2	329.4	363.1	390.2	412.2	430.4	447.8	466.6
170	12.4	18.0	25.9	37.1	63.3	75.6	104.8	141.8	185.1	233.2	283.1	331.7	375.6	413.3	443.7	467.8	488.7	508.2	528.8
150	15.0	21.6	30.9	44.0	62.7	88.2	121.7	163.7	212.6	267.0	323.1	377.1	426.6	468.4	502.4	529.5	552.8	578.1	604.6
190	18.1	25.9	36.6	51.8	73.3	102.6	140.8	188.1	243.3	304.3	367.1	427.7	482.5	532.8	578.1	624.1	669.6	714.6	759.1
200	21.6	30.7	43.1	60.6	85.3	118.7	161.8	215.2	277.0	345.5	415.1	483.0	543.6	600.1	652.6	701.1	748.6	795.1	840.6
210	25.6	36.3	50.6	70.7	98.8	136.4	185.1	245.1	314.5	390.7	468.4	542.9	614.6	683.1	747.6	808.1	865.6	920.1	972.6
220	30.3	42.5	59.0	81.9	113.8	156.4	211.2	278.2	355.1	439.6	525.5	614.6	707.1	793.1	874.6	952.6	1028.1	1101.1	1171.6
230	35.6	49.6	68.5	94.4	130.6	178.3	239.7	314.5	400.2	493.4	584.6	674.6	763.1	849.1	931.6	1010.6	1087.1	1161.1	1232.6
240	41.5	57.6	78.9	108.6	149.2	202.7	270.9	354.1	448.9	546.3	638.6	729.6	819.1	907.1	992.6	1075.6	1156.1	1234.1	1309.6
250	48.3	66.7	90.7	124.1	169.5	229.6	305.6	397.6	502.4	609.6	719.6	829.6	929.6	1028.6	1126.6	1223.6	1319.6	1414.6	1508.6

