

# FUNDAMENTALS OF GAS DEHYDRATION DESIGN AND OPERATION WITH GLYCOL SOLUTIONS

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Natural gas as it occurs in nature contains many contaminants, the most common of which is water. Most natural gases will be near water saturation at the temperature and pressure of production. When the gas is transmitted to the surface for processing and ultimate pipeline transmission, a reduction in pressure and temperature occurs naturally in the well string. This reduces the capacity of the natural gas to hold water vapor and free water is condensed. The liquid water is removed by suitable knockout facilities up stream of any dehydration equipment. The water remaining in the vapor state must be reduced so the gas will meet the normal contract specification range of 2-7 pounds of water/MM scf.

## GAS HYDRATES

The dehydration of natural gas by any means is done primarily to prevent the formation of gas hydrates during the transmission of natural gas. Gas hydrates have a crystalline structure similar in appearance to water crystals, but their formation occurs at considerably higher temperatures. Natural gas hydrates are composed principally of hydrocarbons and water, although gases such as hydrogen sulfide, ammonia, carbon dioxide, acetylene and bromine may also form hydrates. The initial hydrate formation depends upon the presence of free water, but the hydrate can be propagated by water vapor.

It has been shown by Von Steckelberg (1) and Claussen (2) that although hydrocarbon molecules larger than butane will not form hydrates, hydrate cells with as many as 136 molecules of water

may be formed with methane.

The formation of hydrates in pipelines can cause the complete shut-off of gas flow under severe conditions and at best will result in decreased throughput, frozen control valves, plugged orifices and many other operating problems.

In order to determine if a gas under a specific set of conditions and specifications requires dehydration, reference to Figure 1 would indicate the hydrate forming conditions which apply in a given case. The conditions under which hydrates are likely to be encountered in natural gas are: high pressure, the presence of liquid water, and low temperatures. While it is possible to avoid hydrate formation by heating the gas to temperatures above the hydrate point, this approach is ordinarily impractical and uneconomical for regular pipeline transmission. Therefore, in order to prevent hydrate formation, the water is removed from the gas stream to a dew point below the coldest temperature expected during transmission.

On a given set of inlet gas conditions, namely pressure, temperature, and gas volume, reference to Figure 2 (12) would indicate the quantity of water in pounds/MM scf that must be removed to meet a typical contract specification, such as six pounds of water/MM scf.

## DEHYDRATION METHODS

There are a number of processes for the removal of water to meet pipeline specifications. Calcium chloride was one of the early dessicants used for the removal of water from natural gases (3). The removal of water is effected by contacting a gas stream with a bed of anhydrous calcium chloride which has the ability of absorbing 6 moles of water/mole of  $\text{CaCl}_2$  before forming brine. In many units this brine is used for partial removal of water before contacting the dry calcium chloride bed. Although dew point depressions of  $50^\circ\text{-}80^\circ\text{F}$  can be achieved, the process is limited to gases containing low quantities of water to be removed and relatively small volumes of gas to be processed. The equipment is inexpensive and as long as the chemical demand is not high, the operating expenses are minimal.

Expansion refrigeration and expansion refrigeration with hydration inhibitors are used for dehydration of gas streams in conjunction with hydrocarbon recovery (4). These units are based on the principal that a gas under pressure will experience a sharp drop in temperature during a sudden reduction in pressure. This phenomenon is known as the Joule-Thompson effect and is a fairly exact science. The pressure drop obtained and the amount of cooling that will be achieved. Additional cooling may be obtained by expanding the gas through a turbine.

Some units are operated with heat exchange coils in the bottom of the separator so that hydrate formation will be prevented. This technique is fairly successful in gas streams with rather high hydrocarbon recovery rates so that extremely low temperatures are not necessary in the chiller or separator section. The other systems, operating in the 0° to -40°F range, utilize hydrate inhibitors. Ethylene glycol is the most common hydrate inhibitor used in this application.

Low temperature separation units are not, however, used for the primary purpose of dehydration. They are selected when recoverable hydrocarbon is of prime consideration. Desiccant materials such as silica gel, activated aluminum, molecular sieves and activated carbon have also been used for a number of years for the dehydration of gases (5,6). The method by which these materials dehydrate natural gas is due to the extremely high surface area internal within the porous media. Water vapor will be condensed on a solid in extremely small quantities and, on a plain solid surface, will rapidly reach equilibrium. However, in the case of a highly activated material such as silica gel or one of the other dry desiccants, the condensed water migrates into capillaries producing very deep wells with extremely small radii of curvature. The equilibrium water vapor pressure above such concave surfaces is less than the normal value above a plane surface. Therefore, there is a positive driving force from the gas to the dry desiccants until such time as the capillaries have reached capacity and the partial pressure of water in the capillaries equals that in the gas stream. The dry desiccants will have capacities ranging from 5% to a high of 20%; dew points as low as -140°F have been achieved using this technique.

Since heat of adsorption of water on dry desiccants is about 1800 BTU/pound adsorbed, high energy is required for regeneration. The dry desiccants are regenerated by the reverse process in which a heated gas having a high capacity for water passes through the bed and strips the desiccant of its entrapped water. Dry desiccants are among the more expensive processes for gas dehydration but they are used when extremely dry gas is required or where operating conditions preclude the use of other desiccant materials.

Liquid desiccants have found an extremely wide use for the dehydration of natural gas, and the bulk of this discussion will be centered upon glycols as utilized in gas dehydration. The hygroscopic nature of the glycols is responsible for their use in this application (7). At one time other hygroscopic materials such as glycerine were used but thermal stability and viscosity limitations of these materials gradually left the glycols as the only significant liquid desiccant in the gas dehydration field.

The hygroscopicity of glycols is directly related to the solution concentration. The vapor pressure of the water in any aqueous solution is proportional to the mole fraction of the water in the solution multiplied by the vapor pressure of pure water. The water vapor will be absorbed by the solution as long as the partial pressure of the water vapor in the gas in contact with the solution exceeds the partial pressure of the water in the solution.

The molecular compatibility of the solvent and solute--or glycol and water--plays an important role in determining hygroscopicity. In general, the greater the molecular attraction between the glycol and the water, the lower the water vapor pressure of the solution and the greater the hygroscopicity of the solvent. In the liquid state, water is highly associated through hydrogen bonding. Glycols, with their ether and hydroxyl groups, form similar intramolecular associations with water. Consequently, the glycols have a high affinity for water, and the water-glycol solutions formed greatly reduce the water-vapor pressure. In fact, the partial pressure of water vapor over a glycol solution is less than that predicted from Raoult's Law.

#### DEHYDRATION WITH GLYCOL

Glycol solutions have been used for the drying of natural gas for at least 35 years. The early glycol dehydration units utilized diethylene glycol and obtained dew point depressions in the 20°-40°F range. Triethylene glycol came into use (around 1950) primarily because its higher boiling point provided better separation of water and greater dew point depressions without attendant thermal decomposition of the glycol. Tetraethylene glycol has been used in some specialized cases, but the dominant glycol in use today is triethylene glycol and this discussion will be confined largely to that system.

In the event that dehydration of a gas stream will be required, the following information will be needed to determine whether or not a glycol dehydrator will be adequate for the drying application, and if so, what type of unit would be best suited for the particular job.

If the flowing conditions of the well and the contractual dew point requirements indicate a dew point depression not greater than 70°F, an "off the shelf" unit with 4-5 trays will be adequate with a reboiler capacity to provide a 98.5% triethylene glycol concentration. Dew point depression requirements in the 70°-90°F range can be achieved by conventional glycol dehydrators having additional trays in the contactor, a higher than normal glycol circulation rate, and a maximum reboiler temperature of 400°F. Dew Point depressions greater than 90°F will

normally require specialized glycol dehydration equipment.

Vacuum regenerated glycol concentrations approaching the 99.9% level have been achieved and this coupled with 7-14 tray contactors will give dew point depressions in the 140°F range. Vacuum operated glycol units are not commonplace due to their high cost of operation and the unavailability of adequate vacuum sources such as 400 pound steam for steam-jet ejectors or an adequate size vacuum pump (8).

Gas stripping, or the provision of a sufficient quantity of a hot stripping gas, in a suitable contact device will remove residual water from partially dehydrated glycol. Gas from gas operated pumps, flash tanks, as well as additional raw gas may be utilized for this purpose. Glycol concentrations in the 99.97% range have been reported with attendant dew point depressions in the 160°F area (9).

Glycol concentrations of 99.9% and above have been obtained by means of azeotropic distillation, or the DRIZO Process, with reboiler temperatures in the 375°-400°F range (10). Concentrations of glycol have been reported in excess of 99.5% through the Cold Finger Condensor technique (11).

## DESCRIPTION OF PROCESS

For the following description of the process and flow through a typical glycol dehydrator refer to Figure No. 3. The wet inlet gas stream first enters the unit through an inlet gas scrubber where any liquid accumulations are removed. A two phase or distillate-gas scrubber is illustrated in Figure No. 3. If any liquid water is in the gas stream, a three-phase scrubber may be used to discharge the distillate and water from the vessel separately. The mist eliminator aids in removing any entrained liquid particles from the wet gas stream leaving the top of the inlet scrubber.

The wet gas then enters the bottom of the glycol-gas contactor and flows upward through the trays as illustrated countercurrent to the glycol flowing downward through the column. The gas contacts the glycol on each tray and the glycol absorbs the water vapor from the gas stream. The dry gas leaves the top of the contactor vessel through another mist eliminator which aids in removing any entrained glycol droplets from the gas stream. The gas then flows down through a vertical glycol cooler, usually fabricated in the form of a concentric pipe heat exchanger, where the outlet dry gas aids in cooling the hot regenerated glycol before it enters the contactor. The dry gas then leaves the unit from the bottom of the glycol cooler.

The dry glycol enters the top of the glycol-gas contactor from the glycol cooler and is injected onto the top tray. The glycol flows across each tray and down through a downcomer pipe onto the next tray. The bottom tray downcomer is fitted with a seal pot to hold a liquid seal on the trays.

The wet glycol which has now absorbed the water vapor from the gas stream leaves the bottom of the glycol-gas contactor column, passes through a high pressure glycol filter which removes any foreign solid particles that may have been picked up from the gas stream, and enters the power side of the glycol pump. In the glycol pump the wet high pressure glycol from the contactor column is used to pump the dry regenerated glycol into the column. The wet glycol stream flows from the glycol pump to the inlet of the flash separator. The low pressure flash separator allows for the release of the entrained solution gas which has to be used with the wet glycol to pump the dry glycol into the contactor. The gas separated in the flash separator leaves the top of the flash separator vessel and may be used to supplement the fuel gas and stripping gas required for the reboiler. Any excess vent gas is discharged through a back pressure valve.

The flash separator is equipped with a liquid level control and diaphragm motor valve which discharges the wet glycol stream through a heat exchange coil in the surge tank to preheat the wet glycol stream. If the wet glycol stream absorbs any liquid hydrocarbons in the contactor, it may be desirable to use a three phase flash separator to separate the glycol from the liquid hydrocarbons before the stream enters the reboiler. Any liquid hydrocarbons present in the reboiler will cause undue glycol losses from the stripping still.

The wet glycol stream leaves the heat exchange coil in the surge tank and enters the stripping still mounted on top of the reboiler at the feed point in the still. The stripping still is packed with a ceramic intalox saddle type packing and the glycol flows downward through the column and enters the reboiler. The wet glycol passing downward through the still is contacted by hot rising glycol and water vapors passing upward through the column. The water vapors released in the reboiler and stripped from the glycol in the stripping still pass upward through the still column through an atmospheric reflux condenser which provides a partial reflux for the column. The water vapor then leaves the top of the stripping still column and is released to the atmosphere.

The glycol flows through the reboiler in essentially a horizontal path from the stripping still column to the opposite end. In the reboiler the glycol is heated to approximately 350-400°F to remove enough water vapor to reconcentrate it to 99.5% or more. In field dehydration units the reboiler is generally

equipped with a direct fired firebox using a portion of the natural gas stream for fuel. In plant type units the reboiler may be fitted with a hot oil heated coil or steam coil. A temperature control in the reboiler operates a fuel gas motor valve to maintain the proper temperature in the glycol. The reboiler is also generally equipped with a high temperature safety overriding temperature controlled to shut down the fuel gas system in case the primary temperature control should malfunction.

In order to provide extra dry glycol, 99% plus, it is usually necessary to add some stripping gas to the reboiler. A valve and small pressure regulator are generally provided to take a small amount of gas from the fuel gas sytem and inject it into the bottom of the reboiler through a spreader system. This stripping gas will "roll" the glycol in the reboiler to allow any pockets of water vapor to escape which might otherwise remain in the glycol due to its normal high viscosity. This gas will also sweep the water vapor out of the reboiler and stripping still and by lowering the partial pressure of the water vapor in the reboiler and still column, allow the glycol to be reconcentrated to a higher percentage.

The reconcentrated glycol leaves the reboiler through an overflow pipe and passes into the shell side of the heat exchanger-surge tank. In the surge tank the hot reconcentrated glycol is cooled by exchanging heat with the wet glycol stream passing through the coil. The surge tank also acts as a liquid accumulator for feed for the glycol pump. The reconcentrated glycol flows from the surge tank through a strainer and low pressure filter into the glycol pump. From the pump it passes into the shell side of the glycol cooler mounted on the glycol-gas contactor. It then flows upward through the glycol cooler where it is further cooled and enters the column on the top tray.

## DEHYDRATOR DESIGN

Triethylene glycol dehydrators utilizing tray or packed column contactors may be sized from standard models by using the following procedures and associates graphs and tables contained in Appendix B. Custom design glycol dehydrators for specific applications may also be designed using these procedures. The following information must be available on the gas stream to be dehydrated:

1. Gas flow rate, MMSCFD
2. Specific gravity of gas
3. Operating pressure, psig

4. Maximum working pressure of contactor, psig
5. Gas inlet temperature, °F
6. Outlet gas water content required, lbs/MMSCF

Having the above information, it is then necessary to select two points of design criteria.

1. Glycol to water circulation rate based on water removed. A value of 2 to 6 gal. TEG/lb H<sub>2</sub>O removed is adequate for most glycol dehydration requirements. Use 2.5 to 4 gal. TEG/lb H<sub>2</sub>O for most field dehydrators.
2. Lean TEG concentration from reconcentrator. 99.0 to 99.9% lean TEG is available from most glycol reconcentrators. A value of 99.5% lean TEG is adequate for most design considerations.

The following procedures may be used to size a glycol dehydrator for a specific set of conditions, evaluate performance and determine the gas capacity of a given size unit.

A good inlet scrubber is essential for efficient operation of any glycol dehydrator unit. Inlet Scrubber The required diameter of a vertical inlet scrubber may be selected using Figure No. 4 based on the operating pressure of the unit and gas capacity required. Two phase inlet scrubbers are generally constructed with 5' to 7 1/2' shell heights. Additional data on typical standard vertical inlet scrubbers are contained in Tables 4A and 4B in Appendix B.

Select a contactor diameter based on Glycol-Gas Contactor the operating pressure required with the approximate required gas capacity from Figure 5 or 6. Figure 5 is for glycol contactors using trayed columns and Figure 6 is for contactors using packed columns. The gas capacities as determined for a given diameter contactor from Figure 5 or 6 must be corrected for the operating temperature and gas specific gravity.

