

*Guidelines for:*

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# Water Contamination in Ammonia Refrigeration Systems

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International Institute of  
Ammonia Refrigeration

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# WATER CONTAMINATION IN AMMONIA REFRIGERATION SYSTEMS

## I. PREFACE

Water contamination of the ammonia refrigerant is common in many refrigerating systems. The solubility characteristics of ammonia in water allows an aqueous ammonia solution to be formed which replaces the anhydrous ammonia refrigerant. In many systems this change occurs over a period of time and the effects go unnoticed. As a result the penalties experienced in coping with these unknown changing conditions become continuous and increasingly greater.

The effects of the water contamination of ammonia refrigerant are many. The pressure-temperature relationship is impaired. Chemical changes occur to the compressor oil Organic acids and sludges are formed. Pump operation, piping pressure drop and evaporator performance are adversely affected.

The purpose of this bulletin is two-fold. One deals with the changes that occur in the pressure-temperature relationship of the refrigerant. The other relates to means for determining the presence of water, its measurement, its removal, and sources of contamination.

## II. AMMONIA-WATER RELATIONSHIP

Refrigeration grade ammonia is charged into a system whenever refrigerant is needed. This ammonia complies with Federal Specification O-A-445B and has a minimum purity of 99.95 percent. The residue of .05 percent maximum, may contain a maximum of 500 ppm of water, and 5 ppm of oil. Such ammonia is called anhydrous — meaning free of water. As long as a water free environment continues to exist, the pressure-temperature relationship shown in the ammonia refrigerant tables will be valid.

Ammonia and water have a great affinity for each other. For example, at atmospheric pressure and a temperature of 86° F., a saturated solution of ammonia and water will contain approximately 30 percent ammonia by weight. As the temperature of the solution is lowered, the ability to absorb ammonia increases. At 32° F. the wt. percentage increases to 46.5 percent; at -28° F. the percentage increases to 100 percent ammonia by wt.

This affinity, if allowed to occur, will cause the ammonia to be diluted by water. As dilution is experienced a change occurs to the anhydrous ammonia — it starts to become an aqueous ammonia solution. The solubility of ammonia in water is shown in Curve 1.

## III. EFFECTS OF WATER DILUTION

The pressure-temperature relationship for aqueous ammonia solutions is different than that for anhydrous ammonia. At a given pressure the saturated temperature for anhydrous ammonia will be lower than the saturated temperature for an aqueous solution. As the aqueous solution becomes more dilute (water content is increased), the saturated temperature becomes higher.

Curve 2 shows the saturation temperatures of ammonia-water mixtures for four typical suction pressure levels. The pressure-temperature relationship for anhydrous ammonia is read up the vertical 0 percent water by weight axis: i.e., at 10 psig suction, saturated temperature is -28° F., at 29.4 psig suction, saturated temperature is 16° F.

Curves for other suction pressures may be interpolated by entering along the 0 percent water axis at the temperature desired. Then parallel adjacent curves.

The table below lists the saturated temperatures taken from Curve 2 for suction pressures of 8.9" vacuum, 0 psig and 29.4 with water dilution varying from 10 to 30 percent.

<u>% Dilution</u>	<u>Saturated 8.9" Vac.</u>	<u>Suction 0#</u>	<u>Temperature 29.4#</u>
0	-40.4°F	-28°F	16°F
10	-37.5	-25	19.4
20	-33.5	-20	25.7
30	-26.0	-12	36.2

Note that as the water percent increases the incremental increase in saturated suction temperature becomes greater. From the above it is obvious why water contaminated refrigeration systems must operate at lower suction pressure in order to maintain desired room temperatures or to handle process

type loads. With such a change in operating conditions, a two-fold penalty results. For each °F that saturated suction temperature is lowered, compressor capacity is reduced approximately 2½ percent for high stage and 3 percent for boosters. Likewise, as suction pressure is lowered there is an increase in BHP/ton. Simply stated, less compressor capacity as well as increased power consumption are experienced.

#### **IV. WATER CONTAMINATION SOURCES**

Water may gain entrance into a system in a number of ways:

1. It may remain in new vessels which are not properly drained or dried following completion of the ASME hydrostatic test.
2. During construction it may enter through open piping or weld joints which are only tacked in place when either are exposed to the elements.
3. From condensation which may occur in the piping during construction.
4. From condensation which may occur when air has been used as the medium for the final system pressure testing.
5. That which remains in the system as a result of inadequate evacuation procedures on startup or following opening for repair or maintenance.
6. As a result of complex chemical reactions in the system between the aqueous ammonia, oxygen, oils and sludges.
7. Lack of adequate or no purging.
8. Probably the most common source is that which occurs in systems which operate with suction pressure in the vacuum range. This is from leaks in valve stem packing, screwed and flanged piping joints, threaded and welded pipe connections, leaking safety relief valves, pump and booster compressor seals, and leaks in the coils of evaporator units.
9. From the use of improper procedures when draining oil into water filled containers, from vessels in which the pressure is in a vacuum range.
10. Occasionally, a major contamination will occur as the result of the rupture of a tube or tubes in a shell and tube heat exchanger, such as chillers or oil coolers. Incidents of this type are usually the only kind in which operating personnel are alerted to the occurrence because of the upset to normal operating conditions which follows.

#### **V. AREAS OF HIGHEST WATER CONTENT**

Unless steps are taken to control the amount of water infiltration, there will be a continuous increase in water content of the ammonia in the system over a period of time.

In recirculating systems the ammonia having the highest water content will be found in the vessel from which the liquid is supplied to the evaporators. In a pump system this would be the pump receiver. In a gas pressure system it would be the controlled pressure receiver.

In flooded systems the ammonia with the highest water content will be found in the evaporator and surge drum. In DX systems the highest water content will be found in the suction accumulator.

In two-stage systems the highest water concentration will be found in the respective vessels and evaporators serving the low stage portion of the system.

The reason for these concentration buildups in the low side is due to the large difference in vapor pressure between water and ammonia. For example, at 35° F., the vapor pressure of ammonia is 66.3 psia compared to 0.10 psia for water. Since the liquid with the higher vapor pressure will evaporate in greater proportion than the liquid with the lower vapor pressure, a residue is left containing more and more of the lower vapor pressure liquid if infiltration is not corrected.