Adapted from Wilson, University of Illinois, Eng. Expt. Sta. Bull. 146

# TOTAL VAPOR PRESSURE OF AMMONIA-WATER SOLUTIONS

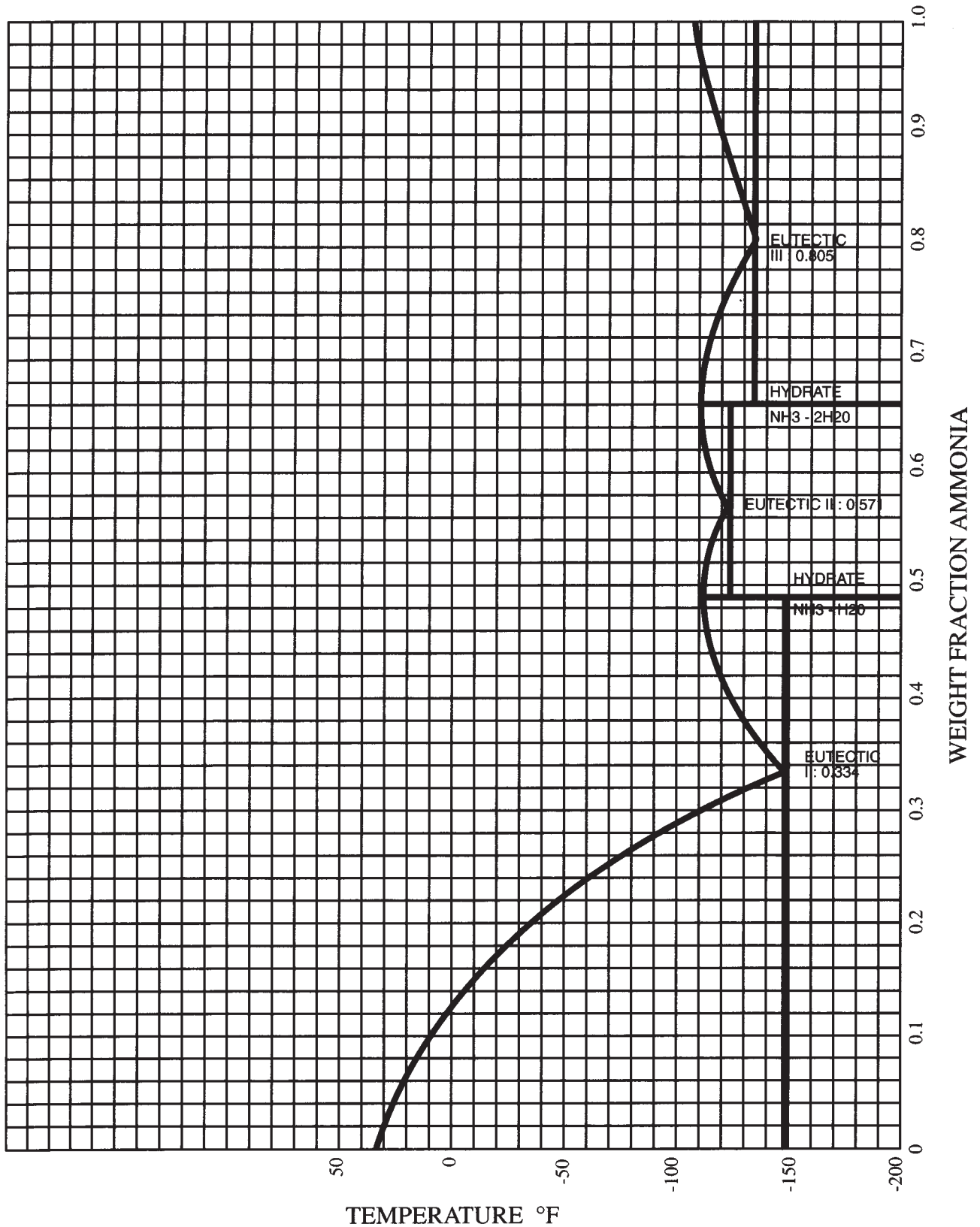


## AMMONIA/WATER SOLUBILITY AT 1 ATMOSPHERE



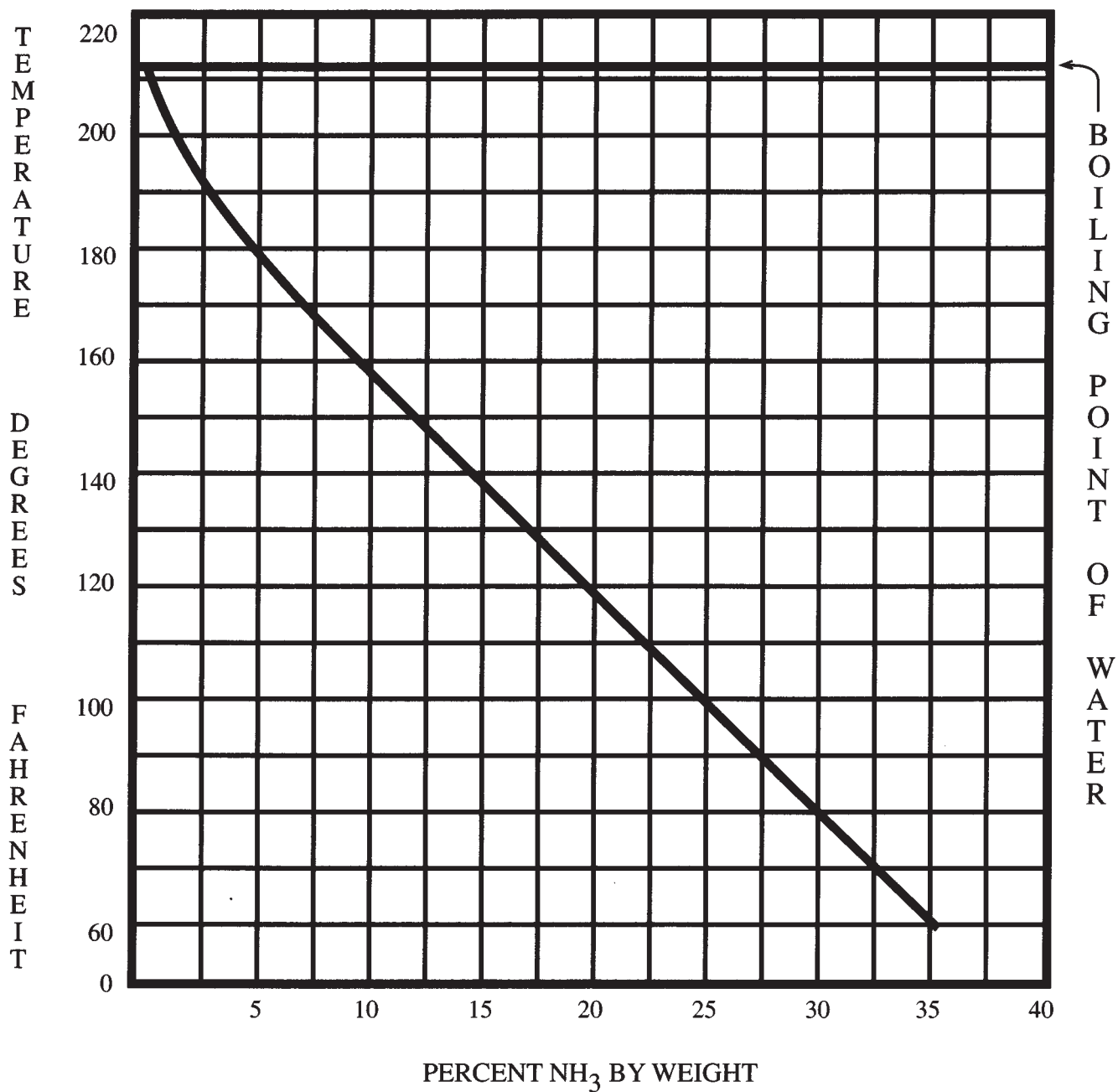


# FREEZING POINT OF AQUA AMMONIA



S. Postma, Recveil des Travaux Chimigues des Pays-Bas 39, 515 (1920)

## BOILING POINT OF AMMONIA SOLUTIONS



# Appendix

## Analytical Methods



## WARNING!

Aqua Ammonia sample containers and contents need to be cooled to between 5°C and 10°C and then maintained at that temperature to prevent loss of ammonia gas (NH<sub>3</sub>) from the sample when opening the container and transferring for tests! All transferring shall be done as quickly as possible!

Failure to follow this requirement is likely to result in erroneous results when testing for ammonia concentrations!

When utilizing hydrometer measurements, realize that a 1 Fahrenheit degree error in the temperature observation results in a concentration measurement error on the order of 0.1%. Similarly, a 0.1 Baumé degree error results in a concentration measurement error on the order of 0.2%. Note that these errors can be additive, e.g., a Baumé reading 0.1° too low concurrent with a temperature reading 1 Fahrenheit degree too high will cause an error in the concentration calculation on the order of 0.3% below the true ammonia concentration!

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The following information is taken from Federal Specification OA-451-F Ammonium Hydroxide Technical, available from the GSA Specification Section, Room 6654, 7th and D Sts., S.W., Washington, D.C. 20407.

Warning - Containers and contents shall be cooled to between 5°C and 10°C and maintained at this temperature to prevent the loss of ammonia gas (NH<sub>3</sub>) when operating and transferring for tests. All transferring shall be done as quickly as possible.

Determine by visual examination.

Evaporate 100 milliliters of the sample to dryness in a tared platinum crucible or other suitable dish, ignite, and weigh. Calculate the gain in weight of the crucible as percent total solids.

Determine specific gravity by method ASTM Designation D1122.

Introduce a glass-stoppered weighing bottle containing about 10g of the sample into an 800ml. Erlenmeyer flask containing about 200ml. Of distilled water and sufficient 0.5N sulfuric acid to combine with the ammonia and about 10ml excess. Stopper the flask and warm gently until the stopper in the weighing bottle is forced out and the ammonia combines with the acid. Upon thorough mixing, allow the solution to cool and titrate the excess acid with 0.5N sodium hydroxide using methyl red as indicator. Calculate the percentage of ammonia in the sample.

$$\% \text{ NH}_3 = [(AB-CD) \times 0.017 \times 100] / s$$

A = ml. Of sulfuric acid

B = Normality of sulfuric acid

C = ml. Of sodium hydroxide solution to titrate excess acid

D = Normality of sodium hydroxide

S = Weight of sample in grams

# AQUA AMMONIA

## Analytical Procedure Ammonium Hydroxide (Aqua Ammonia) Technical Grade

### Procedure For Specific Gravity Test

#### Equipment and Apparatus:

473ml flint glass sample container with cap (Fisher Scientific #03-326-2F or equivalent) 0-120°F thermometer (Taylor Red Line 21413-1 or equivalent)

Vertical cylinder hydrometer flask (Fisher Scientific #08-530K or equivalent) Hydrometer (Fisher Scientific #11-55E or equivalent)

Appendix A, pages 15-18, graphs and charts relating aqua ammonia concentration, degrees Baumé, specific gravity and temperature.