Calculate the gas capacity of the gas-glycol contactor selected for the specific operating conditions.

$$G_o = G_s (C_t) (C_g)$$

Where:  $G_o$  - Gas capacity of contactor at operating conditions, MMSCFD

$G_s$  = Gas capacity of contactor at standard conditions (0.7 sp gr and 100°F), based on operating pressure, MMSCFD



$C_t$  = Correction factor for operating temperature

$C_g$  = Correction factor for gas specific gravity

The temperature and gas specific gravity correction factors for trayed glycol contactors are contained in Tables 1A and 1B respectively. The temperature and specific gravity factors for packed glycol contactors are contained in Tables 2A and 2B respectively.

Next, determine the required dew point depression and the water removed for the glycol dehydration unit from the following:

Dew point depression,  $F = \text{Inlet gas temp. } ^\circ\text{F} - \text{Outlet dew point temp. } ^\circ\text{F}$

$$W_r = \frac{(W_i - W_o) (G)}{24}$$

Where:  $W_r$  = Water removed, lb/hr

$W_i$  = Water content of inlet gas, lb H<sub>2</sub>O/MMCF

$W_o$  = Water content of outlet gas, lb H<sub>2</sub>O/MMCF

$G$  = Gas flow rate, MMSCFD

The outlet dew point temperature can be found on the water content graph, Figure 2 (12), using the outlet gas water content required and the operating pressure. The dew point temperature is the temperature at which the remaining water vapor in the gas will start to condense. The inlet gas temperature is also the inlet dew point temperature since the gas is generally assumed to be water saturated before it is dehydrated. The water content of the inlet gas can be determined from the same water vapor content graph using the inlet gas temperature and the operating pressure.

If the natural gas stream contains appreciable amounts of either carbon dioxide and/or hydrogen sulphide, the water content of these sour gases should be taken into account in determining the total water content of the inlet gas stream. Since both carbon dioxide and hydrogen sulphide gases absorb considerably more water vapor than natural gas, they appreciably increase the total water content and dehydration requirements of the gas stream.

Trayed Contactors                      Select the number of actual trays required from Figure 7 using the required dew point depression and the selected glycol to water

circulation rate. The data contained in Figure 7 will give the approximate number of trays required for rapid sizing of field glycol dehydrators. A more detailed consideration of the actual number of trays required will give the accurate results needed for the most economical size contactor.

For a more detailed study, a modified McCabe-Thiele diagram (15) can be constructed to determine the number of theoretical trays for a triethylene glycol dehydrator. This number can be converted to the actual number of trays required by applying the tray efficiency.

First, determine the concentration of the rich TEG leaving the bottom of the glycol-gas contactor.

$$\rho_i = \text{Sp Gr}(8.34)$$

$$\text{Rich TEG} = \frac{(\text{Lean TEG})(\rho_i)}{\rho_i + \frac{1}{L_w}}$$

Where:  $\rho_i$  = Density of Lean TEG Solution, lb/gal

Sp Gr = Specific gravity of Lean TEG Solution at operating temperature of contactor

Rich TEG = Concentration of TEG in rich solution from contactor, %/100

Lean TEG = Concentration of TEG in lean solution to contactor, %/100

$L_w$  = Glycol to water circulation rate, gal TEG/lb H<sub>2</sub>O

The operating line for the McCabe-Thiele diagram is based on connecting a line between a point indicating the top of the column and a point indicating the bottom of the column.

Top of Column: lb H<sub>2</sub>O/MMCF in outlet gas and lean TEG, %

Bottom of Column: lb H<sub>2</sub>O/MMCF in inlet gas and rich TEG, %

The equilibrium Line on the McCabe-Thiele diagram can be constructed by determining the water content of the gas which would be in equilibrium with various concentrations of triethylene glycol. This can be done by filling in the following Table:

Equilibrium Dew Point Temp. % TEG at Contactor Operating Temp.*	Water Content of Gas at Dew Point Temp. and Contactor Operating Pressure, lb H <sub>2</sub> O/MMCF**
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99  
98  
97  
96  
95

\*Determine from the chart of equilibrium water dew points of glycol solutions at various contact temperature, Figure 8 (13).

\*\*Determine from the chart of water vapor content of gas at various temperatures and pressures, Figure 2 (12).

The modified McCabe-Thiele diagram can then be constructed with the operating line and equilibrium line and then stepped off by triangulation to determine the theoretical number of trays required. This procedure is best illustrated by an example which is included in the Appendix A and Figure 9.

The actual number of trays then required can be determined using the tray efficiency.

$$\text{No. Actual Trays} = \frac{\text{No. Theo. Trays}}{\text{Tray Eff.}}$$

Where: Tray Efficiency = 25% for bubble cap trays  
 = 33-1/3% for valve trays

The number of actual trays required as determined from either Figure 7 or by construction of McCabe-Thiele diagram is based on both theoretical and actual test data using a typical natural gas. Select the next whole number of trays based on the above design procedures after the tray efficiencies have been considered. However, good operation of field dehydrators indicates that a minimum of four trays should be used in any glycol-gas contactor.

Standard field dehydration contactors normally have 24" tray spacing. Due to the tendency of glycol to foam in the presence of liquid hydrocarbons, it is recommended that no less than 24" tray spacing be used to prevent any field problems with the equipment. If any foaming problem does occur, closer tray spacing can result in carryover or entrainment of the glycol in the gas stream, and cause excessive glycol losses as well as decreased efficiency in dehydration of the gas.

Packed Contactors      The same procedures can be used for packed column contactors and the depth of packing required can be determined from Figure 7. It is determined in the same manner using the required dew point depression and the selected glycol to water circulation rate. If a more detailed consideration of the depth of packing is required a modified McCabe-Thiele diagram can be drawn based on the same procedures as described above. The depth of packing required can then be determined from the following empirical relation based on using 1" metal pall rings in the contactor.

$$\text{Depth Packing, ft} = \text{No. Theo. Trays} (3)$$

Then select the next whole number of feet of packing for use in the contactor. However, good operation indicates that a minimum four feet of packing should be used in any gas-glycol contactor.

Additional specifications for standard tray type glycol-gas contactors are contained in Tables 5A and 5B in Appendix B. Data on packed column glycol-gas contactors is contained in Tables 6A and 6B in Appendix B.

Glycol Reconcentrator      For the detailed consideration involved in sizing the various components of the glycol reconcentrator it is first necessary to calculate the required glycol circulation rate.

$$L = \frac{L_w (W_i) (G)}{24}$$

Where: L = Glycol circulation rate, gal/hr

$L_w$  = Glycol to water circulation rate, gal TEG/lb H<sub>2</sub>O

$W_i$  = Water content of inlet gas, lb H<sub>2</sub>O/MMSCF

G = Gas flow rate, MMSCFD

Reboiler      The required heat load for the reboiler can be estimated from the following equation:

$$Q_t = 2000(L)$$

Where:  $Q_t$  = Total heat load on reboiler, BTU/hr

L = Glycol circulation rate, gal/hr

The above formula for determining the required reboiler heat load in an approximation which is accurate enough for most

high pressure glycol dehydrator sizing. A more detailed determination of the required reboiler heat load may be made from the following procedure:

$$Q_1 = L (\rho_i) (c) (T_2 - T_1)$$

$$Q_w = \frac{970.3 (W_i - W_o) (G)}{24}$$

$$Q_r = 0.25 (Q_w)$$

$$Q_h = 5,000 \text{ to } 20,000 \text{ BTU/hr depending on boiler size}$$

$$Q_t = Q_1 + Q_w + Q_r + Q_h$$

Where:  $Q_1$  = Sensible heat required for glycol, BTU/hr

$Q_w$  = Heat of vaporization required for water, BTU/hr

$Q_r$  = Heat to vaporize reflux water in still, BTU/hr

$Q_h$  = Heat loss from reboiler and stripping still, BTU/hr

$Q_t$  = Total reboiler heat load, BTU/hr

$L$  = Glycol circulation rate, gal/hr

$\rho_i$  = Glycol density at average temperature in reboiler, lb/gal = (sp gr) (8.34)

$C$  = Glycol specific heat at average temperature in reboiler, BTU/lb-F

$T_2$  = Glycol outlet temperature, °F

$T_1$  = Glycol inlet temperature, °F

970.3 = Heat of vaporization of water at 212°F 14.7 psi, BTU/lb

$W_i$  = Water content of inlet gas lb H<sub>2</sub>O/MMSCF

$W_o$  = Water content of outlet gas lb H<sub>2</sub>O/MMSCF

$G$  = Gas flow rate, MMSCFD

NOTE: For high pressure glycol dehydrators,  
 $\rho_i (C) (T_2 - T_1) \approx 1200$

If the size of the reboiler and stripping still is known or is estimated, the heat loss can be more accurately determined from the following equation:

$$Q_1 = 0.24 (A_s) (T_v - T_a)$$

Where:  $Q_1$  = Overall heat loss from reboiler and still  
BTU/hr

$A_s$  = Total exposed surface area of reboiler  
and still, sq ft

$T_v$  = Temperature of fluid in vessel, °F

$T_a$  = Minimum ambient air temperature, °F

0.24 = Heat loss from large insulated surfaces,  
BTU/hr sq ft - °F

The actual surface of the firebox required for direct fired reboilers can be determined from the following equation which is based on a design heat flux of 7,000 BTU/hr - sq ft. By determining the diameter and overall length of the U-tube firebox required to give the total surface area as calculated, the general overall size of the reboiler can be determined.

$$A = \frac{Q_t}{7000}$$

Where: A = Total firebox surface area, sq ft

$Q_t$  = Total heat load on reboiler, BTU/hr

**Glycol Circulating Pump** The required size of glycol circulating pump can be readily determined using the glycol circulation rate and the maximum operating pressure of the contactor. The most commonly used type of glycol pump for field dehydrators is the glycol powered pump which uses the rich glycol from the bottom of the contactor to power the pump and pump the lean glycol to the top of the contactor. Sizing data for this type of glycol pump is contained in Table 8 (14) of Appendix B. For motor driven positive displacement or centrifugal pumps the manufacturers of these pumps should be consulted for exact sizing to meet the specific needs of the glycol dehydrator.

**Glycol Flash Separator** It is advisable to install a flash separator downstream from the glycol pump (especially when the glycol powered type pump is used) to remove any entrained hydrocarbons from the rich glycol. A small, 125 psi W.P., vertical two

phase separator is adequate for this purpose. The separator should be sized based on a liquid retention time in the vessel of at least five minutes.

$$V = \frac{L(T)}{60}$$

Where: V - Required settling volume in separator, gal

L = Glycol circulation rate, gal/hr

T = Retention Time - 5.0 minutes

Liquid hydrocarbon should not be allowed to enter the glycol-gas contactor. Should this be a problem, a three phase glycol flash separator will keep these liquid hydrocarbons out of the reboiler and stripping still. A liquid retention time of 10 to 30 minutes should be used in the above equation to size a three phase flash separator.

The hydrocarbon gas released from the flash separator can be piped to the reboiler to use as fuel gas and stripping gas. The amount of gas available from a glycol pump can be determined from the data in Table 8 in Appendix B based on the glycol circulation rate and the operating pressure of the contactor.

The size of the packed stripping still for use with the glycol reconcentrator can be determined from Figure No. 10. The diameter required for the stripping still is normally based on the required diameter at the base of the still using the vapor and liquid loading conditions at that point. The vapor load consists of the water vapor (steam) and stripping gas flowing up through the still. The liquid load consists of the rich glycol stream and reflux flowing downward through the still column. The minimum cross sectional area and/or diameter required for the still as read from Figure No. 10 is based on the glycol to water circulation rate, gal TEG/lb H<sub>2</sub>O and the glycol circulation rate, gal/hr.

Normally, one theoretical tray is sufficient for most stripping still requirements for triethylene glycol dehydration units. For conservative design the height of packing using 1-1/2" ceramic Intalox saddles is held at a minimum of 4'. Conservative design and field test data indicates that this height should be gradually increased with the size of the glycol reconcentrator to a maximum of approximately 8' for a 1,000,000 BTU/hr unit.

The amount of stripping gas required to reconcentrate the glycol to a high percentage will usually be approximately 2 - 10

cu ft per gal of glycol circulated. This stripping gas requirement has been determined using Figure No. 10.

Specifications for the main components of standard size glycol reconcentrators are contained in Table 7 in Appendix B.

## GLYCOL ANALYSIS

The choice of glycol and the merits of various dehydrator fluids are discussed elsewhere (17), it will be assumed here that triethylene glycol (TEG) is the fluid of choice even though in some limited applications other glycols could be used.

Incoming Quality Check of Glycol Although most operators do not perform a quality check on new glycol, this is of concern, and properly so, to several companies. The physical and chemical properties of the glycols are described in detail in the literature (18) and in publications issued by most major glycol producers (19-22). Listed below are some typical properties of TEG which are useful in determining whether a new material is acceptable for use in a gas dehydrator.

### List 1. Some Typical Properties of Technical Grade Triethylene Glycol\*

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#### Composition, % wt

Water	<0.1
Ethylene glycol (EG)	<0.05
Diethylene glycol (DEG)	<1.0
Tetraethylene glycol (T <sub>4</sub> EG)	<1.0

#### Boiling Range\*\* °C

I.B.P.	278
95% B.P.	285
D.P.	300

Chlorides, as Cl <sup>-</sup>	<5 ppm
P <sup>H</sup> (50% aqueous solution)	6-7

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\* Not to be confused with specifications

\*\* See method in Appendix C

Purposely, a number of other properties have been omitted from the above list, specific gravity for one. Although specific



gravity will be part of the specification, it is of little utility as a quality check. It is affected, among other things, by water content. A water analysis, therefore, coupled with a boiling range determination, chloride and pH analysis will serve well in establishing the suitability of new TEG for gas dehydration. Although the water content is usually very low, it should be considered that numerous glycol dehydrators operate normally with 1.0 to 1.5% H<sub>2</sub>O in the lean TEG, so that even 2-3% water should not be cause for rejection. This discussion is not intended to advocate a relaxation in product specifications. On the contrary, it is intended as an aid for interpreting the QC check. Consider, for example, the cost for removing 3.0% H<sub>2</sub>O in a 5,000 gal shipment of glycol:

$$5,000 \text{ gal} \times 9.3 \frac{\text{lb}}{\text{gal}} \times 0.03 \frac{\text{lb H}_2\text{O}}{\text{lb TEG}} \times 970.3 \frac{\text{Btu}}{\text{lb H}_2\text{O}} = 1.35 \times 10^6 \text{ Btu}$$

$$1.35 \times 10^6 \times \$2.00/\text{MM Btu} = \$2.70$$

Clearly, the water content will not have a great impact on the operation of the dehydrator. Very important, instead, can be the boiling range determination of TEG, since it is related to the presence of other glycols--and one should aim to minimize the lower glycols (EG, DEG) and the problems associated with them (17). Similarly, the chloride analysis is an important one since the presence of salt can cause fouling of the equipment, and pH is an indication of the corrosivity of the solution. Both pH and boiling range determinations can be done with very simple equipment even in isolated locations and can be considered as the necessary and sufficient checks to be run routinely on new TEG, with a more complete analysis occasionally.