In all of the above instances, as the water content increases, the performance of all evaporators will progressively deteriorate.

#### **VI. FREEZING TEMPERATURES OF AMMONIA-WATER SOLUTIONS**

Having identified the areas of highest water concentrations it is appropriate to look at the effect water dilution has upon the temperature at which the ammonia-water solutions freeze.

The freezing point of anhydrous ammonia of  $-107.9^{\circ}$  F. Strangely, as the water dilution of the ammonia occurs, the freezing point of the aqueous mixture initially becomes lower.

Curve 3 shows the Freezing Point of aqueous ammonia solutions. Note that when the amount of ammonia in the solution is 33.5 percent, its freezing point is  $-148.5^{\circ}$  F. Also note that any ammonia-water solution containing more than 28.5 percent ammonia will have a freezing point which is lower than  $-100^{\circ}$  F.

As the ammonia dilution continues, the freezing point rises rapidly until it reaches  $+32^{\circ}$  F when the solution is 100 percent water.

## VII. DETECTION OF WATER CONTAMINATION

Detection of the effects of water contamination of a system may take years before the problem is recognized. During this time room temperatures may have been compromised, more compressors operated, suction pressure lowered and much additional electrical energy consumed and wasted.

The presence of water in the ammonia can be determined by a method similar to that used for testing samples of ammonia shipped by manufacturers or large distributors.

### APPARATUS

The apparatus required for obtaining the sample and making the test consists of:

1. Ammonia evaporation test tube\*
2. Test tube tongs
3. Sampling converter assembly\*
4. Vented stopper
5. Water bath maintained at  $80^{\circ}$  to  $90^{\circ}$  F
6. Fume hood
7. Rubber gloves
8. Face shield

\*These items may be obtained from the Industrial Products Department, USS Agri-Chemicals Division, United States Steel Co. See Appendices A and B.

### SAMPLING LOCATIONS

Suggested locations from which to take samples are:

Pump Systems	- from pump discharge line
Gas Pressure Systems	- from transfer line between transfer drum and controlled pressure receiver
Flooded Systems	- from oil drain valve on liquid leg of surge drum
DX Systems	- from liquid transfer line from accumulator

### SAMPLING PROCEDURES

The sampling procedure should follow the operating instructions furnished for the USS Cold-Flo sampling converter. A copy is contained herein as Appendix C.

Care should be exercised while drawing the sample to prevent the entrance of air into the test tube, otherwise, moisture in this air will condense, frost and remain in the tube thus distorting the test findings.

When the 100 milliliter (ml) sample is drawn and the sampling converter valve is closed, remove the evaporation test tube from the sampler and, immediately, securely place the vented stopper in the test tube. Stopper must be vented to prevent buildup of pressure in test tube.

## TEST PROCEDURE

The test procedure is as follows:

1. Place evaporation tube in the water bath with fume hood.
2. Maintain temperature of the water from 80° to 90° F. Do not increase temperature of the water to speed up evaporation otherwise test results will be incorrect. Care should also be taken to prevent water in bath from entering the test tube.
3. After the ammonia has evaporated, remove test tube from water.
4. If the residue which remains is greater than the 0.5 ml which can be read in the bottom of the test tube, it should be transferred to a container graduated so that 0.5 ml can be read or estimated.
5. Record the volume of the residue to the nearest 0.5 ml. If oil is present on top of the residue, do not include that amount in the quantity of the residue.

## VIII. PERCENT WATER DETERMINATION

The residue remaining is a mixture of water, oil, non-volatile impurities and ammonia. If the temperature of the water bath was properly maintained, the water residue will contain approximately 30 percent ammonia (see Curve 1).

The procedures listed below have been modified from those used for more precise determinations of the presence of small quantities of water. Our analysis is not concerned with small percentages in the range of 0.2 percent or less, but with the determination of percentages ranging from 5 percent to 30 percent or more.

To determine percent by weight water contamination:

1. Record the operating pressure in the vessel from which the sample is taken. (Do not record the pressure in the line if the ammonia was pumped or transferred by gas pressure.)
2. Determine the Evaporation Factor (EF) from Curve 4.
3. Record the initial volume of sample in milliliters (ml).
4. Record the volume of residue in millimeters (ml).
5. Percent water by weight =  $\frac{\text{ml residue} \times 0.914 \times \text{EF}}{\text{ml sample}} \times 100^*$

EXAMPLE:

1. Operating pressure in vessel is 29.4 psig.
2. Evaporation Factor (EF) is 0.919.
3. Volume of initial sample is 100 ml.
4. Volume of residue is 10 ml.
5. Percent water by weight =  $\frac{10 \text{ ml} \times 0.914 \times 0.919}{100 \text{ ml}} \times 100$   
= 8.40%

Reference to Curve 2 shows that with a suction pressure of 29.4 psig the saturated evaporating temperature is 18.6° F with 8.40% of water in the ammonia as compared with 16.0° F for anhydrous ammonia.

\*Derivation of the formula is shown in Appendix D.

## IX. REMOVAL OF WATER FROM SYSTEM

The removal of water from a system is best accomplished by use of a batch method in which external heat is utilized. The heat source may be hot gas, warm or hot water. The most practical and cost effective source, without a question, is hot gas.

Because of the high cost of energy and the quantity of heat required, heat from other sources should be obtained as a result of some heat recovery process such as use of oil cooler, compressor jacket or condenser water. The use of vessels containing internal coils through which liquid ammonia is circulated is not practical nor suggested.

The need to remove water from ammonia refrigeration systems has not characteristically been considered an integral part of system design. As a result, systems typically lack the means to remove this water.

Several methods which are known to have been improvised to remove water are described.

One method which uses hot gas is shown in Diagram 1. This partially jacketed vessel was designed with stub connections oriented for connection into existing piping.

Another method uses an ammonia regenerator or oil still which was common in many plants some years ago. Diagram 2 shows this unit along with all piping connections and operating instructions. Warm or hot water are used as its heat source.

In the application of external heat the limitations of temperature stated in the test procedure do not apply for the regenerating process. It is essential that the regenerating vessel be designed for high pressure service and provided with the required safety relief valves.