#### Assay:

1. Cool the first four items mentions above to at least 5-10°C (40-50°F).
2. Open sample valve and flush lines. It is recommended that the sample line is routed through a chiller or ice bath to ensure that the sample being withdrawn is cooled to at least 5-10°C (40-50°F) before it is exposed to the atmosphere. Place the thermometer inside a cooled sample container. Fill about 3/4 full of aqua ammonia. Recap sample container. Allow for air bubbles to settle. Record temperature.
3. Place the hydrometer inside the vertical cylinder hydrometer flask. Pour sample into the hydrometer flask. Maintain flask and contents at 5-10°C (40-50°F) if the specific gravity reading requires more than 20 seconds to complete. Allow the hydrometer to stabilize. Record the specific gravity or degrees Baumé reading.
4. Refer to the aqua ammonia concentration conversion graph and additional tables as necessary in Appendix A, pages 15-28. Calculate the final concentration.

5. Compare readings from hydrometers in service with new stand-by hydrometers on a monthly basis. Hydrometers shall be calibrated by an external test laboratory on an annual basis or as often as desired. Calibration shall be done with certified hydrometers or by comparison to titration results.

## Analytical Procedure

### Ammonium Hydroxide (Aqua Ammonia)

#### Procedure For Assay By Titration

##### Principle:

A sample of aqua ammonia is added to an excess of 3N hydrochloric acid. The excess is then backtitrated with 2N sodium hydroxide.

Equipment and Apparatus:

Bottle, pressure, 200ml flask shape, with stopper and spring clamp.

Bottle, weighing, high-form, glass stopper, 40 x 80 mm (70ml) or equivalent. A glass-stoppered 250 ml Erlenmeyer flask is suitable.

Pipet, 50ml calibrated.

Pipet sampling device. This consists of a two-hole #2 rubber stopper through which are inserted a 10ml pipet and a short piece of glass tubing bent at a right angle. A 2oz rubber bulb is used to force aqua ammonia from the sample bottle into the pipet. Since the glass tube is small, a short piece of tubing is needed as an adapter between the glass tube and the bulb.

Buret, 25ml calibrated.

##### Reagents:

Hydrochloric acid, 3N solution. Purchase standardized solution of dilute 250ml of concentrated, reagent grade hydrochloric acid to 1L with distilled water and standardize against standard 2N sodium hydroxide.

Sodium hydroxide, standard 2N solution. Purchase standardized or dilute 160g of 50% sodium hydroxide solution to 1L with recently boiled, distilled water. Standardize against primary standard benzoic acid or potassium acid phthalate.

Methyl red indicator, 0.1% solution in water. Dissolve 0.10g of the sodium salt of methyl red in 100ml of distilled water. If necessary, adjust the pH to 7.

##### Procedure:

1. The sample is received in a pressure bottle. Cool the sample to 50°F or lower as a safety measure and to prevent losses of ammonia.
2. Pipet exactly 50ml of standard 3N hydrochloric acid into the weighing bottle, and obtain the tare weight. By means of the sampling device, and allow it to drain into the weighing bottle with stirring until about 1/3 remains. Lift the pipet, touch off the last drop of drainage, and remove. Obtain the weight of the bottle containing acid sample.
3. Carefully pour the contents of the weighing bottle into a 250ml Erlenmeyer flask. Add 3 drops of methyl red indicator solution and titrate the excess acid with standard 2N sodium hydroxide until the indicator color changes to yellow. Rinse the weighing bottle into the flask and complete the titration.
4. Calculation:  
$$[(3A-2C)/W] \times 0.01703 \times 100 = \% \text{ ammonia by weight}$$
where A=ml 3N HCl, C=ml 2N NaOH, and w=weight of sample in grams.



# AQUA AMMONIA

## Analytical Procedure Ammonium Hydroxide (Aqua Ammonia) FCC Grade

The following test procedures are reprinted with permission from the FOOD Chemical Codex. Fourth Edition, Copyright 1996 by the National Academy of Sciences. Courtesy of the National Academy Press, Washington, D.C.

### Assay:

Tare accurately a 125ml glass-stoppered Erlenmeyer flask containing 35.0 ml of 1 N sulfuric acid. Partially fill a 10ml graduated pipet from near the bottom of a sample, previously cooled in the original sample bottle to 10°C or lower. (Do not use a vacuum for drawing up the sample.) Wipe off any liquid adhering to the outside of the pipet, and discard the first ml. Hold the pipet just above the surface of the acid, and transfer 2ml into the flask, mix, and weigh again to obtain the weight of the sample. Add methyl red TS, and titrate the excess acid with 1N sodium hydroxide. Each ml of 1N sulfuric acid is equivalent to 17.03mg of NH<sub>3</sub>.

### Methyl Red TS:

Dissolve 100mg of methyl red in 100ml of alcohol, and filter if necessary.

### Arsenic:

Evaporate 11ml (10g sample) to about 2ml on a steam bath, dilute to 50ml with water, and mix. A 5 ml portion of this solution meets the requirements of the Arsenic Test, page 47.

[For Arsenic Test, see following pages.]

### Heavy Metal:

Transfer 22ml (20g sample) to a beaker, add about 5mg of sodium chloride, evaporate to dryness on a steam bath, and dissolve the residue in 2ml of diluted acetic acid TS and sufficient water to make 50ml. A 10ml portion of this solution, diluted to 25ml with water, meets the requirements of the Heavy Metals Test, page 512, using 20µg of lead ion (Pb) in the control (Solution A).

### Acetic Acid TS, Diluted:

A solution containing about 6% (w/v) of CH<sub>3</sub>COOH. Prepare by diluting 60.0ml of glacial acetic acid, or 166.6ml of 36% acetic acid (6N), with sufficient water to make 1000ml.