Monitoring Glycol Conditions - List 2 outlines the various analyses run on glycol samples. Analysis and Troubleshooting - Much can be learned from simple observations done in the field by the operator and often corrective measures can be deduced from the visual inspection. For example, the presence of a finely divided, black precipitate, iron corrosion products such as FeS and Fe<sub>3</sub>O<sub>4</sub>, reveals ongoing corrosion and may also indicate the inadequacy of the filters. A black, viscous solution indicates that heavy, tarry hydrocarbons are carried over with the gas. A sweet burnt sugar smell accompanied by low pH and a dark, clear solution signals that thermal degradation is occurring in the reboiler.

List 2. Glycol Analysis for Dehydrator Field Samples

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	<u>Method</u>
1. Water analysis	Karl Fischer Titration
2. pH (1:1 dilution)	Potentiometric
3. Emulsion formation	Visual
4. Gas chromatography (H <sub>2</sub> O, Glycol, Acid Gases, hydrocarbons)	Instrumental
5. Hydrocarbon distillation	Modified Dean-Stark or ASTM
6. Foaming tendency	Appendix D
7. Chlorides	
Inorganic	AgNO <sub>3</sub> - Volhard
Total	Phrolysis + coulometric titration
8. Na, K	Atomic absorption (A.A.)
9. Ca	A.A. & qualitative
10. Fe	Colorimetric
11. Other metals	Emission methods
12. Other analyses as needed	-

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Note: Several detailed procedures for the analysis of glycols are given in Ref. 6; some, modified and updated, appear in Appendix C. Instrumental methods of analysis, e.g., A.A. and emission are described in standard texts of analytical chemistry (23).

Often, however, specific recommendations can be made only after a complete analysis; therefore, the methods of List 2 and their significance will be described in detail.

Water Analysis      The most reliable and convenient method of analysis for glycol solutions is the Karl Fischer titration. In an alcohol-pyridine

solution, iodine is not reduced by sulfur dioxide unless water is present. Karl Fischer reagent, which contains all of these components except water, can be used for titrating the latter. The only significant interference is due to the presence of sulfides in the glycol sample, e.g., H<sub>2</sub>S. When these are suspected, or identified by a qualitative test (19), a separate analysis for either total sulfur or H<sub>2</sub>S can be done to determine the extent of interference.

Relatively inexpensive, automatic Karl Fischer titrators can be utilized to advantage since the end point is determined electronically and the results are displayed directly as micrograms of water. Heavy contaminations of hydrocarbons do not interfere with these analyses but contribute to fouling of the electrodes, so that the titration cell must be cleaned frequently, to avoid instability of the instrument.

A water determination of both "lean" and "rich" TEG samples can be useful in assessing the overall dehydration of efficiency by calculating a material balance around the absorber. An example will be discussed later.

**p<sup>H</sup> Determination** A potentiometric determination of p<sup>H</sup> is done in the laboratory on a glycol sample which has been diluted with an equal volume of deionized or distilled water whose p<sup>H</sup> has been adjusted to 7.0. Since the dilution is exothermic, care must be taken to calibrate the instrument at the temperature of the solution or, more conveniently, allow the sample to reach room temperature. A glass electrode is used for the measurement with a saturated calomel or Ag/AgCl electrode as the reference. Combination electrodes, where the reference and the glass electrodes form one integral unit, are available and are recommended for this use. After calibration of the meter with fresh buffer solutions (p<sup>H</sup> 4, 7, 10), the p<sup>H</sup> of the glycol sample is determined. Gentle swirling or stirring of the solution is necessary to stabilize the instrument reading.

**Gas Chromatography** Pioneering work on the development of gas chromatography for the analysis of glycol and amine solutions has been reported by Wisniewski and Stalker (24), and by Arnold and Pearce (25). The method described here and attached in Appendix C, is based on these earlier developments and differs mainly in the utilization of automated instrumentation and of more readily available, prepared column packings, as described in Figure 11.

A typical chromatogram for a synthetic mixture of glycols is shown in Figure 12. The method consists of adding a known amount of an internal standard, e.g., 1, 3-butanediol (1, 3-BD), to a weighted amount of a glycol field sample. A

0.2-0.5  $\mu$ l portion of the sample is then injected and analyzed. By appropriate calibration of the instrument with a standard mixture similar to the one shown in Figure 12, the results are calculated, manually or by the instrument, and reported as % wt. Since the main glycol component is not always known for the samples received in this laboratory, a preliminary screening is run without the addition of the internal standard. This permits the selection of the calibration standard which more closely approaches the composition of the unknown. The presence of acid gases ( $\text{CO}_2$ ), ( $\text{H}_2\text{S}$ ), which interfere with the water elution peak, and of other impurities such as hydrocarbons, can also be established by a preliminary run. Samples heavily contaminated with hydrocarbon, solids, or inorganic salts may be analyzed without preliminary sample treatment, however, they tend to shorten the column life. Another inconvenience with these samples is the fact that the impurities may influence the retention times to such an extent that one or more of the component, or reference, peaks are missed where the instrument is programmed to calculate composition with the internal standard method. With some of the programmable integrators the calibration can be edited manually and the report completed.

Most of the hydrocarbon and surfactant-type contaminants can be removed prior to the analysis by slurring a sample of the glycol with approximately 5% wt granular activated carbon (12 x 24 mesh) for one hour in a closed container. The slurry is then filtered (Whatman type 41 paper, with a vacuum or, even better, pressure filtration apparatus. The clear, purified glycol filtrate is then analyzed as described above.

It has been found that inorganic salts and other contaminants will eventually decrease the effectiveness of a GC column and ultimately even physically plug it. Although the column can be partially rejuvenated by the injection of HCl (24), large amounts of water (steam stripping) silylating compounds, or various other solvents, the best solution is often to discard it and start with a new column. Stainless steel, 1/8" x 3', columns are relatively inexpensive, easy to pack and to condition.

With proper precautions the gas chromatographic analysis, internal standard method, can yield results that are accurate to + 2%, or better. This analysis can be very useful in completing a material balance of the field sample. By comparing the amounts of diethylene glycol and ethylene glycol found in a sample of TEG with those to be expected in fresh material (see List 1) one can deduce whether thermal cracking of the glycol is occurring in the reboiler. Care must be exercised, however, in interpreting these results because the lower glycols, due to their higher vapor pressure, are more easily vaporized than TEG and therefore lost from solution.

Hydrocarbon Analysis      The distillation method to be found in Appendix C affords a rapid determination for light hydrocarbons in glycol solutions. Heavier hydrocarbons are more difficult to extract and analyze. One method employed when it is important to identify these contaminants is to extract a sample of the glycol with a nonpolar solvent, e.g., carbon tetrachloride, then analyze the extract by infrared spectroscopy. Calibration of the -C-H stretching band ( $2990\text{ cm}^{-1}$ ) permits at least a semi-quantitative determination of the heavier components. Even more important, by this method, and with the availability of comparison standards, it has been possible to distinguish between contamination resulting from carry-over of hydrocarbons from the natural gas and that originating from compressor oil (26). More specialized techniques such as gas chromatography/mass spectrometry, gel permeation chromatography and nuclear magnetic resonance spectrometry, can be used if more specific identification or quantification is necessary to solve a particularly difficult problem. It is seldom necessary to conduct an extensive, rigorous analysis for these contaminants since the presence of hydrocarbons is readily detected as described here, or qualitatively by observing the formation of an emulsion when the glycol is diluted with water. In most cases, the emulsion can be broken by "salting out" techniques and centrifugation then affords a semi-quantitative analysis.

Foaming Characteristics      The presence of well-treating compounds (surfactant type materials), hydrocarbon oils, solids and other contaminants can cause foaming in gas dehydrators. Excessively high vapor velocities in the absorber, insufficient removal of dissolved natural gas from the glycol, and low contact temperatures (less than  $60^{\circ}\text{F}$ ) also contribute to this problem. The effect of the latter, physical contributors to the foaming problem is difficult to reproduce in the laboratory. The extent of foaming caused by chemical agents, however, can be easily tested. The method and apparatus shown in Appendix C can be used to determine foaming tendencies of dehydrator fluids and the effect of various foam inhibitors, antifoams and treatment with activated carbon. Although normally run at room temperature the test can be easily adapted to study temperature effects by the use of a temperature controlled water bath.

New or clean TEG solutions will form less than 100 ml of foam which will collapse in a few seconds. Solutions causing foam problems in the field, however, will give widely differing results in the laboratory test. Only a few ml of stable foam, one that persists for more than 5-10 seconds, could be an indication of an incipient problem. It is impossible to set strict guidelines for used glycol samples. The test should be employed as a monitoring tool and to screen the effect of

various additives. Many commercial products are effective in controlling foaming problems, especially the silicones; however, they should be considered palliatives. In most cases, the installation of full-flow carbon filters (also commercially available in various standard sizes) eliminates the problem. Clean glycol solutions do not foam!

Inorganic Salts Chloride analysis is one of the most significant tests for glycol solutions. Chlorides are introduced as either sodium or calcium salts ( $\text{NaCl}$ ,  $\text{CaCl}_2$ ) which are the most common and troublesome of the organic contaminants.

Inorganic chlorides may be determined by the classic Volhard method (19) or by an instrumental modification. In the latter, a sample of glycol is titrated with standard  $\text{AgNO}_3$  solution and the end point determined potentiometrically with a silver electrode.

Glycol samples used to dehydrate sour gas may contain  $\text{H}_2\text{S}$  which will interfere with the analysis; the samples are acidified with  $\text{HNO}_3$ ; gentle heating and stirring for several minutes should liberate the volatile hydrogen sulfide. Other interferences are due to the presence of bromide, iodide and thiocyanate ions ( $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{SCN}^-$ ). The first two, however, can be determined simultaneously with  $\text{Cl}^-$  by recording the titration curve and looking for the additional inflection points (breaks in the curve) as shown in Figure 13. The break due to  $\text{SCN}^-$  is usually not sufficiently well defined to permit a separate analysis. Unless a separate determination is made, thiocyanate will be reported as chloride. The precision of the titration, i.e., the sharpness of the end point or the break in the curve, can be improved by keeping the titration vessel at approximately  $40^\circ\text{F}$  by means of an ice-water bath.

A total chloride analysis is sometimes also useful in determining whether interference from sulfur and/or organic chlorides is present. The sample is placed in a platinum boat and oxidized in a furnace at  $800^\circ\text{C}$ . The resulting chloride and oxychloride ions are then titrated coulometrically (27) (see Figure 14). Sulfur compounds are oxidized to  $\text{SO}_2$  and will not, therefore, interfere. Interference from  $\text{Br}^-$  and  $\text{I}^-$ , however, is still possible.

To determine whether the salt contamination is due entirely to sodium chloride or other inorganic salts ( $\text{MgCl}_2$ ,  $\text{KCl}$ ,  $\text{K}_2\text{CO}_3$ ) etc) a metal scan is sometimes run by instrumental methods. Atomic absorption spectrophotometry is very useful for the rapid determination of Na, K, Ca. Other metals are analyzed by emission methods (23).

The presence of calcium may be determined qualitatively by

making a sample of the glycol alkaline, with either an amine or caustic, and the addition of a few drops of 2-hydroxy-1-(2-Hydroxy-4-sulfo-1-naphthylazo)-3-naphthoic acid indicator\* which will form a red complex with  $\text{Ca}^{++}$  ions. If calcium chloride is detected in the glycol solution, a quantitative determination can be done according to the method of reference 6 or by atomic absorption. The concentration of NaCl then may be calculated by difference from the chloride analysis.

Another analysis often run on TEG samples is that for Iron total iron. A colorimetric method using ferrion as the iron complexing agent is described in Appendix C. Portable kits with the reactants predozed and in capsule form are available for field determinations (28). The iron analysis is useful in monitoring the occurrence of corrosion together with information gathered on  $\text{p}^{\text{H}}$ , the presence of acid gases, degradation of glycols, and so forth.

\*Cal-Red Indicator

## OPERATIONAL PROBLEMS

The glycol dehydration unit in general does not have a wide variety of operational problems. The problems can be categorized into three general areas: foaming, contamination and corrosion.

Corrosion/ $\text{p}^{\text{H}}$  Corrosion in a glycol unit can usually be quite easily controlled by an occasional check of the  $\text{p}^{\text{H}}$  of the glycol solution and maintenance of the  $\text{p}^{\text{H}}$  in the 6-8.5 range. The use of Nacap (sodium mercatobenzothiazol) at approximately 0.5 % of monoethanolamine will usually provide adequate protection. In the event corrosion is occurring in the still column vapor space, the use of monoethanolamine at the rate of 1/4 pound/100 gallons of glycol solution will give protection. Diethanolamine may also be used for this purpose, but due to its higher boiling point will not give as much protection in the vapor space.

Corrosion in the glycol system may be caused from acid formation during the degradation of the glycol. This is particularly true if small quantities of oxygen are present. Storage vessels and surge tanks should be blanketed with natural gas to exclude oxygen. Small quantities of acid gases such as  $\text{CO}_2$  or hydrogen sulfide may also contribute to corrosion in the glycol circuit. Mono-, di- or triethanolamine may be utilized to provide adequate corrosion protection in this case. In an older field that is losing pressure and generating a few grains of hydrogen sulfide gas may be adequately sweetened by triethanolamine in the amount of at least 2 moles of TEA for each mole of acid gas to be removed.

The dehydration of gas streams containing significant quantities of acid gas will require a slightly different approach to corrosion control. A sufficient quantity of alkanolamine to react with the acid gas may regenerate  $H_2S$  and  $CO_2$  in the reboiler because contact stages in the stripper column or stripping vapor is insufficient to remove the acid gas before it reaches the reboiler. This requires 2 moles of  $H_2O$ /mole of acid gas for adequate stripping. The stripping  $CO_2$  and/or  $H_2S$  in the reboiler will, of course, result in severe corrosion within the reboiler vapor space. The combination of Nacap and MEA will provide some protection for rich circuit and adequate protection for the lean circuit.

The effect of  $p^H$  on the corrosivity of glycols have been extensively studied. Figure 15 illustrates graphically the effect of  $p^H$  and free acid, respectively, on the corrosivity of DEG on mild steel. Clearly, to prevent corrosion problems it is sufficient to maintain the glycol neutral or slightly alkaline. A  $p^H$  much above 9.0 is undesirable because it can lead to emulsification in the presence of hydrocarbons and result in foaming problems. In glycol dehydrators, acidic products are formed as the result of thermal degradation and/or oxidation of the glycol. The decomposition products are many and the mechanism for their formation is complex. Triethylene glycol, at the conditions found in the reboiler, can be decomposed--more or less gradually--to the lower glycols and these in turn to a host of acidic products. Conditions which are known to favor the decomposition are: high metal skin temperature or bulk solution temperature; the presence of oxygen:  $p^H$  which catalyzes the auto-oxidation of the glycol and metal ions in solution.

Salt Contamination      Salt in glycol solutions is perhaps the most difficult problem encountered with either dehydration or hydrate inhibition applications. Carry-over of brine solutions can occur in almost any formation of natural gas; it is prevalent where well completion fluids may be produced along with the gas, or where wells are produced at higher than normal rates. The most serious problems occur, however, in underground storage wells.

Underground storage sites are usually located near metropolitan areas and quite often in abandoned mines, depleted gas fields, depleted brine formations--all of which produce an inordinate amount of salt. Calcium chloride together with sodium chloride will considerably complicate an already troublesome situation.