In the few incidents where large amounts of water enter a system, emergency measures are needed for fast removal. In such cases, it is important that, where possible, the operation of certain evaporators be modified so they can be operated as regenerators. Such an instance would be the use of a flooded plate coil evaporator over which warm or hot water was circulated. It is essential that it be equipped with a relief valve.

In an extreme emergency, it may be necessary to drain those contaminated low side portions of the system.

## **X. SAFETY PRECAUTIONS**

Safe working conditions and prudent concern for detail are absolutely necessary to avoid injury.

In preparation for the taking of samples, locate a portable fan so that the working area will be well ventilated and free of ammonia fumes as the sample is being drawn. Personnel should protect themselves from direct contact with liquid ammonia when taking samples and performing the tests. Protective hats, glasses, gloves and clothing should be worn. A source of water should be nearby.

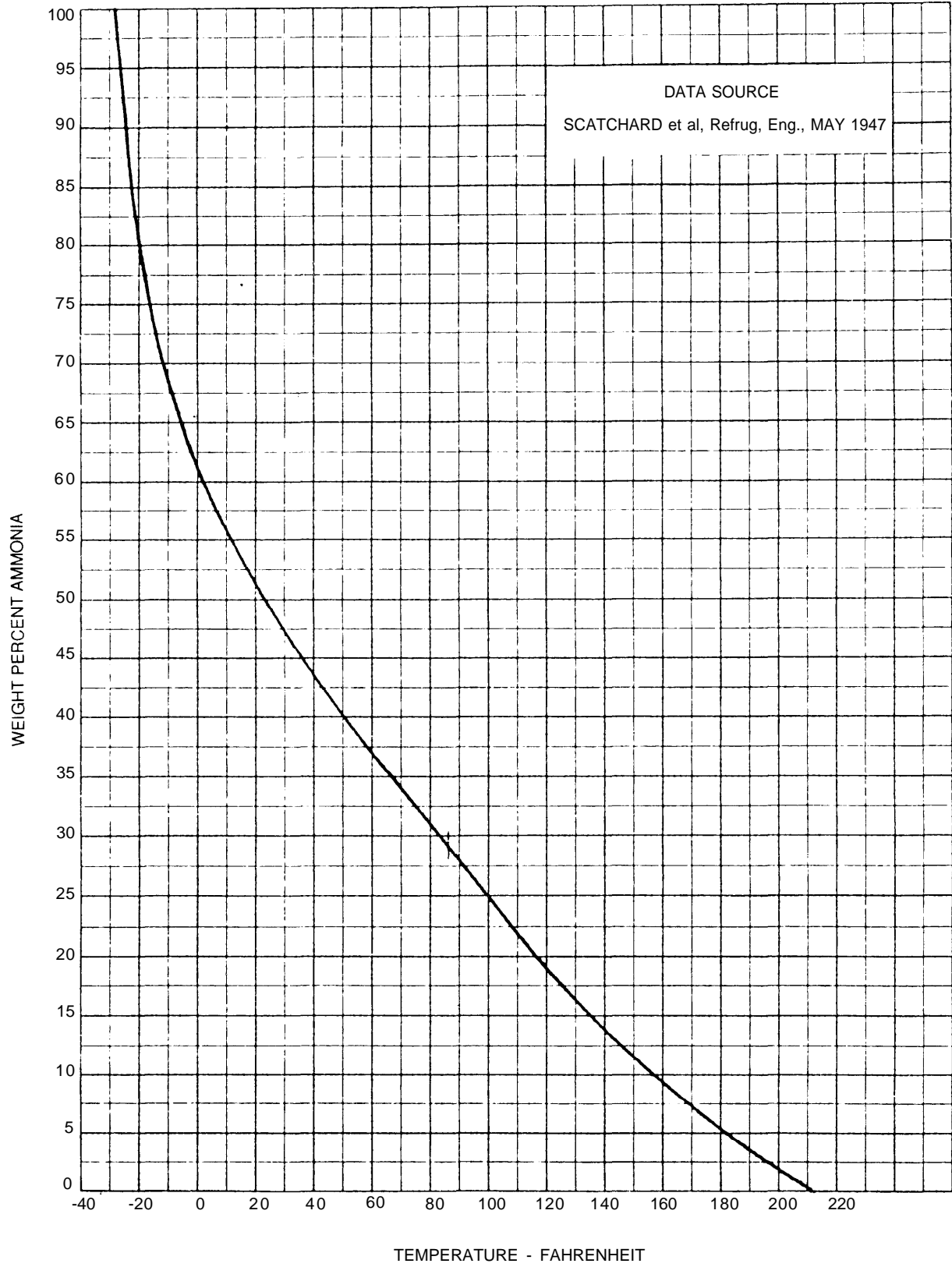
### **OTHER PRECAUTIONS**

In the event of a rapid influx of water into the ammonia contained in a heat exchanger, two abnormal conditions occur. The ammonia water solution becomes heated because of the reaction heat generated when the water and ammonia mix. When this happens thermal-hydraulic shock occurs. It is important to stop water flow in and out of the heat exchanger and to stop the ammonia flow in the suction line from the heat exchanger.

Since the operation of such improvised equipment involves use of some unusual procedures which can involve pressure buildup, we stress that it be done by knowledgeable operating personnel.