### Nonvolatile Residue:

Evaporate 11ml (10g sample) in a tared platinum or porcelain dish to dryness, dry at 105°C for 1 h, cool, and weigh.

### Readily Oxidizable Substances:

Dilute 4ml with 6 ml of water, and add a slight excess of diluted sulfuric acid TS and 0.1ml of 0.1N potassium permanganate. The pink color does not completely disappear within 10min.

### Sulfuric Acid TS, Diluted:

A solution containing 10% (w/v) of H<sub>2</sub>SO<sub>4</sub>. Prepare by cautiously adding 57ml of sulfuric acid (95% to 98%) or sulfuric acid TS to about 100 ml of water, then cool to room temperature, and dilute with water to 1000ml.

### Packaging and Storage:

Store in tight containers, preferably at a temperature not exceeding 25°C.

### Functional Use In Food:

Alkali

## Silver Diethyldithiocarbamate Colorimetric Method

Note: All reagents used in this test should be very low in arsenic content.

The general apparatus shown in Fig. 2 is to be used unless otherwise specified in an individual monograph. It consists of a 125ml arsine generator flask (a) fitted with a scrubber unit (e) and an absorber tube (e), with a 24/40 standard-taper joint (b) and a ball-and-socket joint (d), secured with a No. 12 clamp, connecting the units. The tubing between d and e and between d and c is a capillary diameter of 8mm. Alternatively, an apparatus embodying the principal of the general assembly described and illustrated may be used.

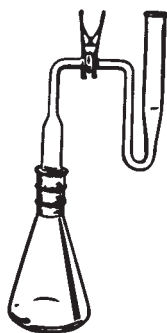


FIGURE 2 General Apparatus for Arsenic Test (Courtesy of the Fisher Scientific Co., Pittsburgh, Pa.)

Weigh accurately 132.0mg of arsenic trioxide that has been previously dried at 105° for 1h, and dissolve it in 5ml of sodium hydroxide solution (1 in 5). Neutralize the solution with diluted sulfuric acid TS, add 10ml in excess and dilute to 1000.0ml with recently boiled water. Transfer 10.0ml of this solution into a 1000-ml volumetric flask, add 10ml of diluted sulfuric acid TS, dilute to volume with recently boiled water, and mix. Use this final solution which contains 1 µg of arsenic (As) in each ml, within 3 days.

Dissolve 1g of recrystallized Silver Diethyldithiocarbamate,  $(C_2H_5)_2NCSSAg$ , in 200ml of reagent-grade pyridine. Store this solution in a light-resistant container and use within 1 month.

Silver Diethyldithiocarbamate is available commercially or may be prepared as follows: Dissolve 1.7g of reagent grade silver nitrate in

100ml of water. In a separate container, dissolve 2.3 g of sodium diethyldithiocarbamate  $(C_2H_5)_2NCSSNa \cdot 3H_2O$ , in 100ml of water and filter. Cool both solutions to about 15°, mix the two solutions, while stirring, collect the yellow precipitate in a medium-porosity sintered glass crucible or funnel, and wash with about 200ml of cold water.

Recrystallize the reagent, whether prepared as directed above or obtained commercially, as follows. Dissolve in freshly distilled pyridine, using about 100ml of solvent for each g of reagent, and filter. Add an equal volume of cold water to the pyridine solution, while stirring. Filter off the precipitate, using suction, wash with cold water, and dry in vacuum at room temperature for 2 to 3 h. The dry salt is pure yellow in color and should show no change in character after one month when stored in a light-resistant container. Discard any material that changes in color or develops a strong odor.

Dissolve 40 g of reagent-grade stannous chloride dihydrate,  $SnCl_2 \cdot 2H_2O$ , in 100ml of hydrochloric acid. Store the solution in glass containers and use within 3 months.

Soak cotton in a saturated solution of reagent-grade lead acetate, squeeze out the excess solution, and dry in a vacuum at room temperature.

The solution obtained by treating the sample as directed in an individual monograph is used directly as the Sample Solution in the Procedure. Sample solutions of organic compounds are prepared in the generator flask (a), unless otherwise directed. According to the following general procedure:

Caution: Some substances may react unexpectedly with explosive violence when digested with hydrogen peroxide.

Appropriate safety precautions must be employed at all times.

NOTE: If halogen-containing compounds are present, use a lower temperature while heating the sample with sulfuric acid, do not boil the mixture, and add the peroxide with caution, before charring begins, to prevent loss of trivalent arsenic.

Transfer 1.0g of the sample into the generator flask, add 5ml of sulfuric acid and a few glass beads, and digest at a temperature not exceeding 120° until charring begins, using preferably a hot plate in a fume hood. (Additional sulfuric acid may be necessary to completely wet some samples, but the total volume added should not exceed about 10ml.) After the sample has been initially decomposed by the acid, add with caution, dropwise, 30% hydrogen peroxide, allowing the reaction to subside and reheating between drops. The first few drops must be added very slowly with sufficient mixing to prevent a rapid reaction, and heating should be discontinued if foaming becomes excessive. Swirl the solution in the flask to prevent unreacted substance from caking on the walls, or bottom of the flask during digestion. Maintain oxidizing conditions at all times during the digestion by adding small quantities of the peroxide whenever the mixture turns brown or darkens. Continue the digestion until the organic matter is destroyed, gradually raising the temperature of the hot plate to 250°-300° until fumes of sulfur trioxide are copiously evolved and the solution becomes colorless or retains only a slight straw color. Cool, add cautiously 10ml of water, again evaporate to strong fuming, and cool. Add cautiously 10ml of water mix, wash the sides of the flask with a few ml of water, and dilute to 35ml.