Figures 16 and 17 show the solubilities of  $NaCl$  and  $CaCl_2$  in TEG solutions. Notice that sodium chloride has an inverse solubility, i.e., the amount held in solution decreases with increasing temperature. At the conditions of the reboiler,



~400°F and ~99% TEG, most of the NaCl will fall out of solution and deposit on hot surfaces; the fire tube. The decomposition of glycol can then result even under controlled heat flux conditions through the formation of a salt layer which acts as an insulator. The surface temperature of the fire tube builds up at the point of the deposits causing what is commonly known as "hot spots". How severe this problem can be is illustrated by simulating the conditions of the reboiler in the laboratory. A flask containing fresh TEG was externally heated to 400°F in a nitrogen atmosphere. An electric heater was also immersed in the center of the solution to simulate the fire tube. The surface temperature of the electric heater was also controlled at 400°F and monitored by a thermocouple. Without any salt in solution, both the glycol bulk temperature and the heater surface remained at 400°F. With 1% salt, however, the temperature at the surface of the electric heater slowly started to increase and reached 430°F in a few days. The thick salt deposits formed on the heated surface are shown in Figure 18. In practice this can result in tube burn-out and failure. When both hydrocarbons and salt are present, the encrustations on the fire tube become so hardened that ordinary cleaning methods are not effective.

Calcium chloride, which is less soluble at lower temperatures, will precipitate in the cold portion of the plant, and, in severe cases, completely stops the flow of glycol. An additional problem with calcium chloride is the loss of dehydrating capacity. Calcium chloride reacts to form glycolates which are not hygroscopic, thereby reducing the water-carrying capacity of the glycol.

The first and most obvious approach to prevent salt carry-over is to provide efficient knock-out facilities. It is virtually impossible to remove all the brine from gas streams in areas of heavy contamination, therefore the removal of salt from the glycol solution is a necessary step.

Calcium Chloride/Sodium Chloride Removal

In a case of extreme contamination, salt removal has been achieved by utilizing scraped surface heat exchangers (SSE) as shown in Figure 19. The triethylene glycol solution was supersaturated with respect to  $\text{CaCl}_2$  but NaCl was also present so that co-precipitation of the two salts occurred. The discharge from the SSE exchangers went to centrifuges where separation of the glycol from the salt slurry was effected. The use of the centrifuge was found to be more economical and efficient than conventional filtration methods. The co-precipitation of the two salts effected a better purification than what would be expected from the solubility curves for  $\text{CaCl}_2$ . Concentration of chlorides in the cleaned effluent were found to be as low as 400-500 ppm  $\text{Cl}^-$ . Salt contaminated glycol may be also reclaimed by vacuum distillation, ion exchange or ion retardation techniques.

Vacuum Distillation      Figure 20 shows the vapor-liquid composition curves for triethylene glycol at various pressures. Vacuum reclamation at a temperature no greater than 400°F is recommended to reduce thermal degradation. At this temperature and at a pressure of 100 mm Hg, the overhead product will consist of 90-95% wt TEG, depending on the amount of salt and tars present in the glycol. This reclaimed glycol should not be far from the concentration of circulating rich glycol and can be added directly to this stream for reconcentration in the reboiler. The pH of the glycol will be on the acidic side and should be brought between 7.0 and 8.5 by the addition of a base (alkanolamines, borax,  $K_2HPO_4$  and name-brand inhibitors are readily available for this purpose).

Vacuum reclaimers are obtainable from several manufacturers and can be operated either in line or as batch reclaimers depending on the severity of the contamination and the volume of glycol to be purified. There are as well commercial firms that specialize in the reclamation of industrial wastes such as glycol.

Ion exchange      Strong acid/strong base ion exchange resins have been utilized (29) to effect essentially complete removal of NaCl from a glycol solution containing 1.0 % wt of this salt. The mechanism is illustrated in Figure 21 and the simplified flow diagram in Figure 22. In the first bed (A) sodium ions are exchanged from hydrogen ions to form hydrogen chloride. The second resins (B) then exchanges the chloride ion and neutralizes the acid. The beds are regenerated for 1 hr in a 24-hour cycle by the addition of HCl and caustic to A and B, respectively. This system was designed to purify a 10 gpm stream of glycol in a plant which processed 100 MMSCFD of raw gas.

Ion Retardation      This technique, described by Simpson and Bauman (30) is based on the difference in rate of flow through an ion-exchange resin of non-ionic solvent such as glycol containing an ionizable salt ( $NaCl$  and/or  $CaCl_2$ ) and the ionizable salt itself. A resin of special porosity is utilized in a column operation in which the contaminated glycol flows through the voids of the bed and also through the ion exchange beads. The ionic substance will flow only through the voids of the bed since the charge on the ion exchange sites will match that of the ionized salt. No net ion exchange takes place. This process, although promising to effect a successful purification of the glycol, has not yet been commercialized.

Fire Tube Clean-up      The removal of salt from a fire tube is usually not accomplished by pumping water into the boiler to dissolve the salt

during a turn-around situation. The reason for this is deposition of hydrocarbon or coke laid down with the salt makes the salt fairly well insulated from the water and this requires a mechanical cleaning job. A cleaning solution could contain water and a detergent type material for removal of the hydrocarbon and coke but this should be used with great care since any residual detergent in the glycol reboiler will cause foaming problems for many days to come. Water wash in conjunction with mechanical means are usually adequate for fire tube clean up.

Hydrocarbons      The contamination of the glycol solution by hydrocarbons, well treating compounds, dust, dirt and mill scale are all undesirable but are not always detrimental. The dehydration capabilities of the glycol solution depends upon the aqueous partial pressure of water above the solution at the conditions of the top tray of the absorber; the dilution of the glycol solution by contaminants such as hydrocarbon even to the extent of 2 or 4% will not in itself depress the dehydration capabilities of the system.

A more serious effect of a contaminant, such as hydrocarbon, is its coating of contact surfaces produces less efficient contact which will be reflected in a slightly higher dew point than that obtained under normal conditions. Although hydrocarbons will usually be regenerated with the water vapor in the still column, heavier ends will gradually accumulate in the solution. Some of these will be gradually coked and deposited on the fire tube. A gas field producing a fair amount of these heavy hydrocarbons that override the gas scrubbers can present a serious problem in the reboiler. Coking of hydrocarbons on the fire tube will cause hot spots and can result in a fire burnout, or loss of heat transfer efficiency.

Most hydrocarbon problems are eliminated by proper selection of inlet separators/scrubbers for the gas, installation of an oil skimmer in conjunction with the flash tank (3 phase separator) and filtration of the rich, hot glycol before it reaches the regenerator.

Foam      The presence of salt in the glycol solution is many times indirectly related to both foaming and corrosion. Occasionally foaming is observed when the salt concentration is on the increase due to well treating compounds containing surfactant materials and not salt alone. In controlled laboratory experiments, a clean glycol solution and either sodium chloride or calcium chloride will not produce a foam condition greater than that of the control. Likewise, salt added to the glycol solution will not in itself contribute to an increase in corrosion. However, a small amount of oxygen will greatly accelerate corrosion in the presence of salt.

The deposition of salt on a fire tube may result in corrosion at the border of the deposition. This is due more to a potential difference between the surrounding metal and the salt deposit than it is the action of the salt itself.

Triethylene glycol will not exhibit a high degree of foam if it is kept free of surfactant type materials. These materials may be introduced through compressor oil, plugcock lubricant, and corrosion inhibitors used in either the formation or in the gas gathering system. The absorption of an appreciable quantity of natural gas in the glycol solution will result in the manifestation of foaming conditions in the glycol reboiler and still column. This usually occurs when the glycol is operated at contact temperatures less than 80°F where higher than normal quantities of natural gas are absorbed and viscosity of the glycol is increased above 30 cps.

Filtration      The removal of solid contaminants can be accomplished by adequate filtration on the rich circuit. This should be done at the highest temperature point before discharging the glycol into the still column in order to take advantage of the lower viscosity.

If appreciable quantities of natural gas are absorbed in the contactor, a flash tank should be provided down stream of the glycol-glycol heat exchanger. The vent gases from the flash tank may be sent to flare or utilized for gas stripping or for plant fuel in the event that a low pressure drop is not taken at the flash tank.

Contaminants such as hydrocarbon and surfactants may be adequately removed on activated carbon filters. The activated carbon filter is usually operated on a 10% side stream with activated carbon particles in the 4-20 mesh range. The flow rate is usually designed to give flows of 1-5 gallon/minute/ft<sup>2</sup> of cross sectional area.

Thermal degradation of the glycol as a result of excessive skin temperatures or hot spots on the fire tube is manifested by a burnt sugar odor of the glycol, a lowering of the pH and a black color. Activated carbon is generally not successful in cleaning up the thermally degraded glycol solution completely, but will give a muddy yellow color to the effluent. In general, the degradation products are water soluble and will not seriously affect the performance of the glycol unit; however, close control of pH should be maintained on any system that regularly produces thermally degraded glycol contaminants. Antifoaming agents should be maintained on hand at any glycol dehydrator. A silicone type antifoam agent is usually successful in the 25-150 ppm range and good results have been obtained with the proprietary compound, Corexit 7669.

## CONCLUSIONS

The family of ethylene glycols has been found to be an excellent medium for the dehydration of natural gas by removing the entrained water vapor in it. Specifically triethylene glycol has emerged as probably the most suitable of the glycols for this purpose. It has gained widespread acceptance and is used throughout the natural gas industry for the dehydration of natural gas.

The procedures and descriptions contained in this technical publication may be used to design the major components of a typical triethylene glycol dehydration system, as well as evaluate the performance of existing equipment. Also, numerous chemical procedures are described so that the engineer and/or operator can maintain the quality of the glycol solution in the dehydration unit. Operational problems and solutions have been discussed so that the operator may be forewarned and through proper maintenance may prevent malfunction, and ensure proper operation of his dehydration equipment.

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## APPENDICES

Appendix A	Design Example	A-1 thru A-4
Appendix B	Drawings, Graphs, and Tables	B-0 thru B-34
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APPENDIX A  
DESIGN EXAMPLE

GAS CONVERSION EQUATIONS

From the basic laws the following useful conversion equations can be derived.

At Standard Conditions of 14.7 psia and 60° F:

$$\text{Molecular weight of gas} = 28.97 \text{ (sp gr)}$$

$$\text{Density of gas, lb/cu ft} = 0.0764 \text{ (sp gr)} = \frac{\text{mol wt}}{379} = \frac{28.97 \text{ (sp gr)}}{379}$$

$$\text{Specific volume of gas, cu ft/lb} = \frac{13.08}{\text{sp gr}} = \frac{379}{\text{mol wt}}$$

$$\text{Gas flow, mol/day} = \frac{\text{Gas flow, cu ft/day}}{379}$$

$$\text{Mass flow rate, lb/hr} = 3185 \text{ (MMSCFD)} \text{ (sp gr)}$$

At Conditions Other Than Standard:

$$\text{Density of ideal gas, lb/cu ft} = \frac{2.703 \text{ (sp gr)}(\text{Pressure, psia})}{(\text{Temp, F} + 460)}$$

$$\text{Density of actual gas, lb/cu ft} = \frac{2.703 \text{ (sp gr)}(\text{Pressure, psia})}{(\text{Temp, F} + 460)(Z)}$$

$$\text{Ideal gas flow, cu ft/day} = \frac{(\text{Gas flow, SCFD})(14.7)(\text{Temp F} + 460)}{(\text{Pressure, psia})(520)}$$

$$\text{Actual gas flow, cu ft/day} = \frac{(\text{Gas flow, SCFD})(14.7)(\text{Temp, F} + 460)(Z)}{(\text{Pressure, psia})(520)}$$

$$\text{Actual gas flow, cu ft/sec} = \frac{0.327 \text{ (MMSCFD)}(\text{Temp, F} + 460)(Z)}{(\text{Pressure, psia})}$$

$$\text{Volume of mol, cu ft/mol} = \frac{379 (\text{Temp, F} + 460)(14.7)}{(520)(\text{Pressure, psia})}$$

Where: Z = Compressibility factor

EXAMPLE PROBLEM - DEHYDRATOR DESIGN

Size a glycol dehydrator for a field installation from standard models to meet the following requirements.

1. Gas flow rate: 10.0 MMSCFD
2. Gas specific gravity: 0.70
3. Operating line pressure: 1000 psig
4. Maximum working pressure of contactor: 1440 psig
5. Gas inlet temperature: 100° F
6. Outlet gas water content: 7 lb H<sub>2</sub>O/MMSCF

Select additional design criteria:

1. Glycol to water circulation rate: 3.0 gal TEG/lb H<sub>2</sub>O
2. Lean glycol concentration: 99.5% TEG
3. Use trayed type contactor with valve trays

Contactor Size:

From Figure No. 5 select a contactor diameter with the approximate gas capacity at operating pressure.

$$G_s \text{ for 24" O.D. contactor at 1000 psig} = 11.3 \text{ MMSCFD}$$

Correct for operating conditions from Tables No. 1A and 1B

$$G_s = G_s (C_t)(C_g)$$

$$G_o = 11.3 (1.0) (1.0) = 11.3 \text{ MMSCFD}$$

Required Dew Point Depression and Water Removed:

From Water Content Chart at 1000 psig

	<u>Dew Pt Temp</u>	<u>Water Content lb H<sub>2</sub>O/MMCF</u>
Inlet	110° F	61
Outlet	33° F	7
	<u>67° F</u>	<u>54 lb H<sub>2</sub>O/MMCF</u>

Number of Trays Required:

From Figure No. 7 at 3 gal. TEG/lb H<sub>2</sub>O and 67° F dew point depression, No. actual trays = 4.5

For a more detailed study, construct a modified McCabe-Thiele diagram.

$$\begin{aligned} \text{Density of lean glycol at } 100^{\circ} \text{ F, } \rho_L &= (\text{Sp gr})(8.34) \\ &= (1.111)(8.34) \\ &= 9.266 \text{ lb/gal.} \end{aligned}$$

$$\begin{aligned} \text{Rich TEG} &= \frac{(\text{Lean TEG})(\rho_L)}{\rho_L + \frac{1}{L_w}} \\ &= \frac{(0.995)(9.266)}{9.266 + \frac{1}{3.0}} = 0.960 = 96.0 \end{aligned}$$

Operating line points:

Top of Column: 7.0 lb H<sub>2</sub>O/MMCF & 99.5% TEG  
 Bottom of Column: 61 lb H<sub>2</sub>O/MMCF & 96.0% TEG

Equilibrium line points:

<u>% TEG</u>	<u>Equilibrium Dew Point Temp. at 100° F</u>	<u>Water Content of Gas at Dew Point Temp. &amp; 1000 psig</u>
99	12	3.2 lb H <sub>2</sub> O/MMCF
98	30	6.3
97	40	9.0
96	47	11.7
95	51	13.3

Construct a McCabe-Thiele diagram and determine the number of theoretical trays required. See Figure No. 9.

$$\begin{aligned} \text{No. Actual Trays} &= \frac{\text{No. Theo. Trays}}{\text{Tray Eff.}} \\ &= \frac{1.48}{0.333} = 4.44 \end{aligned}$$

The results from the McCabe-Thiele diagram are to close to that determined from the approximation curve, Figure No. 7. In either case the next whole number of trays should be used.