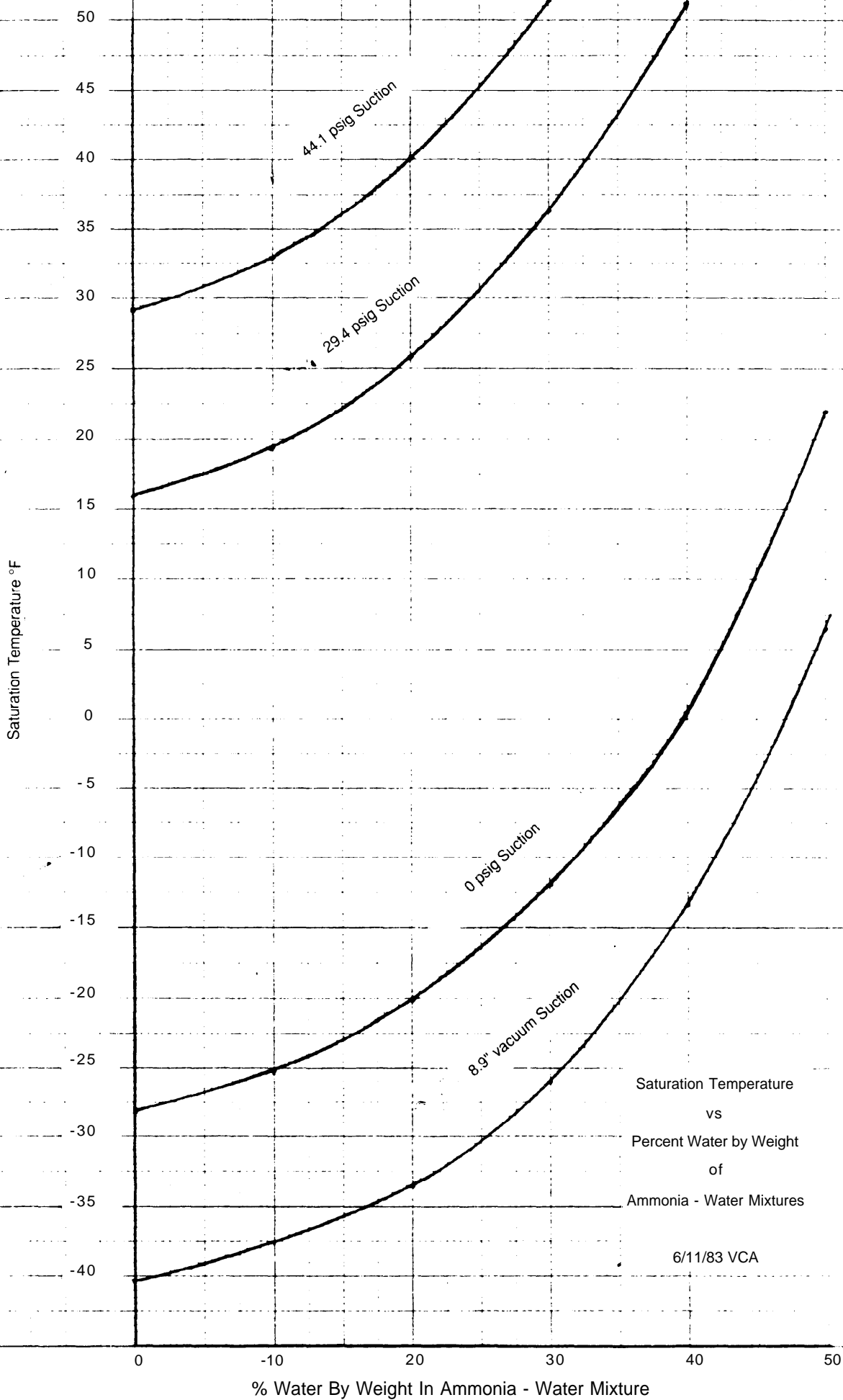


# CURVE 1



SOLUBILITY CURVE FOR AMMONIA IN WATER

# CURVE 2

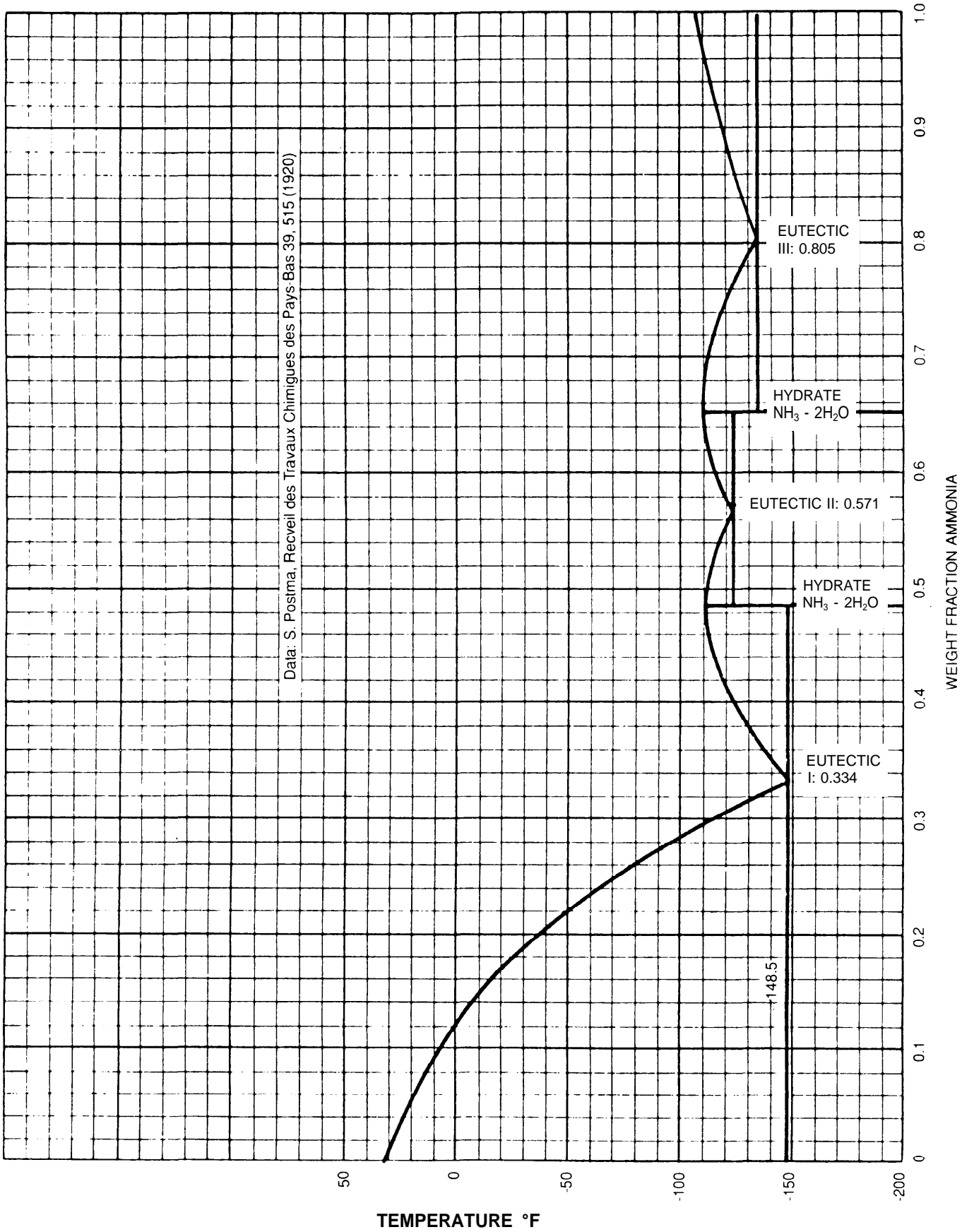


Saturation Temperature  
vs  
Percent Water by Weight  
of  
Ammonia - Water Mixtures