## Procedure

If the Sample Solution was not prepared in the generator flask, transfer to the flask a volume of the solution, prepared as directed, equivalent to 1.0g of the substance being tested, and water to make 35ml. Add 20ml of dilute sulfuric acid (1 in 5), 2 ml of potassium iodide TS, and 0.5 ml of Stannous Chloride Solution, and mix. Allow the mixture to stand for 30 min at room temperature. Pack the Impregnated Cotton, leaving a small air space between the two plugs, lubricate joints b and d with stopcock grease, if necessary, and connect the 3.0ml of Silver Diethyldithiocarbamate Solution to the absorber tube, add 3.0g of granular zinc (20-mesh) to the mixture in the flask, and immediately insert the standard-taper joint in the flask. Allow the evolution of hydrogen and color development to

proceed at room temperature (25° ±3°) for 45 minutes, swirling the flask gently at 10min intervals. (The addition of a small amount of isopropanol to the generator flask may improve the uniformity of the rate of gas evolution.) Disconnect the absorber tube from the generator and scrubber units and transfer the Silver Diethyldithiocarbamate Solution to a 1cm absorption cell. Determine the absorbance at the wavelength of maximum absorption between 535nm and 540nm, with a suitable spectromotometer or colorimeter, using Silver Diethyldithiocarbamate Solution as the blank. The absorbance due to any red color from the solution of the sample does not exceed that produced by 3.0ml of Standard Arsenic Solution (3 µg As) when treated in the same manner and under the same conditions as the sample. The room temperature during the generation of arsine from the standard should be held to within ±2° of that observed during the determination of the sample.

## Interferences

Metals, or salts of metals such as chromium, cobalt, copper, mercury, molybdenum, nickel, palladium and silver are said to interfere with the evolution of arsine. Antimony, which forms stibine, is the only metal likely to produce a positive interference in the color development with the silver diethyldithiocarbamate. Stibine forms a red color that has a maximum absorbance at 510nm, but at 535 to 540 the absorbance of the antimony complex is so diminished that the results of the determination would not be altered significantly.

## Heavy Metal Test

This test is designed to limit the content of common metallic impurities that are colored by sulfide ion (Ag, As, Bi, Cd, Cu, Hg, Pb, Sb, Sn) under the specified test conditions. It demonstrates that the test substance is not grossly contaminated by such heavy metals and, within the precision of the test, that it does not exceed the Heavy Metals limit given in the individual monograph, as determined by concomitant visual comparison with a control solution. It has been found that, in the specified pH range, the optimum concentration of lead ion (Pb) for matching purposes by this method is 20 µg in 50 ml of solution.

### Special Reagents

**Ammonia TS** - dilute 400ml of ACS reagent-grade ammonium hydroxide to 1000ml with water.

**Hydrochloric Acid** - Sulfuric Acid, Nitric Acid, 30% Hydrogen Peroxide Use ACS reagent-grade chemicals.

**Lead Nitrate Stock Solution** - Dissolve 159.8mg of ACS reagent-grade lead nitrate,  $\text{Pb}(\text{NO}_3)_2$ , in 100ml of water containing 1 ml of nitric acid, dilute with water to 1000.0 ml, and mix. Prepare and store this solution in glass containers that are free from lead salts.

**Standard Lead Solution** - On the day of use dilute 10.0ml of Lead Nitrate Stock solution with water to 100.0 ml. Each ml of Standard Lead Solution contains the equivalent of 10µg of lead ion (Pb).

### Procedure

NOTE: In the following procedures, failure to adjust accurately the pH of the solution within the specified limits may result in a significant loss of test sensitivity.

**Solution A** - Pipet 2.0ml of Standard Lead Solution (20µg of Pb) into a 50ml color-comparison tube, and add water to make 25ml. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS or ammonia TS, dilute with water to 40ml, and mix.

**Solution B** - Place 25ml of the solution prepared as directed in the individual monograph in a 50ml color-comparison tube that matches the one used for Solution A, adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS or ammonia TS, dilute with water to 40ml, and mix.

**Solution C** - Into a third color comparison tube that matches those used for Solutions A and B, place 25ml of the solution prepared as directed in the individual monograph, and add 2.0ml of Standard Lead Solution. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by addition of diluted acetic acid TS or ammonia TS, dilute with water to 40ml, and mix.

To each tube add 10ml of freshly prepared hydrogen sulfide TS, mix, allow to stand for 5min, and view downward over a white surface. The color of Solution B is not darker than that of Solution A, and the intensity of the color of Solution C is equal to or greater than that of Solution A. If the color of Solution C is lighter than that of Solution A, the test substance is providing an interference with the test procedure and Method II must be used for the substance under examination.



# Appendix

## Federal DOT Regulations



## 49 CFR 172.101 – Hazardous Materials Table

Symbols	Hazardous materials description and proper shipping name	Hazard class or Division	Identification Numbers	Packing group	Label(s) required (if not expected)
(1)	(2)	(3)	(4)	(5)	(6)
	Ammonia solutions, relative density between 0.880 and 0.957 at 15 degrees C in water, with more than 10 percent but not more than 35 percent ammonia	8	UN2672	III	CORROSIVE

Special provisions (§172.102)	(8) Packaging authorizations (§173.***)			(9) Quantity limitations		(10) Vessel stowage requirements	
	Exceptions	Non-bulk packaging	Bulk packaging	Passenger aircraft or railcar	Cargo aircraft only	Vessel stowage	Other stowage provisions (§176.84)
(7)	(8A)	(8B)	(8C)	(9A)	(9B)	(10A)	(10B)
T14	154	203	241	5 L	60 L	A	40, 85

(2) The proper shipping name (PSN) is required on shipping papers. See also 172.203 for additional description requirements.

(3) The hazard Class or Division is required on shipping papers and determines labeling and placarding. See also 172.504 for general placarding requirements. Hazard class 8 indicates a corrosive material

(4) The Id number is required on shipping and packages, and usually appears on placecards. See also 172.302 et seq. for general information on marking requirements.

(5) The Packing group indicates the degree of danger of a hazardous material and determines packaging, per hazard class and division.

(6) This indicates minimum labeling requirements.

(7) Special provisions include packaging limitations, restrictions, and added requirements. T14 indicates that special provisions are applicable only to intermodal tanks.

(8A) 173.154 contains detailed packaging exceptions. Containers of not more than 4.0L of aqua ammonia are excepted from labeling requirements if not being offered for air transport.

(8B) (8C) A bulk packaging is defined as one having a maximum capacity greater than 450L for aqua ammonia.

(9A) Transportation on passenger aircraft or passenger railcar is limited to 5L net per package.

(9B) Transportation on cargo-only aircraft is limited to 60L net.

(10) For information on water transport of aqua ammonia, see 172.101(k), 176.63, and 176.84.