No. Actual Trays Required = 5

Reconcentrator:

Determine the required glycol circulation rate.

$$\begin{aligned} L &= \frac{L_w(W_i)(G)}{24} \\ &= \frac{3.0(61)(10.0)}{24} = 76.25 \text{ gal/hr} \end{aligned}$$

## Reboiler Duty:

Determine the heat load required.

$$\begin{aligned} \text{Approximate heat load, } Q_t &= 2000 \text{ (L)} \\ &= 2000 (76.25) = 152,500 \text{ BTU/hr} \end{aligned}$$

Detailed calculation of heat required:

$$\begin{aligned} Q_1 &= L (1200) &= 76.24 (1200) &= 91,488 \\ Q_w &= \frac{970.3 (W_i - W_o) (G)}{24} &= \frac{970.3 (61.7) (10)}{24} &= 21,832 \\ \\ Q_r &= 0.25 (Q_w) = 0.25 (21,832) = && 5,458 \\ Q_1 &= 10,000 &= & \underline{10,000} \\ Q_t &= && 128,778 \text{ BTU/hr} \end{aligned}$$

## Stripping Still:

Determine the diameter of the packed column required from Figure 10 based on:

$$\begin{aligned} L &= 76.25 \text{ gal/hr} \\ L_w &= 3.0 \text{ gal. TEG/lb H}_2\text{O} \\ A &= 24 \text{ sq in.} \end{aligned}$$

$$\text{Minimum I.D.} = 5.5 \text{ in.}$$

Standard Size Unit Required:

Summary of requirements:

Glycol-Gas Contactor: 23" O.D. with 5 trays, 1440 psi W.P.  
Glycol Pump: 76.25 gal/hr  
Reboiler: 128,773 BTU/hr  
Stripping Still: 5.5 in. I.D.

Standard Size Unit:

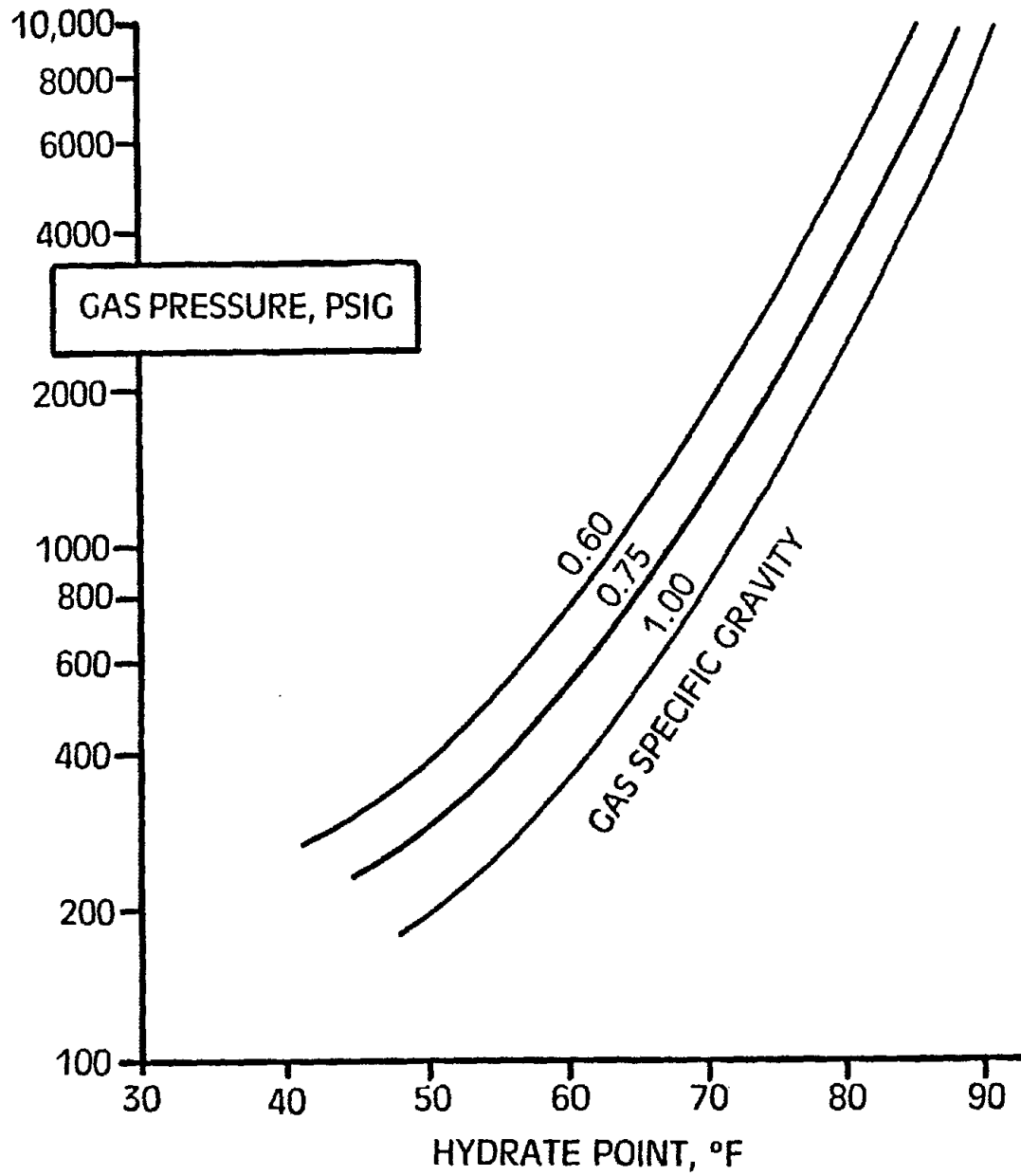
Contactor: 24" O.D. x 14', 1440 psi W.P. with 5 valve trays  
Glycol Reconcentrator: 175,000 BTU/hr reboiler, 9015-PV pump,  
8-5/8" O.D. x 4'-6" stripping still

## APPENDIX B

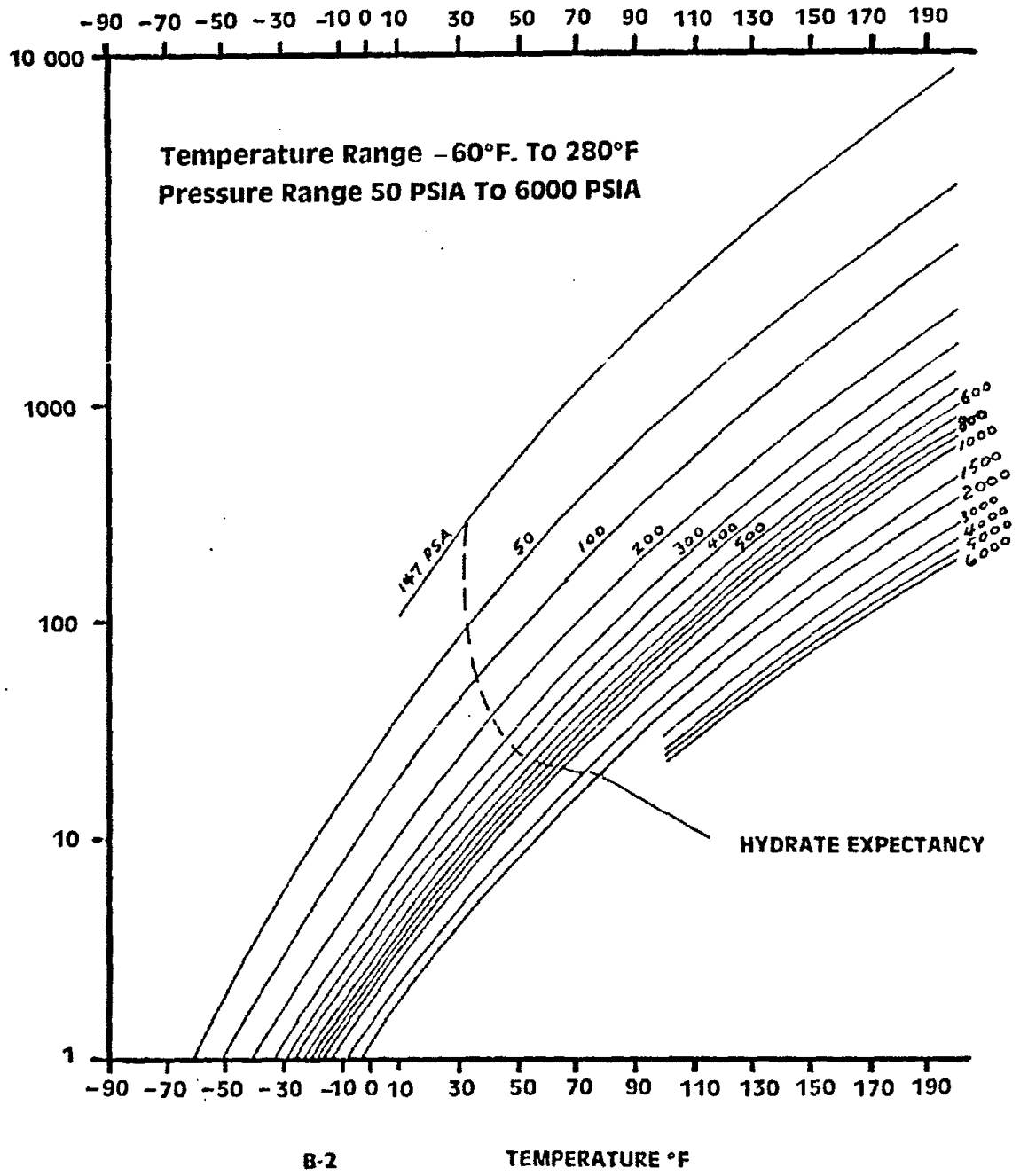
## FIGURES AND TABLES

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**Figure 1**  
**Conditions For Hydrate Formation of Natural Gas**



# Water Vapor Content of Natural Gas at Saturation

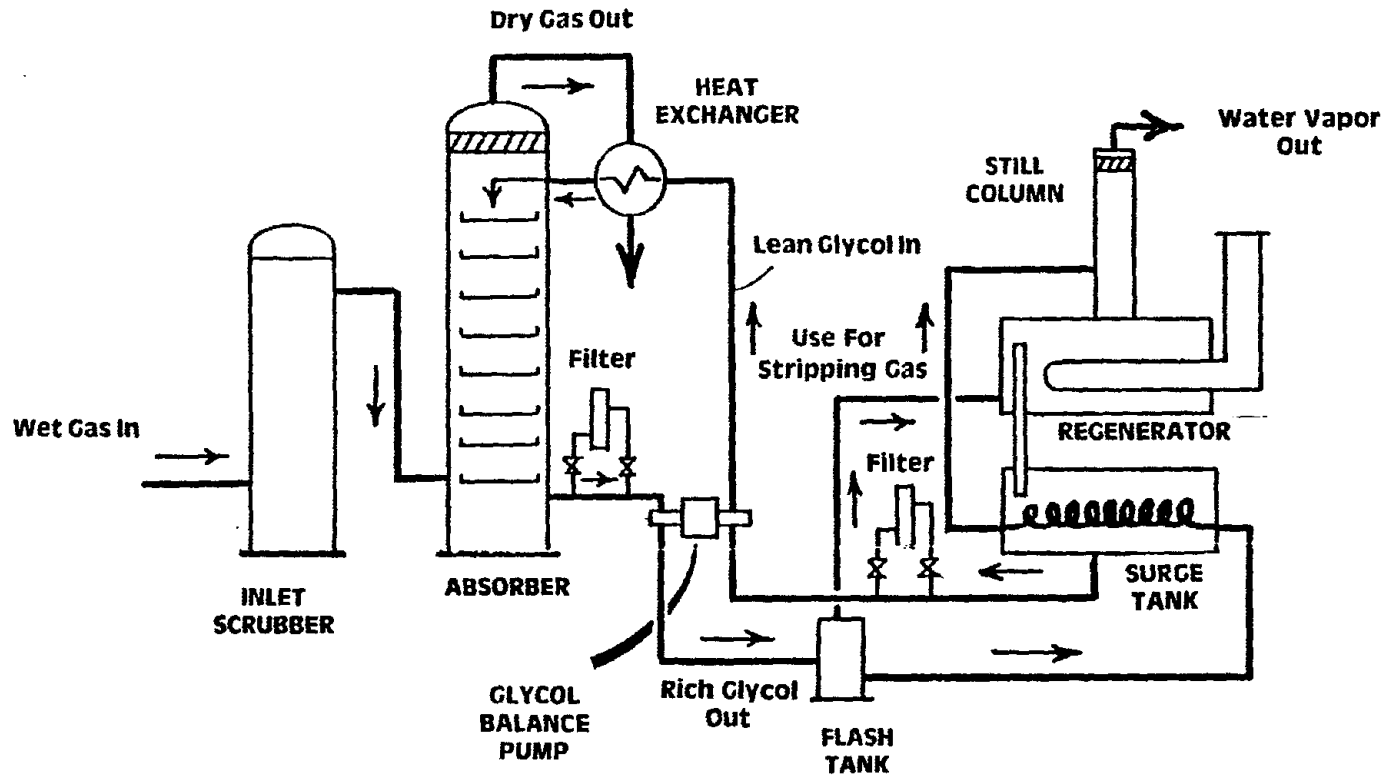


WATER CONTENT,  
LBS. PER MM CUBIC FEET  
AT 14.7 PSIA AND 60°F.