6/11/83 VCA

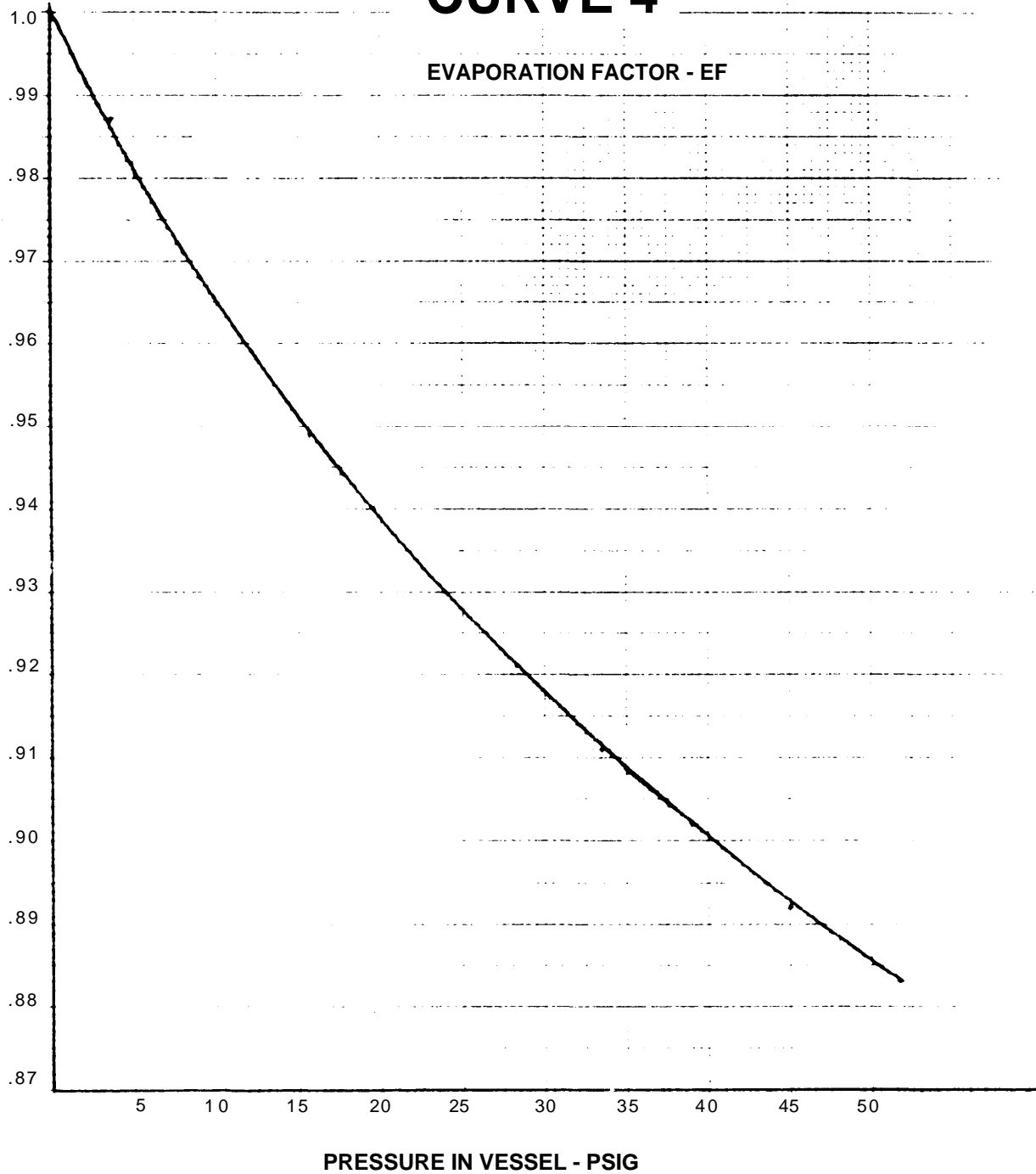
# CURVE 3

## FREEZING POINT OF AQUEOUS AMMONIA



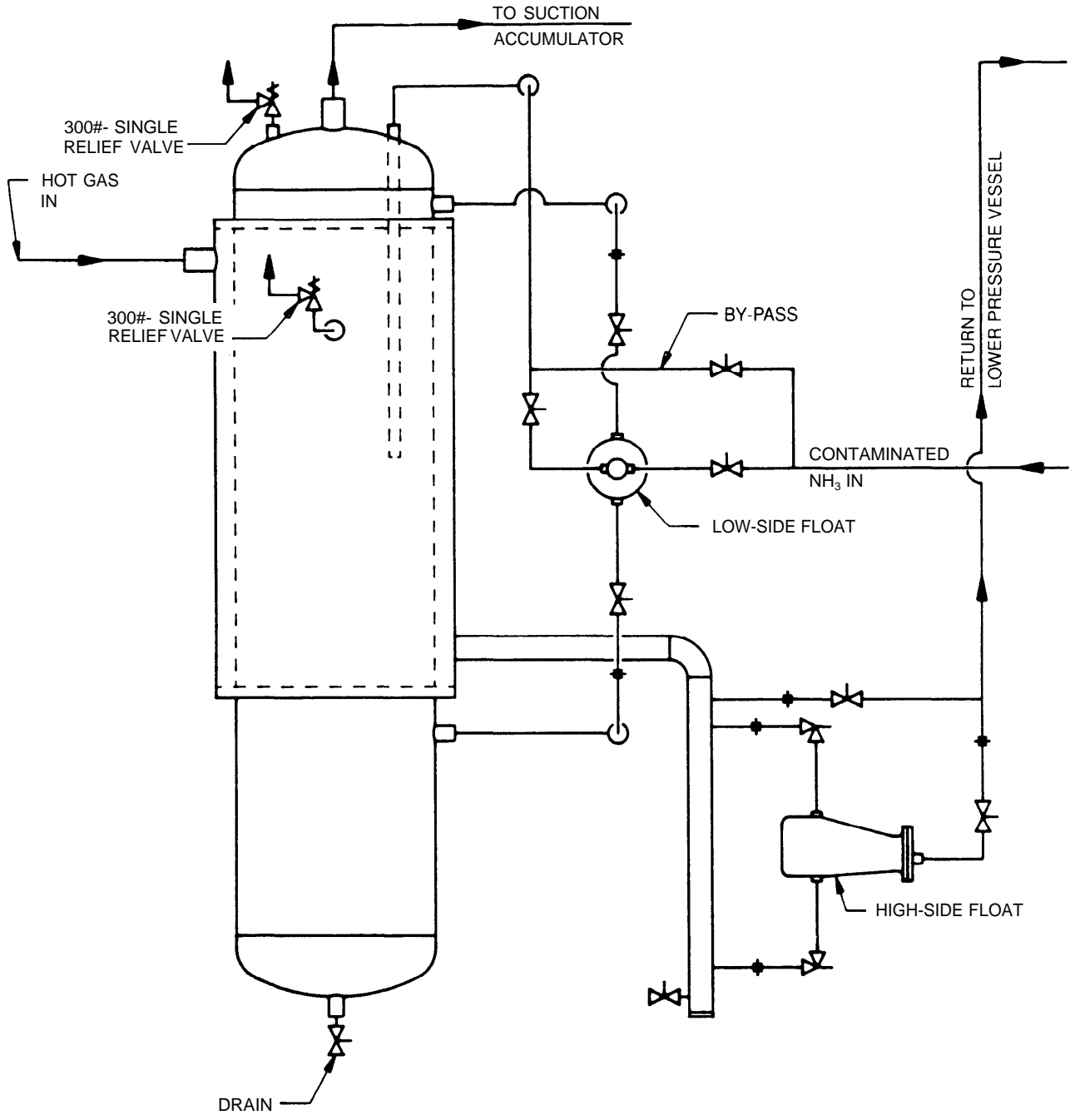
# CURVE 4

EVAPORATION FACTOR - EF