## 49 CFR 173.203 Non-Bulk Packagings For Liquid Hazardous Materials In Packaging Group III

(a) When 172.101 of this subchapter specifies that a liquid hazardous material be packaged under this section, only non-bulk packagings prescribed in this section may be used for its transportation. Each packaging must conform to the general packaging requirements of subpart B of part 173, to the requirements of part 178 of this subchapter at the Packing Group I, II, or III performance level, and to the requirements of the special provisions of Column 7 of the 172.101 Table.

(b) The following combination packagings are authorized:

Outer packagings:

Steel drum: 1A1 or 1A2

Aluminum drum: 1B1 or 1B2

Metal drum other than steel or aluminum: 1N1 or 1N2

Plywood drum: 1D

Fiber drum: 1G

Plastic drum: 1H1 or 1H2

Wooden barrel: 2C2

Steel jerrican: 3A1 or 3A2

Plastic jerrican: 3H1 or 3H2

Steel box: 4A

Aluminum box: 4B

Natural wood box: 4C1 or 4C2

Plywood box: 4D

Reconstituted wood box: 4F

Fiberboard box: 4G

Expanded plastic box: 4H1

Solid plastic box: 4H2

Inner packagings:

Glass or earthenware receptacles

Plastic receptacles

Metal receptacles

Glass ampoules

(c) The following single packagings are authorized:

Steel drum: 1A1 or 1A2

Aluminum drum: 1B1 or 1B2

Metal drum other than steel or aluminum: 1N1

Plastic drum: 1H1 or 1H2

Fiber drum: 1G (with liner)

Wooden barrel: 2C1

Steel jerrican: 3A1 or 3A2

Plastic jerrican: 3H1 or 3H2

Plastic receptacle in steel, aluminum, fiber or plastic drum: 6HA1, 6HB1, 6HG1, 6HH1

Plastic receptacle in steel, aluminum, wooden, plywood or fiberboard box: 6HA2, 6HB2, 6HC, 6HD2 or 6HG2

Glass, porcelain or stoneware in steel, aluminum or fiber drum: 6PA1, 6PB1 or 6PG1

Glass, porcelain or stoneware in steel, aluminum, wooden or fiberboard box: 6PA2, 6PB2, 6PC or 6PG2

Glass, porcelain or stoneware in solid or expanded plastic packaging: 6PH1 or 6PH2

Plastic receptacle in plywood drum: 6HD1

Glass, porcelain or stoneware in plywood drum or wickerwork hamper: 6PD1 or 6PD2

Cylinders, prescribed for any compressed gas, except for Specifications 8 and 3HT

[Source Note: At 56 FR 66271, December 20, 1991, revised paragraph (c) by adding entry of "Fiber drum: 1G (with liner)"; and at 59 FR 67518, December 29, 1994, removed the wording 4A1 of 4A2, 4B1 or 4B2 and 6HH and replaced with 4A, 4B, and 6HH1 in paragraphs (b) and (c).]

## 49 CFR 173.241 Bulk Packaging For Certain Low Level Hazard Liquid And Solid Materials

When 172.101 of this subchapter specifies that a hazardous material be packaged under this section, only the following bulk packagings are authorized, subject to the requirements of Subparts A and B of Part 173 of this subchapter and the special provisions specified in Column 7 of the 172.101 Table.

**Rail cars:** DOT Class 103, 104, 105, 109, 111, 112, 114, or 115 tank car tanks; Class 106 or 110 multi-unit tank cars and AAR Class 203W, 206W and 211W tank car tanks,

**Cargo Tanks:** DOT specification MC 300, MC 301, MC 302, MC 303, MC 304, MC 305, MC 306, MC 307, MC 310, MC 311, MC 312, MC 330, MC 331, DOT 406, DOT 407, and DOT 412 cargo tank motor vehicles suitable for transport of liquids.

**Portable Tanks:** DOT 51, 52, 56, 57 and 60 portable tanks; IMO type 1, 2 and 5, and IM 101 and IM 102 portable tanks; marine portable tanks conforming to 46 CFR part 64I and non-DOT specification portable tanks suitable for transport of liquids.

### Intermediate bulk containers:

**I.** Intermediate bulk containers are authorized subject to the conditions and limitations of this paragraph and paragraph (2) of this section provided they conform to the requirements in Subpart O of part 178 of this subchapter at the Packaging Group performance level specified in column 5 of the 172.101 Table of this subchapter for the material being transported.

**(A)** The following are authorized for liquids of solids:

**(a)** Composite: 31HZ1 or 31HZE. For each container, the letter “Z” must be replaced with a capital letter which indicates the material of construction of the outer packaging. For example, 31HA1 is a composite intermediate bulk container with a metal outer packaging (see 178.702 of this subchapter);

**(b)** Metal: 31A, 31B, or 31N; or

**(c)** Rigid plastic: 31H1 or 31H2

**(B)** The following are authorized for solids only:

**(a)** Composite: 11HZ1, 11HZ2, 21HZ1, or 21HZ2. For each composite intermediate bulk container, the letter “Z” must be replaced with a capital letter which indicates the material of construction of the outer packaging. For example, 21HA1 is a composite intermediate bulk container with a metal out packaging (see 178.702 of this subchapter);

**(b)** Fiberboard: 11G;

**(c)** Flexible: 13H1, 13H2, 13H3, 13H4, 13H5, 13L1, 13L2, 13L3, 13L4, or 13M2;

**(d)** Metal: 11A, 11B, 11N, 21A, 21B, or 21N;

**(e)** Rigid plastic: 11H1, 11H2, 21H1, or 21H2; or

**(f)** Wooden: 11C, 11D, 11F.

**II.** The following conditions and limitations apply to the use of the intermediate bulk containers.

**(a)** Flexible, fiberboard and wooden intermediate bulk containers are intended for the transport of solids only and may not be used for liquids or materials that may become liquid during transportation.

**(b)** Only liquids with a vapor pressure less than or equal to 100 kPa (16 psig) at 50° (122°F), or 130 kPa (18.9 psig) at 55°C (131°F), are authorized in metal intermediate bulk containers; or

**(c)** Flexible, fiberboard, or wooden intermediate bulk containers containing materials in Packing Group II must be packed in a closed freight container or a closed transport vehicle.