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Figure 3

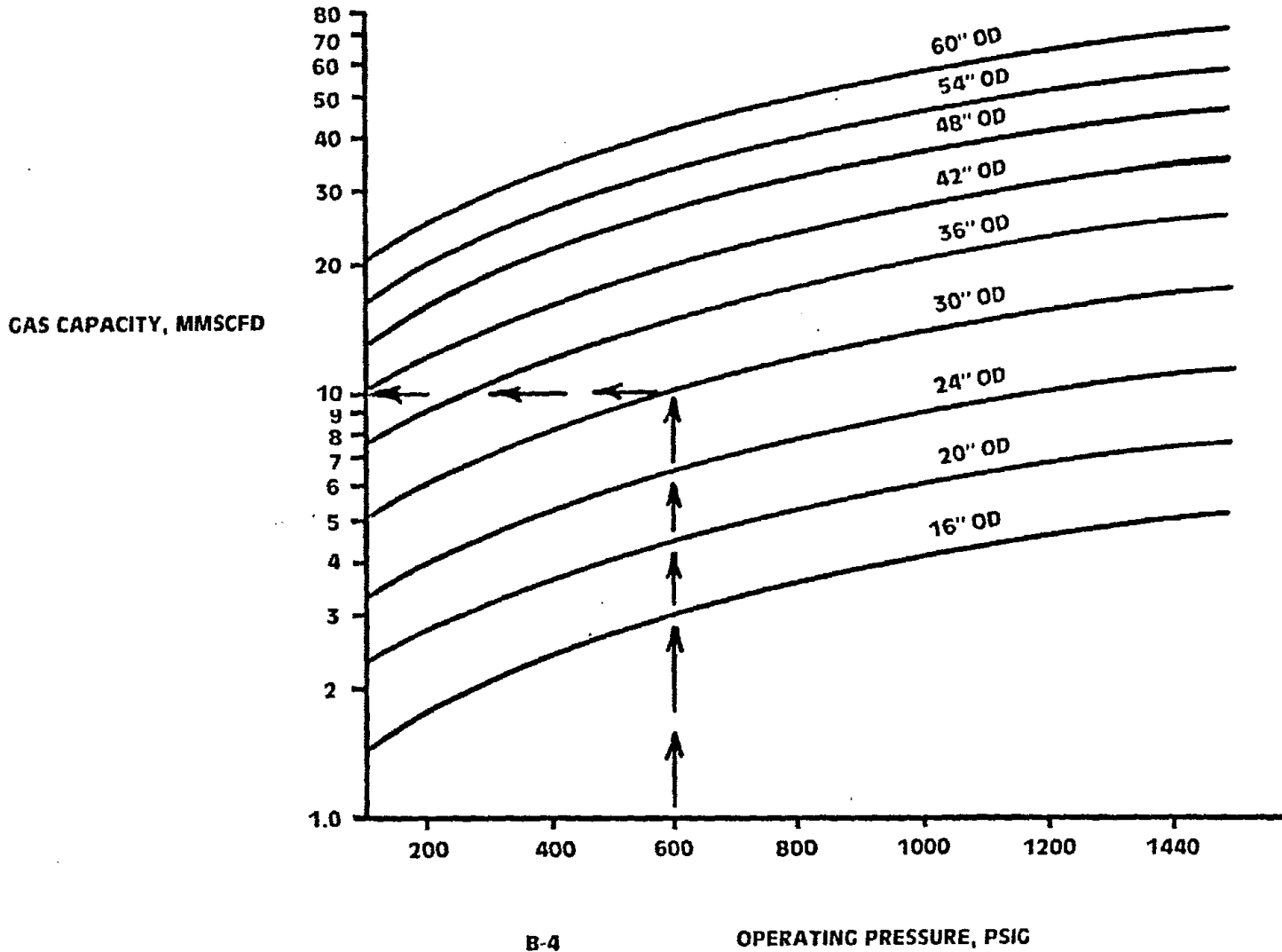
Flow Diagram Typical Glycol Dehydrator



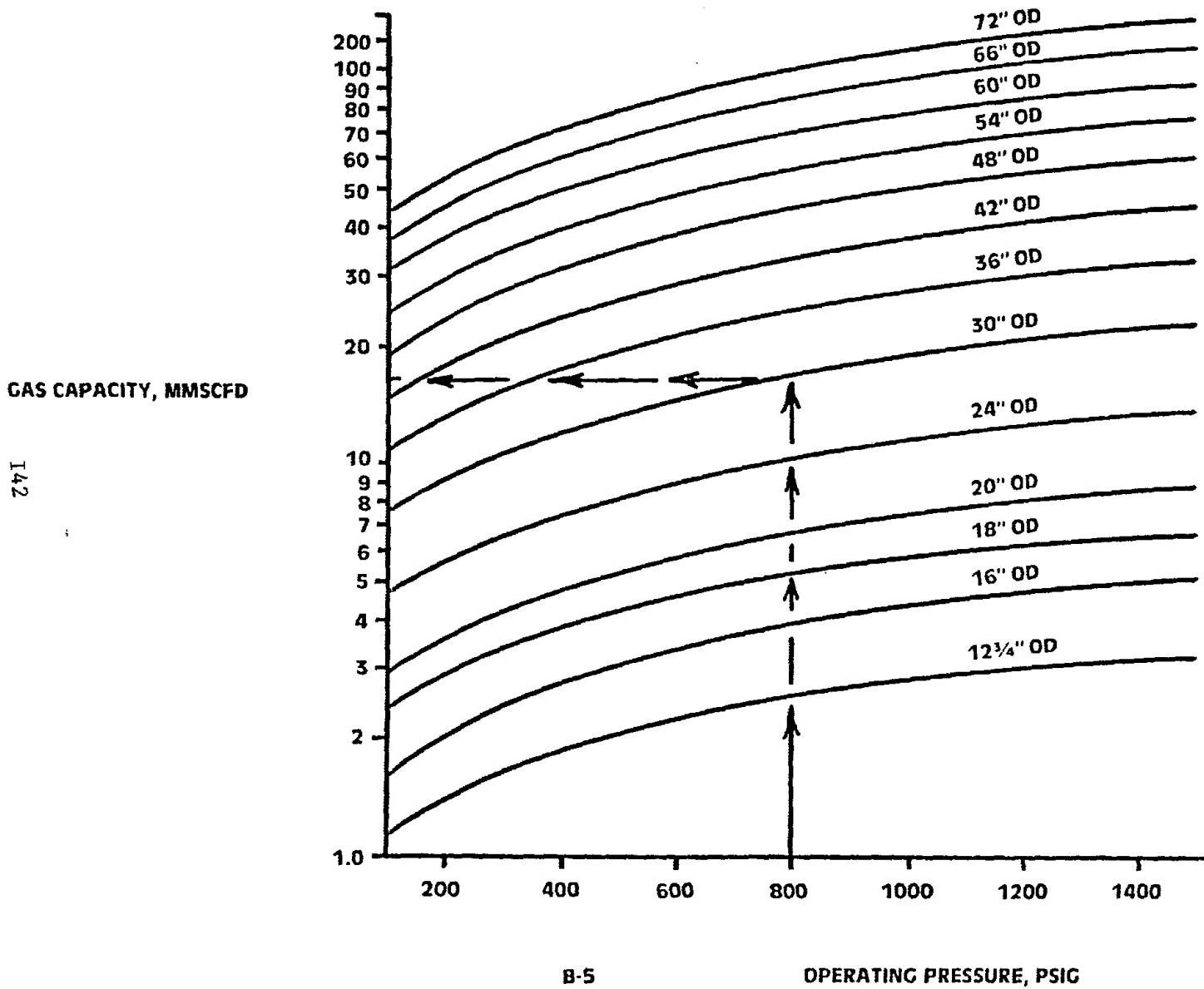
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**Figure 4**  
**Gas Capacity of Vertical Gas Inlet Scrubbers**  
**Based on 0.7 SP GR 100°F**



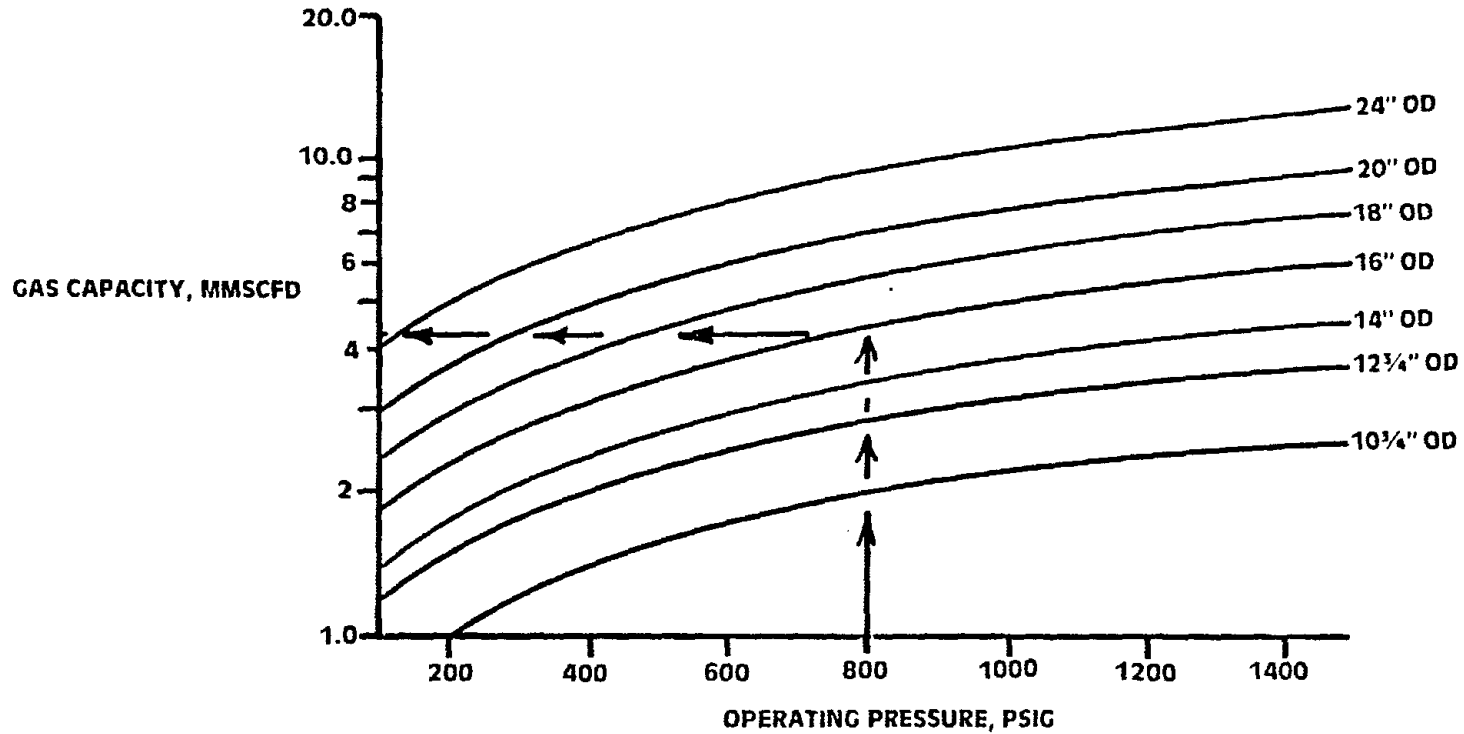
**Figure 5**  
**Gas Capacity For Trayed Glycol—Gas Contactors**  
**Based on 0.70 SP GR 100°F**



GAS CAPACITY, MMSCFD

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**Figure 6**  
**Gas Capacity For Packed Glycol—Gas Contactors**  
**Based on 0.70 SP GR 100°F**



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**Figure 7**  
**Trays Or Packing Required For Glycol Dehydrators**