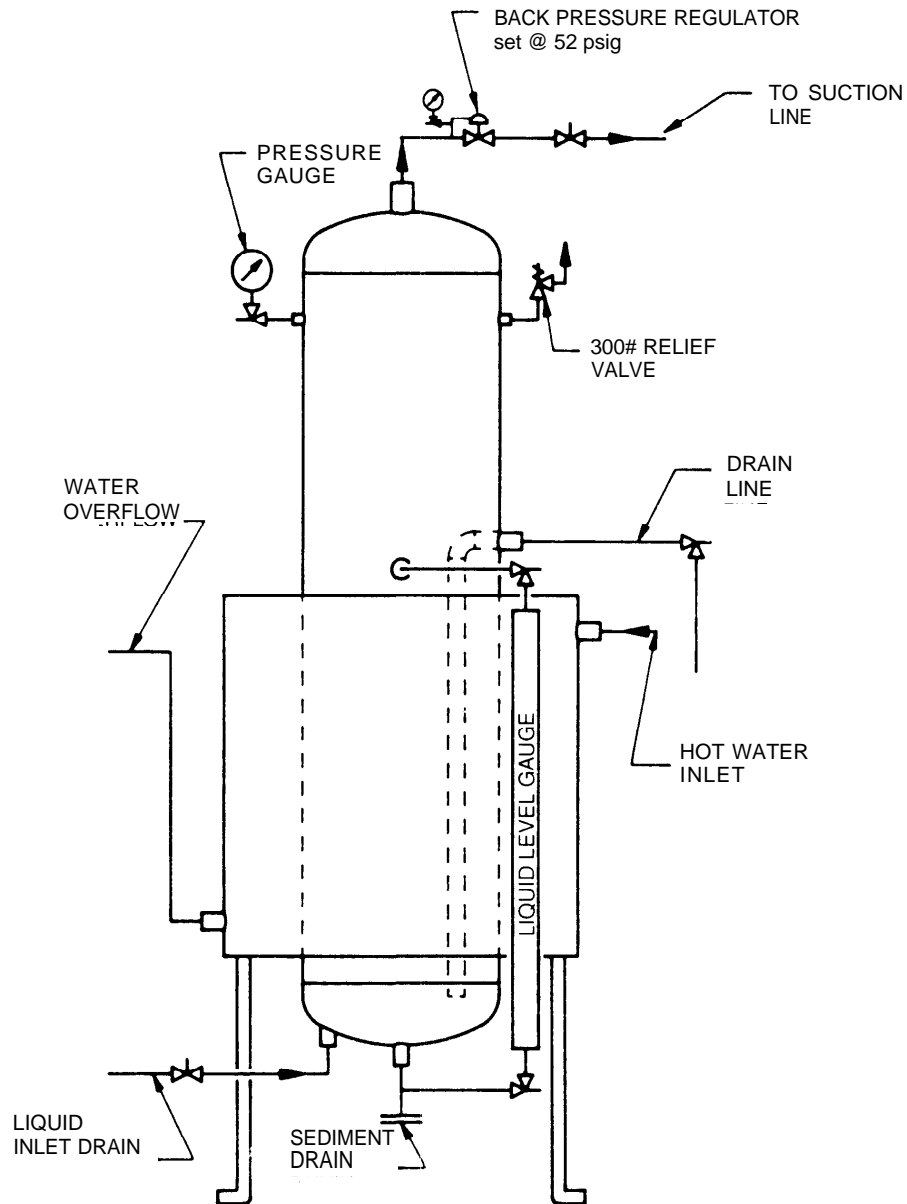


THE EVAPORATION FACTOR REPRESENTS THE PERCENT OF LIQUID AMMONIA REMAINING IN THE SAMPLE AFTER A PORTION IS LOST DUE TO FLASH AS THE SAMPLE IS BEING TAKEN.