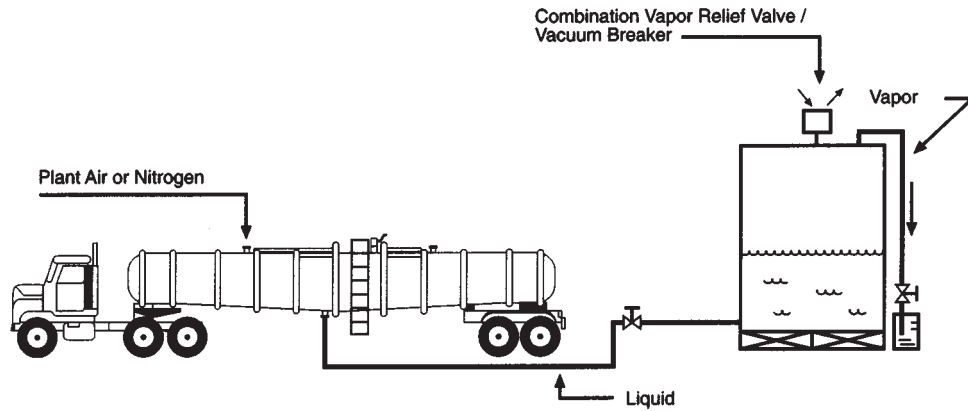
[Source Note: At 56 FR 66275, December 20, 1991, revised (c); and at 59 FR 38067, July 26, 1994, added (d).]