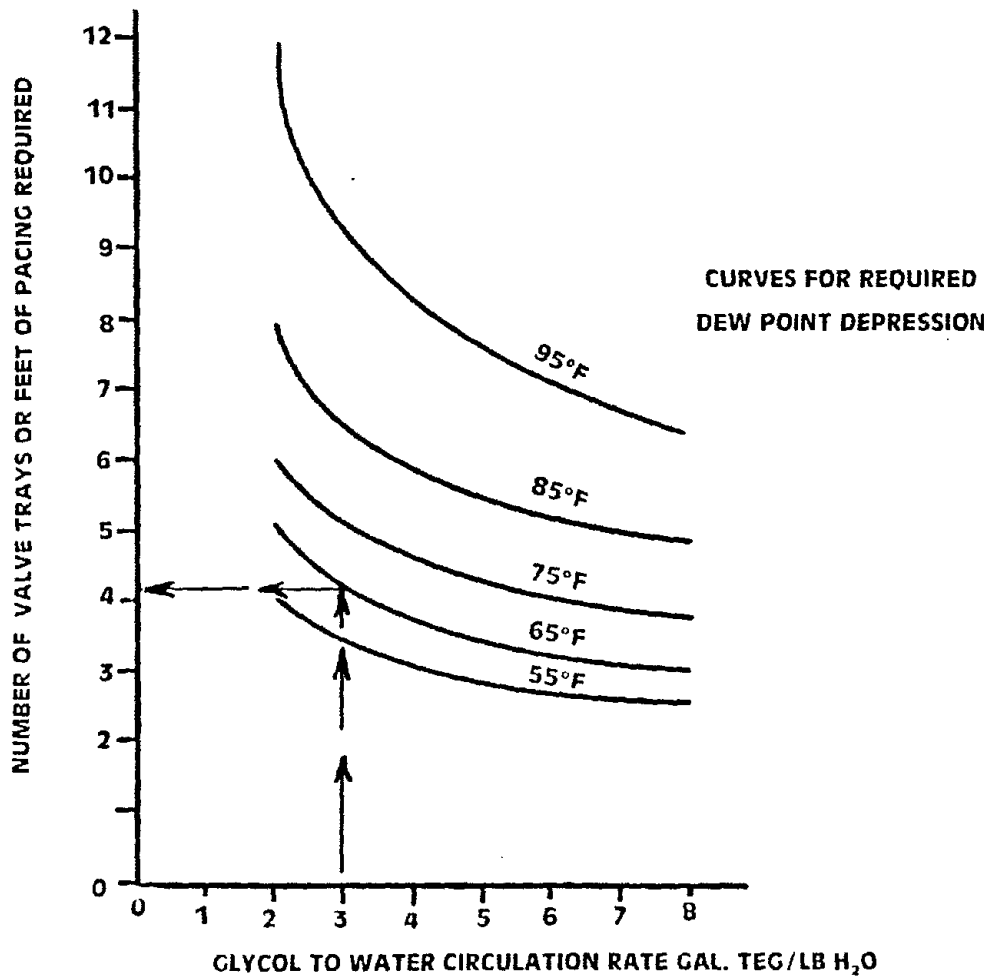


Figure 8

### Dew Points of Aqueous Triethylene Glycol Solutions Versus Temperature

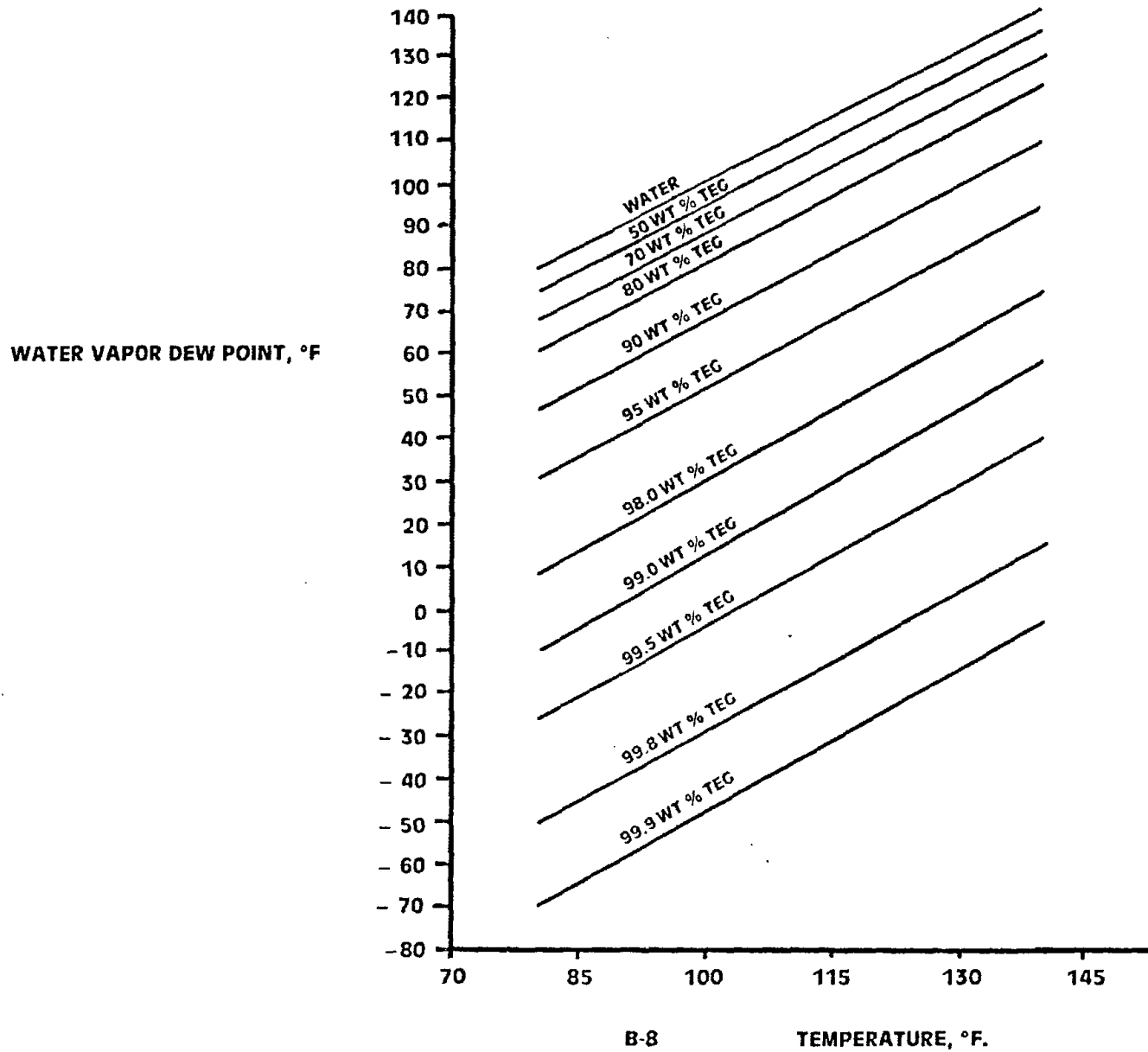


Figure 9  
Example of Modified McCabe-Thiele Diagram

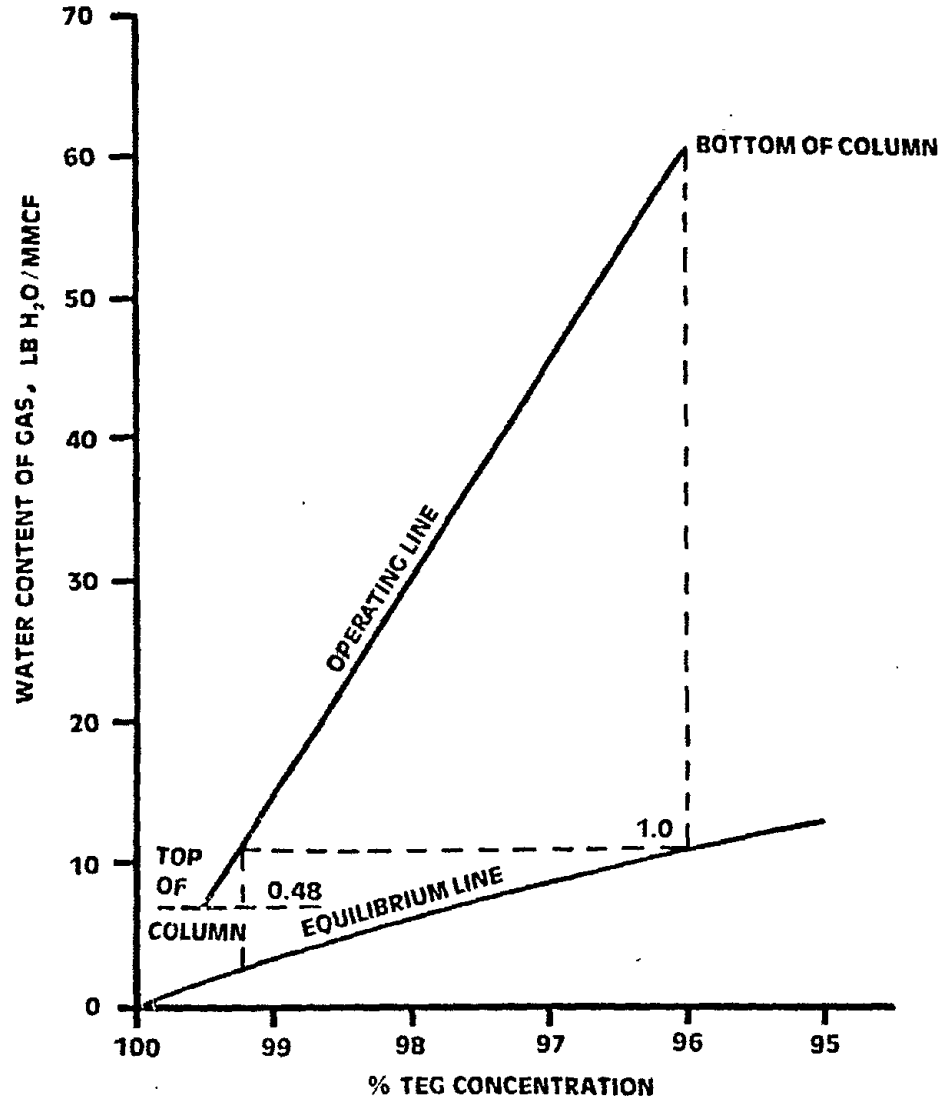
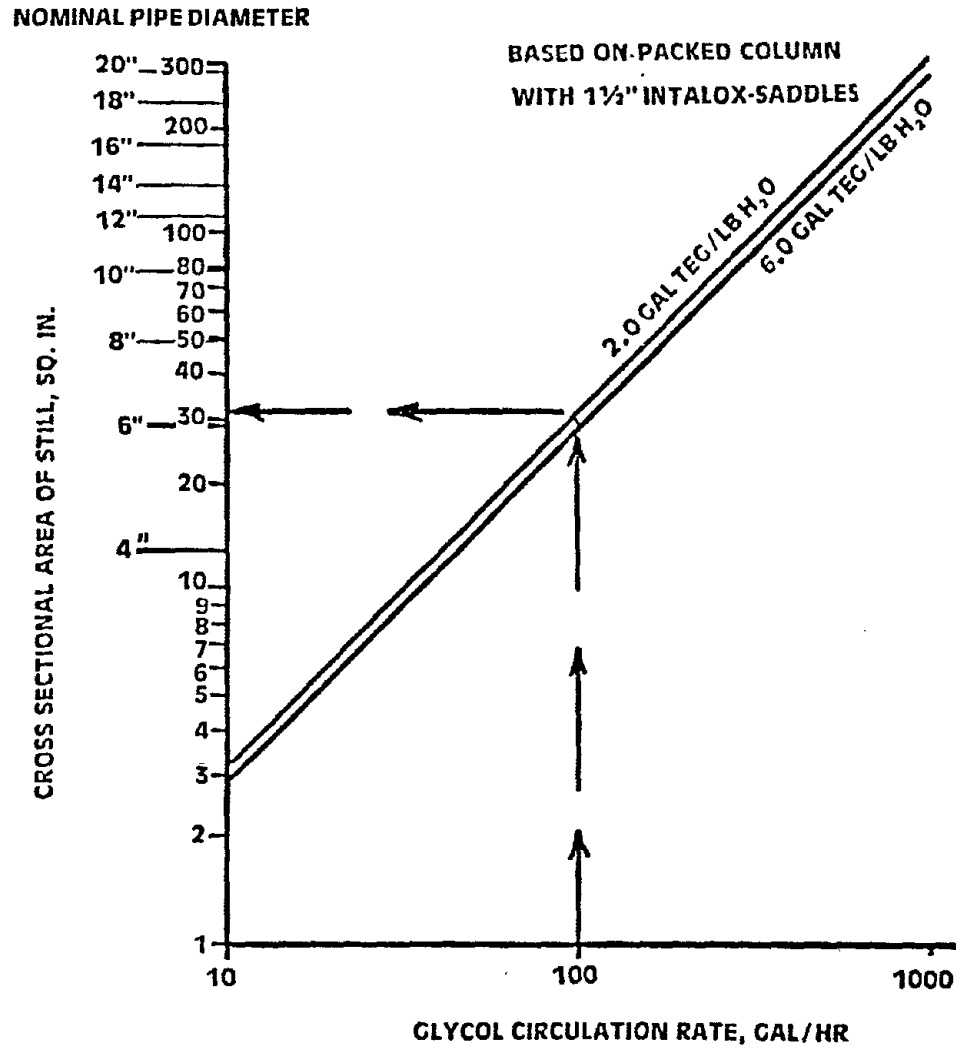


Figure 10  
Stripping Still Size For Glycol Dehydrators



## Figure 11. Typical Gas Chromatograph

### Column Conditions

---

Column: 4' x 2 mm i.d., glass Sample volume: 0.2 ul  
Packing: TENAX GC, 60/80 mesh. Carrier: Helium @  
30 ml/min

Oven Temp, Start: 100°C . . . . . End: 285°C, max.

Programming Rate: 15°C/min

Time held at max temp: 5 min

Injection port temp: 250°C

Thermal conductivity detector: 300°C

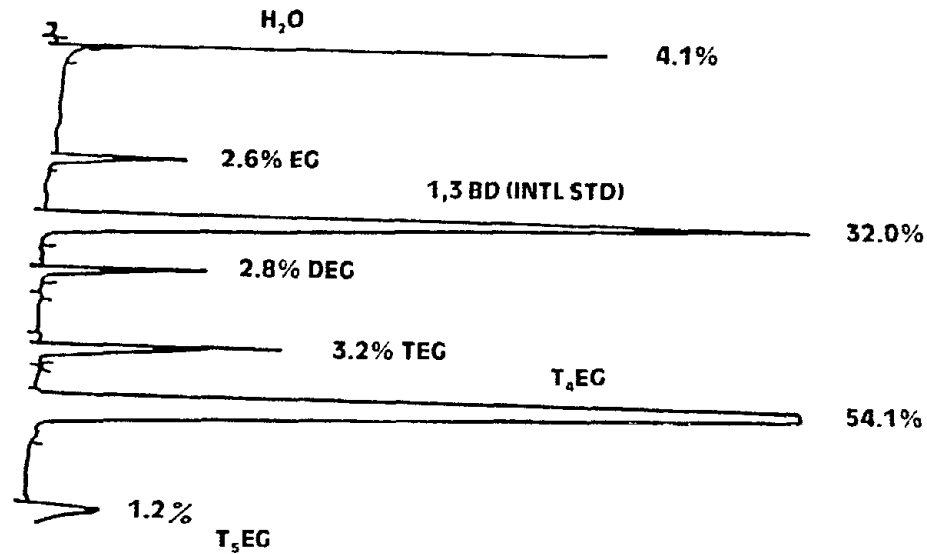
Attenuation: 2<sup>5</sup> Chart speed: 0.50 cm/min

Slope Sensitivity: 0.04 Dual columns



Figure 12

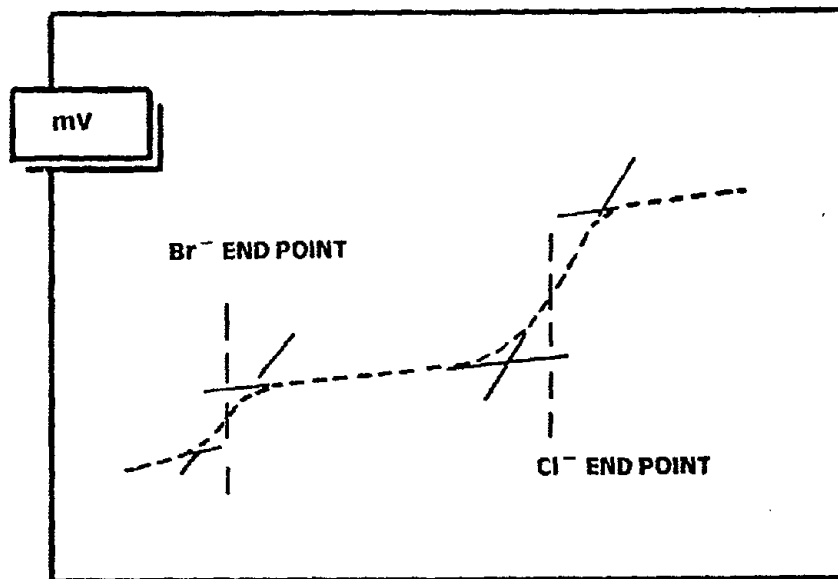
Typical Gas Chromatogram of Glycol Mixture  
with Tetraethylene Glycol as Main Component



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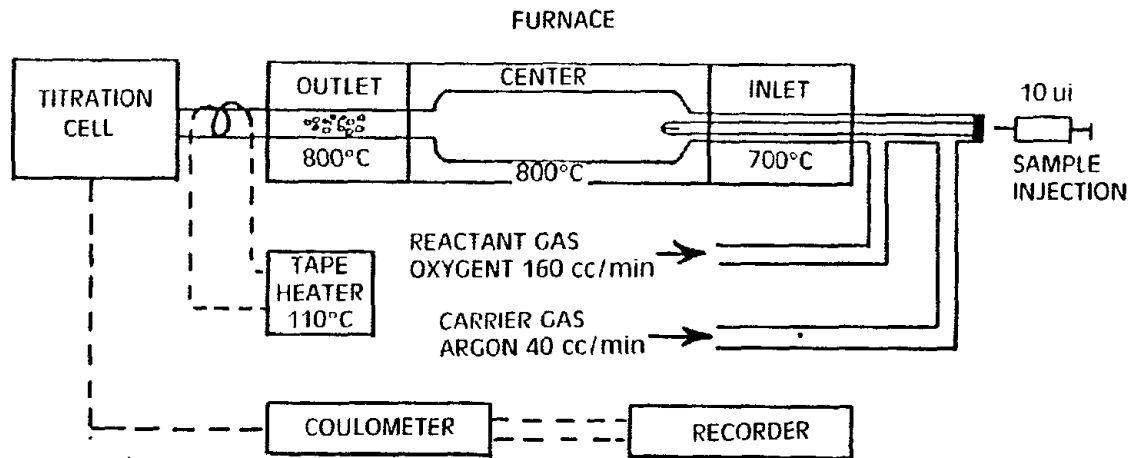
**Figure 13**  
**Potentiometric Titration of Halides**  
**With  $\text{AgNO}_3$  and Silver Electrode**



ML 0.1 N  $\text{AgNO}_3$

**B-13**

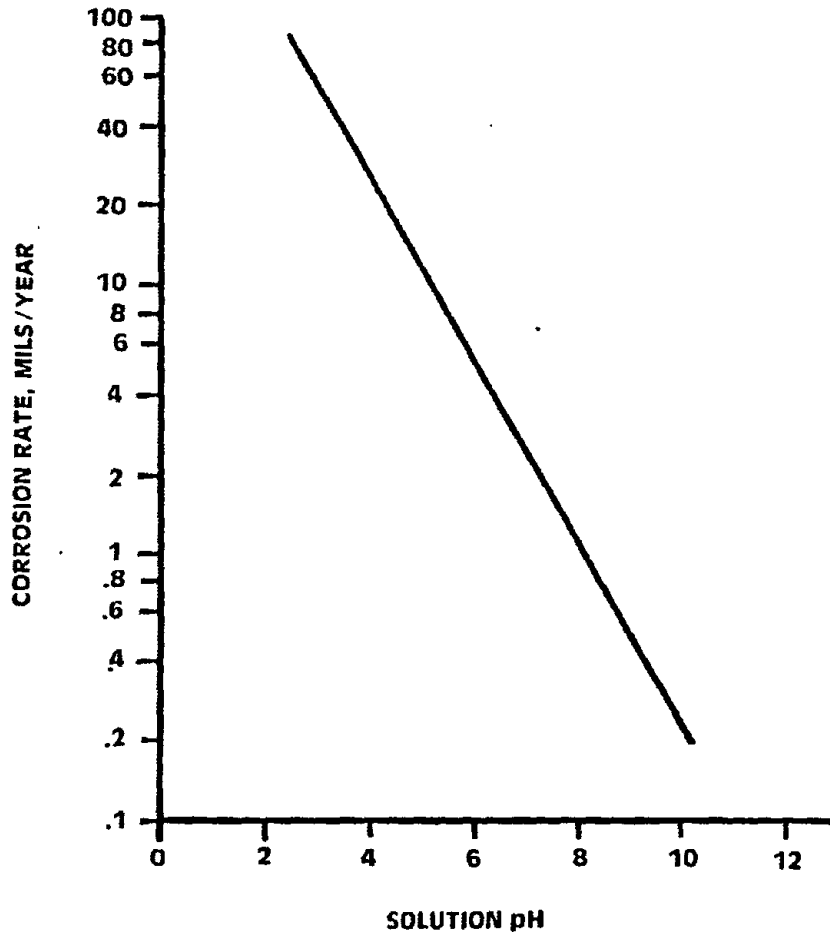
**Figure 14**  
**Apparatus For Total Chloride Analysis**