# DIAGRAM 1



# DIAGRAM 2



**AMMONIA REGENERATOR**

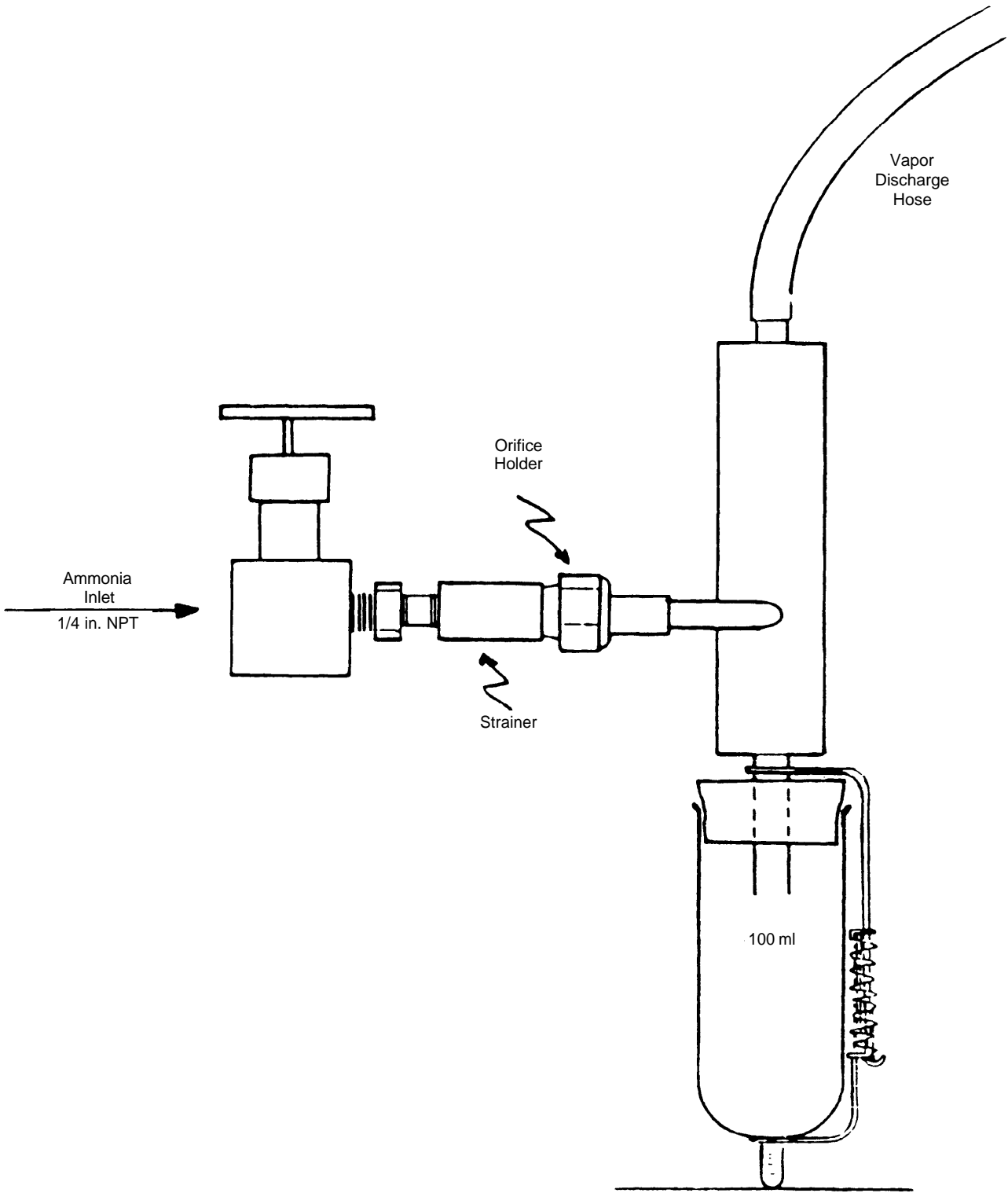
The ammonia regenerator shown schematically shall be designed in accordance with the ASME unfired pressure vessel code and ANSI/ASHARE 15 standard.

The regenerator consists of a vertical pipe or shell with welded dished heads, the lower end of the pipe being surrounded by a hot water jacket. Inlet and outlet connections for hot water shall be provided in the jacket as shown. Minimum valves and fittings shall include a liquid inlet valve, a suction valve, a drain valve, a safety relief valve, a pressure gauge with shut-off valve and a sediment drain or cleanout plug.