# Appendix

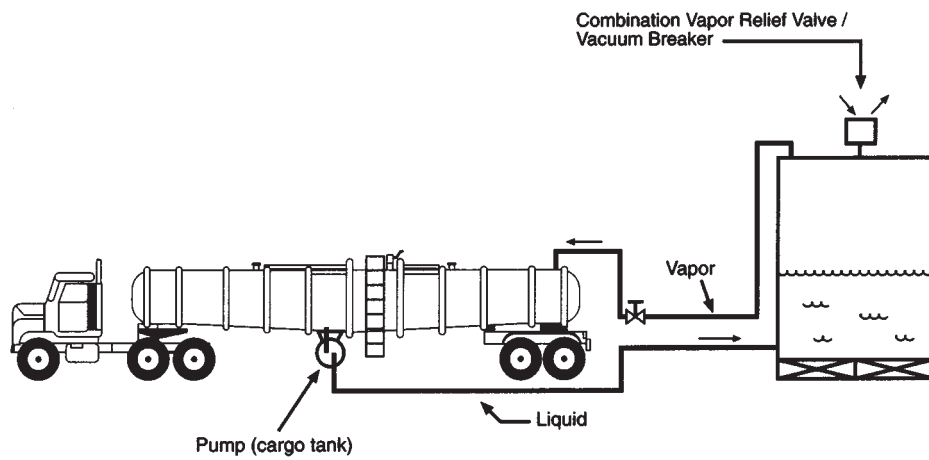
## Delivery & Storage Information



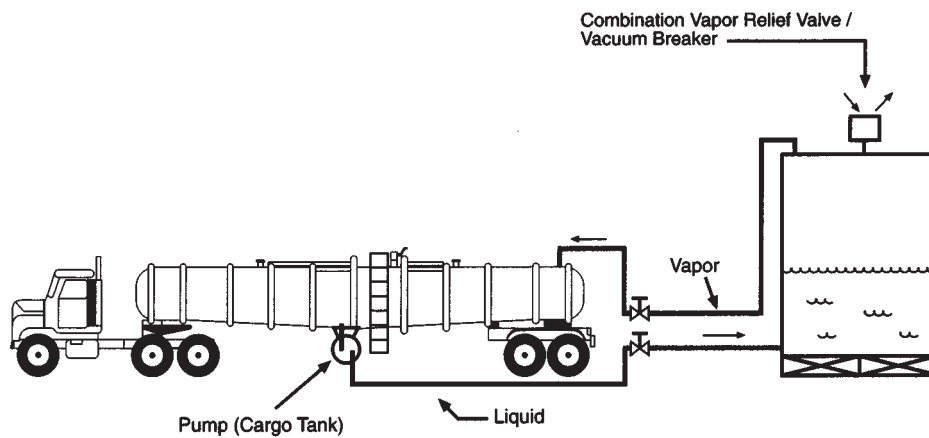
## Truck Delivery Methods



**Pressure Method**

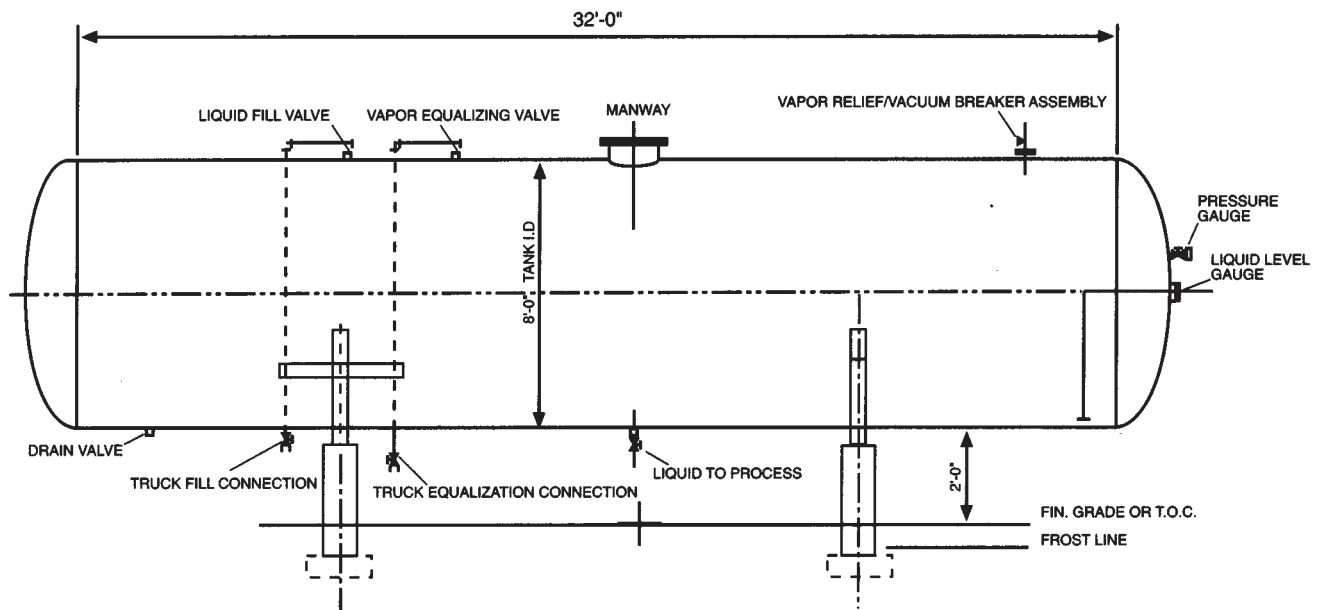


**Pump Method "A"**



**Pump Method "B"**

## TYPICAL HORIZONTAL AQUA AMMONIA STORAGE TANK



The 12,000 gallon aqua ammonia storage tank shown above can store approximately 85,000 lbs. of 26° Bé (29.4%) aqua ammonia. Although vented atmospheric pressure tanks are used by some aqua consumers, Airgas Inc. recommends the use of a minimum 25 PSIG design working pressure storage vessel built in accordance with the ASME Code for Unfired Pressure Vessels (Section VIII). The vessel can be fabricated from carbon steel, aluminum, or stainless steel.

A typical arrangement of tank openings which is suitable for most storage system requirements is shown in the drawing. See reverse side for additional information. It is possible, however, to order tanks with size dimensions and schedules of openings to meet any specific user requirement. For further information about storage systems for aqua ammonia, contact Airgas Inc.

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**Page 1 of 2**  
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 Duluth, GA 30097  
 (770) 717-2200

## Functions of Storage Tanks Components

### Liquid Fill Line

The liquid fill line allows the transfer of product from the delivering cargo tank. It consists of a quick disconnect fitting and shutoff valve at the truck liquid fill connection point, fill line piping, and shutoff valve located on the top of the tank.

### Equalizing Line

The vapor equalizing line allows equalization of pressure to occur between the vapor space of the storage tanks and the vapor space of the delivering cargo tank. It consists of a quick disconnect fitting and shutoff valve at the truck vapor equalizing connection point, equalizing line piping, and a shutoff valve located on top of the tank.

### Vapor Pressure Relief/Vacuum Breaker Valve

A combination pressure relief valve and vacuum breaker protects the storage vessel from pressure in excess of the design working pressure and from sub-atmospheric pressures.

### Liquid Level Float Gauge

The dial on this float gauge is calibrated in percentage of total tank volume to indicate the volume of aqua ammonia in the storage tank. A metal bulb floating on the surface of the aqua ammonia actuates an indicator pointer on the dial.

### Pressure Gauge

The pressure gauge indicates the internal pressure in the storage tank. A pressure gauge calibrated from 30" vacuum to about 50 PSIG can be used if an indication of sub-atmospheric pressure is desired.

### Liquid To Process Valve

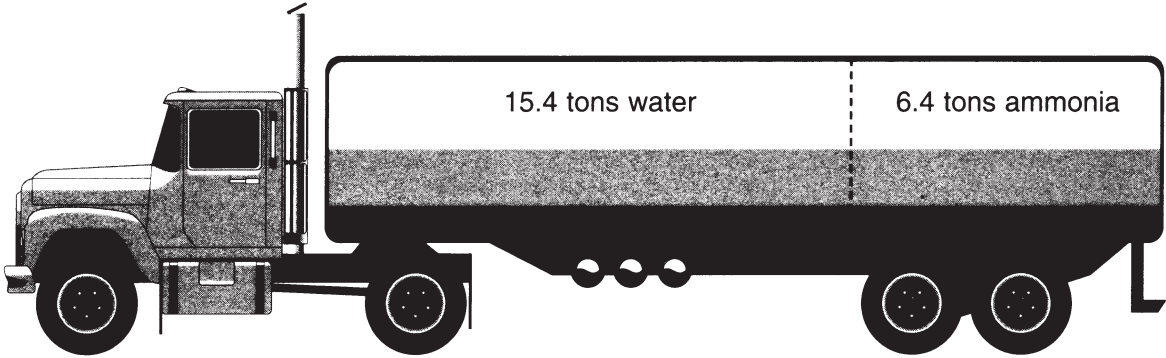
Piping from this valve is connected to the consumer's process.

### Drain Valve

This valve is used to remove product from the storage tank of the performance of some maintenance procedures or for the removal of the tank from service.

**AQUA AMMONIA TERMINOLOGY**

Confused by aqua ammonia terminology and billing procedures?  
Contained vs. solution basis? Delivered vs. FOB?  
Wet vs. dry? Pounds vs. tons vs. gallons?



This trailer holds 6.4 tons of “contained” ammonia or “dry” ammonia product. The total weight of the load is 21.8 tons. This is called the “solution” weight or “wet” weight.

Airgas Inc. bills aqua ammonia on a contained ammonia basis. Freight costs are billed on a solution basis. Suppose that your contained ammonia product cost is \$420.00 per ton and that your freight rate is \$15.00 per ton. The invoice for this truck load would be calculated as follows:

Ammonia	6.4 tons X \$420/ton = \$2,688.00
Freight	21.8 tones x 15/ton = \$327.00
	Total: \$3,015.00

Another method for billing uses a solution or “wet” basis. The solution weight for this trailer is 21.8 tons. For the cost conditions given above, the first line of the invoice would read 21.8 tons x \$123.30/ton = \$2,688.00. Both invoices would show the same total dollar amount. The solution or “wet” price per ton is much less, but remember that you are being charged for the ammonia and the water.

Delivered pricing on a contained ammonia basis is a little more complicated. In the example above, the total freight cost was \$327.00. To establish a freight rate based on contained ammonia, it is necessary to divide the freight cost (\$327.00) by 6.4 tons. This calculates to \$51.10 freight per ton of contained ammonia. Therefore, the delivered price is \$471.00 per ton (contained ammonia basis).

At a concentration of 29.4%, aqua ammonia weighs 7.5 lbs/gallon. A trailer containing 21.8 tons of aqua ammonia has a product volume equal to 21.8 tons x 2,000 lbs/ton x 1 gallon/7.5 lbs or 5,813 gallons. Degrees Baumé is a measurement system for specific gravity (density). Aqua ammonia with a specific gravity (density) of 26 ° Baumé has an ammonia weight concentration of 29.4%

**TB 5-3**  
**3/96**

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