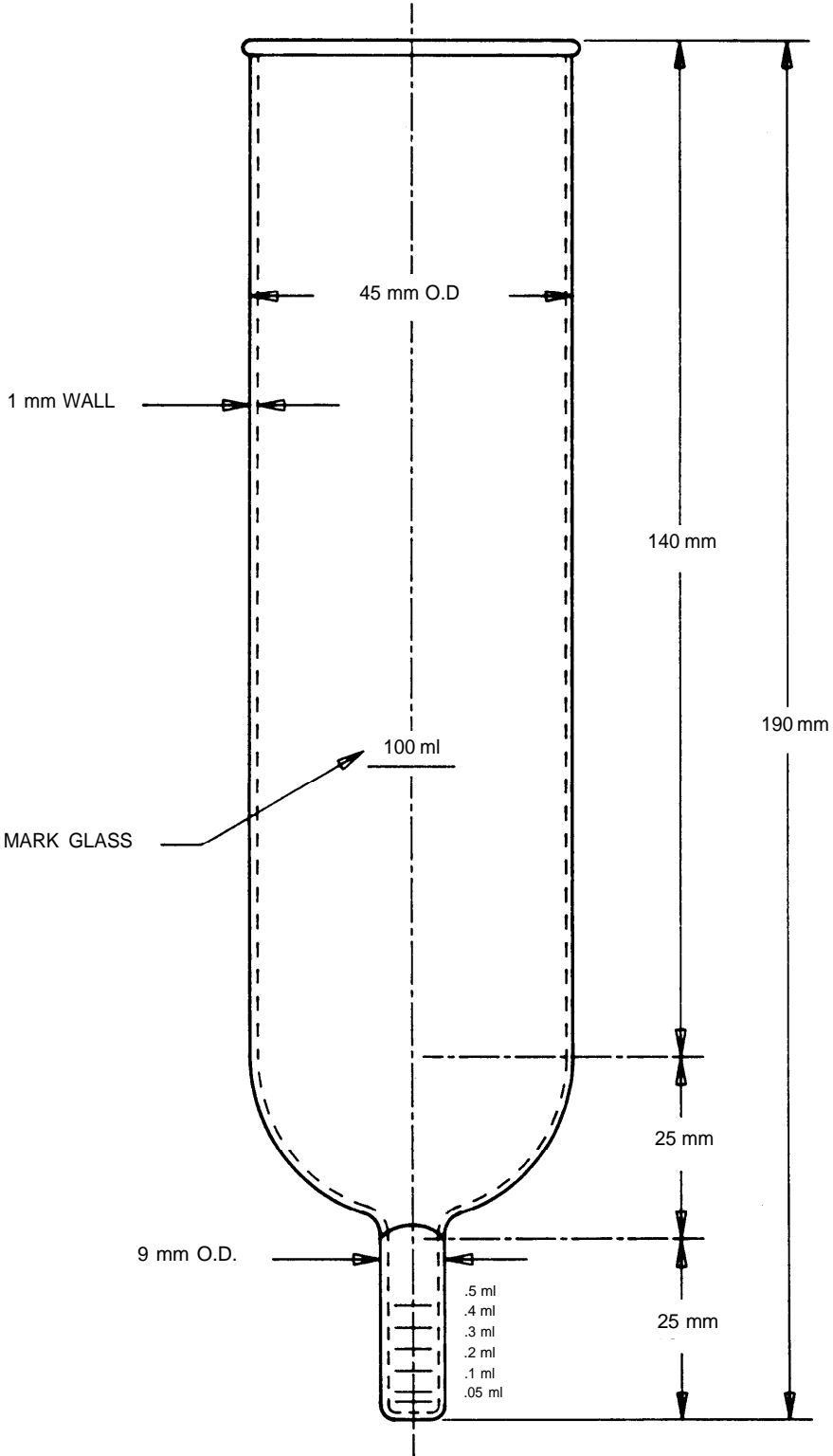
Contaminated ammonia is introduced through the liquid inlet drain. Regenerator must be located so that sufficient head exists to drive the liquid ammonia into it. The suction valve is then opened and warm water at a temperature of approximately 125°F is circulated through the water jacket. After all the ammonia is evaporated, the remaining liquid contaminant may be blown off through the drain line.

# APPENDIX A

## USS Cold-flo Sampling Converter Assembly



# APPENDIX B



AMMONIA EVAPORATION TEST TUBE



## APPENDIX C

### COLD-FLO™ SAMPLING CONVERTER Operating Instructions

The Cold-Flo sampling converter allows safe and convenient sampling of compressed anhydrous ammonia by separating the liquid from the vapor phase. A complete sampling unit consists of a shut-off valve, orifice plate holder, converter, vapor hose, and glass tube. The device includes an orifice plate at the inlet to limit the maximum flow of ammonia. To assure safe and reliable operation follow the instructions listed below. WEAR GLOVES AND GOGGLES.

1. Select orifice plate. Two plates are provided for various pressures. The converter assembly comes with the high-pressure orifice plate installed.

HOT - At pressures above 100 psig use orifice plate #.014

COLD - Below 100 psig use orifice plate #.025

2. Connect the sampling converter to a liquid outlet of the ammonia tank. Make sure the glass tube is securely attached to the converter outlet. Arrange the vapor hose to discharge downwind.
3. Close the valve at the sampling converter and crack the valve at the tank. Check for leaks
4. Open converter valve one turn. Depending on the length of pipe upstream of the converter and the temperature, it may take several minutes for the air and vaporized ammonia to pass. Do not assume orifice is plugged, if you do not immediately receive liquid ammonia - wait at least 5 minutes.

If unit is operating properly it should take approximately 30-60 seconds to collect 100 ml liquid ammonia. Close the converter valve when the liquid in the glass vessel is approximately 0.5 in. below the 100 ml mark to allow for run off. Fill to mark by intermittent opening of valve.

5. It is best to discard first sample to eliminate contaminants from piping. Attach new glass tube and use second sample for purity determination.

## APPENDIX D

The formula for the percent of water by weight referenced on Page 9 is derived as follows:

1. Determine the weight of the initial sample.

$$\begin{aligned}\text{Weight (grams)} &= \text{ml of sample} \times 0.683 \text{ gms/ml (density of anhydrous ammonia at } -28^\circ \text{ F)} \\ &= \text{ml of sample} \times 0.683\end{aligned}$$

2. Determine the weight of the ammonia removed from the system.

$$\begin{aligned}\text{Weight (grams)} &= \frac{\text{weight of the initial sample}}{\text{Evaporation Factor}} \\ &= \frac{\text{ml of sample} \times 0.683}{\text{EF}}\end{aligned}$$

3. Determine the weight of water in the residue solution.

$$\begin{aligned}\text{Weight (grams)} &= \text{ml of residue} \times 0.892 \text{ (density of a 30\% ammonia solution)} \\ &\quad \times 0.70 \text{ (residue solution 70\% water)} \\ &= \text{ml residue} \times 0.892 \times 0.70 \\ &= \text{ml residue} \times 0.624\end{aligned}$$

4. Percent water by weight =  $\frac{\text{weight of water in residue}}{\text{weight of ammonia removed from system}} \times 100$

$$\begin{aligned}&= \frac{\text{ml residue} \times 0.624}{\frac{\text{ml sample} \times 0.683}{\text{EF}}} \times 100 \\ &= \frac{\text{ml residue} \times 0.914 \times \text{EF}}{\text{ml sample}} \times 100\end{aligned}$$

## REFERENCES

*ASHRAE, 1980 Systems Handbook*, Chapter 30

*Compressed Gas Association, Pamphlet G-21977*, Anhydrous Ammonia

*Federal Specification, Ammonia Technical* Number O-A-445B, February 25, 1975

*U.S. Steel, Synthetic Ammonia*, Bulletin ADVCO 85043-48



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