**B-14**

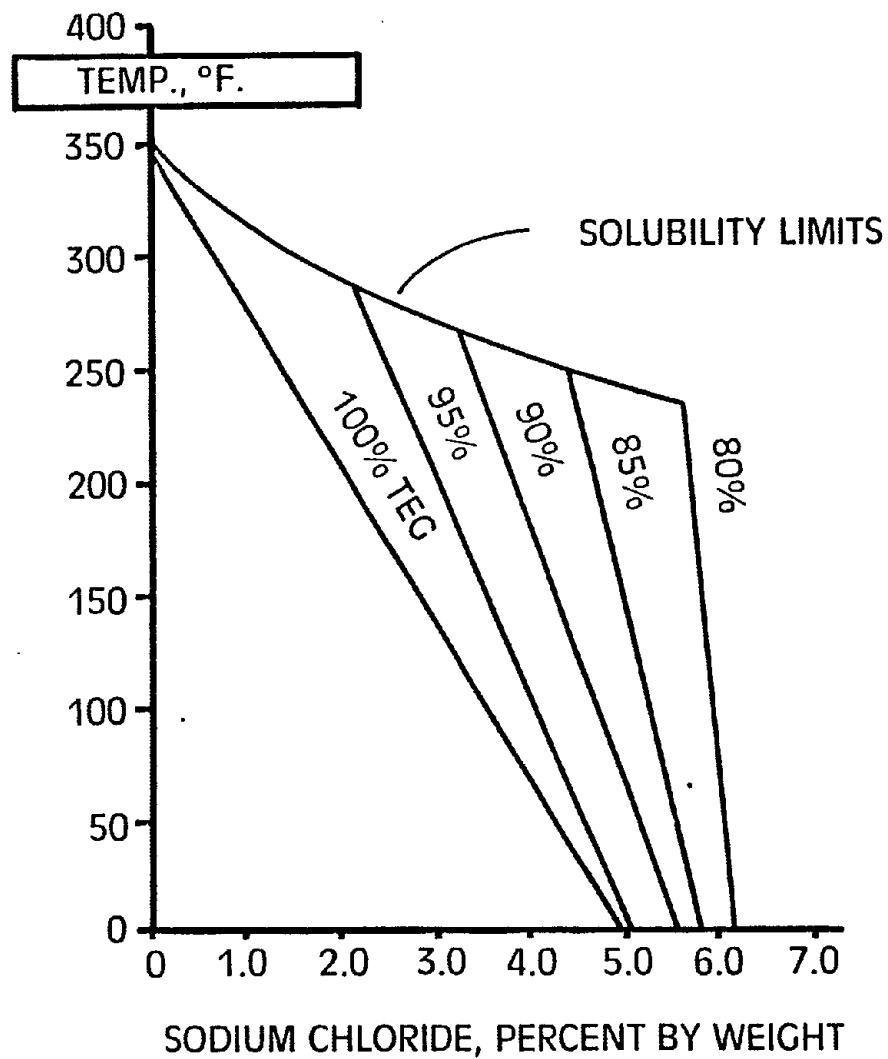
**Figure 15**

**Relationship Between Diethylene Glycol pH  
And Corrosion**

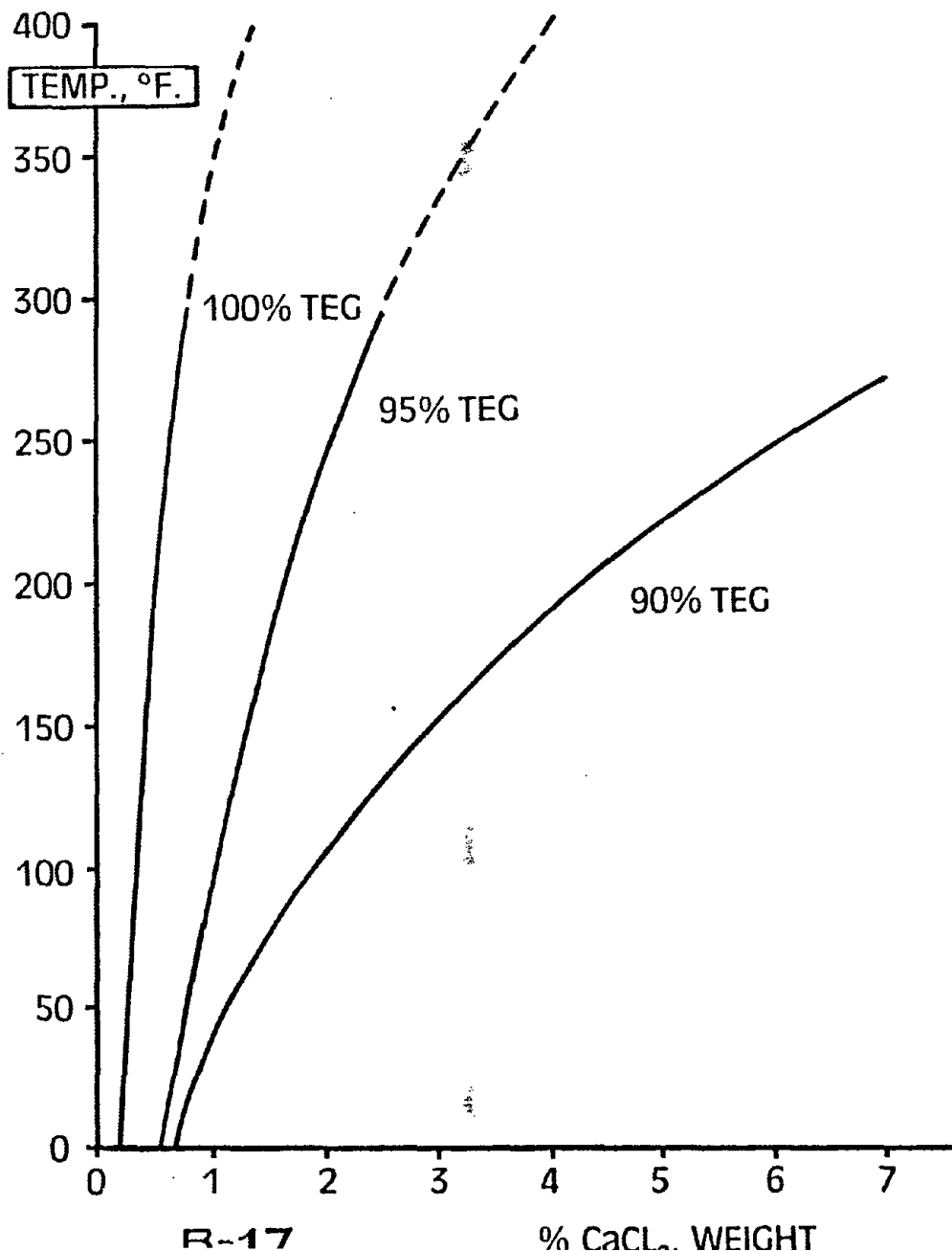


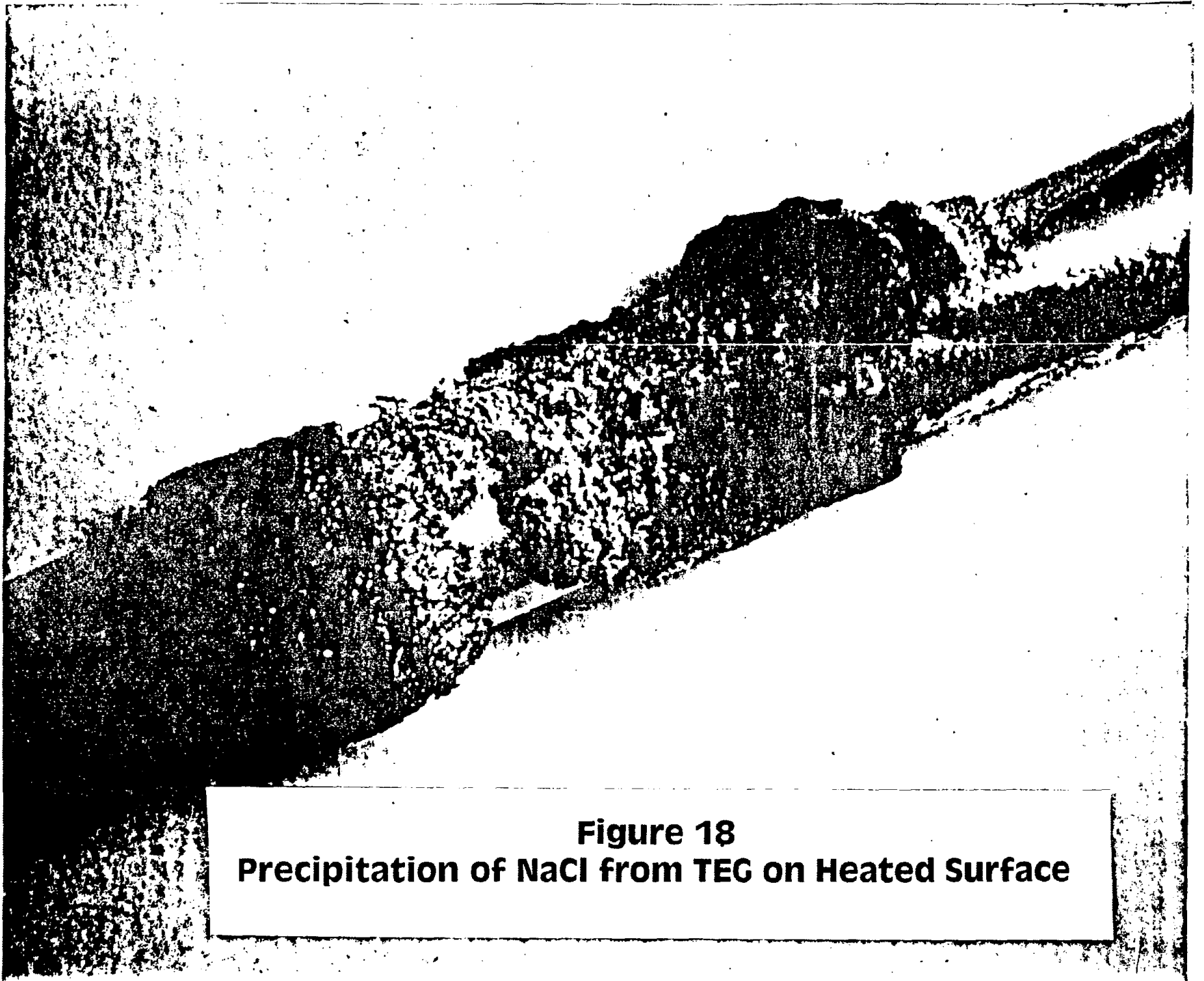
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Figure 16  
Solubility Of Sodium Chloride In Triethylene Glycol



**Figure 17**  
**CaCl<sub>2</sub> Solubility In Aqueous Triethylene Glycol**

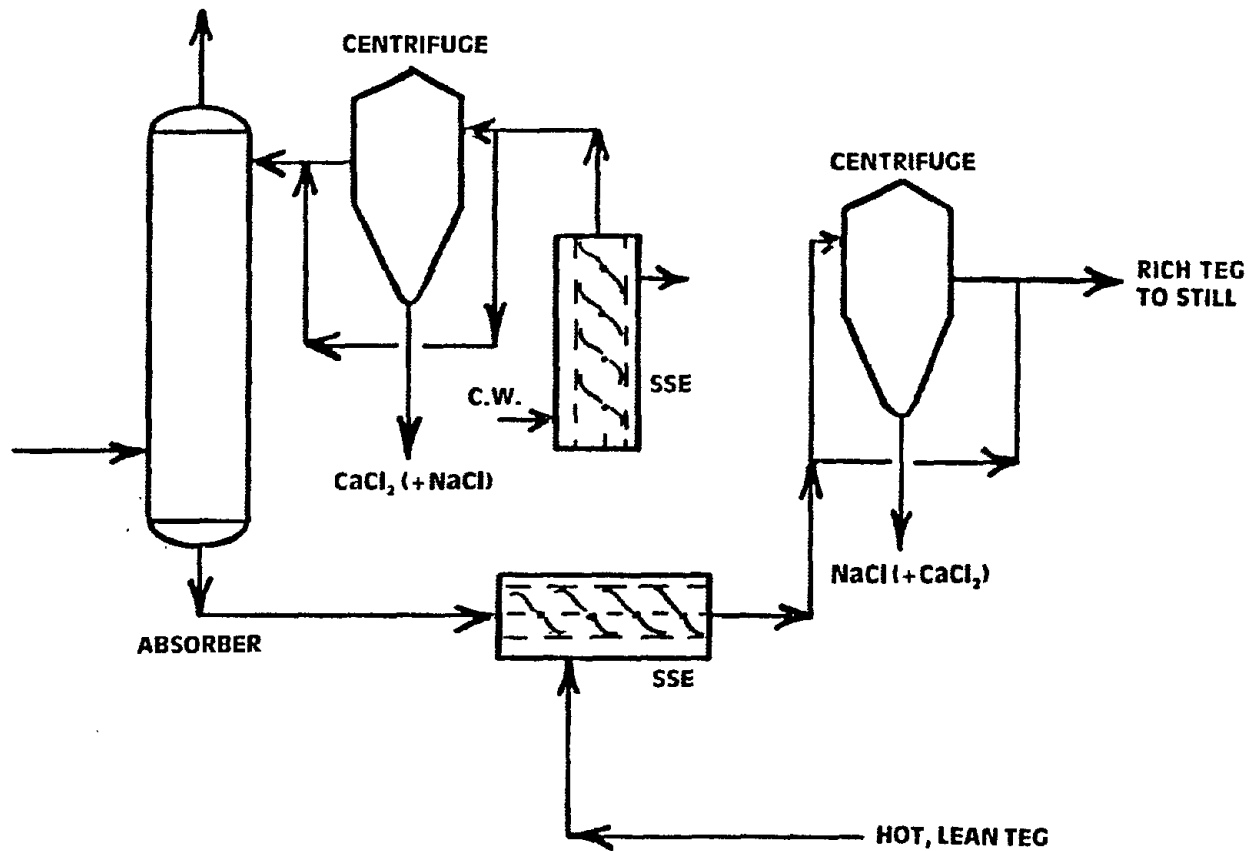




**Figure 18**  
**Precipitation of NaCl from TEG on Heated Surface**

Figure 19

Salt Removal from TEG  
by Scraped Surface Heat Exchangers

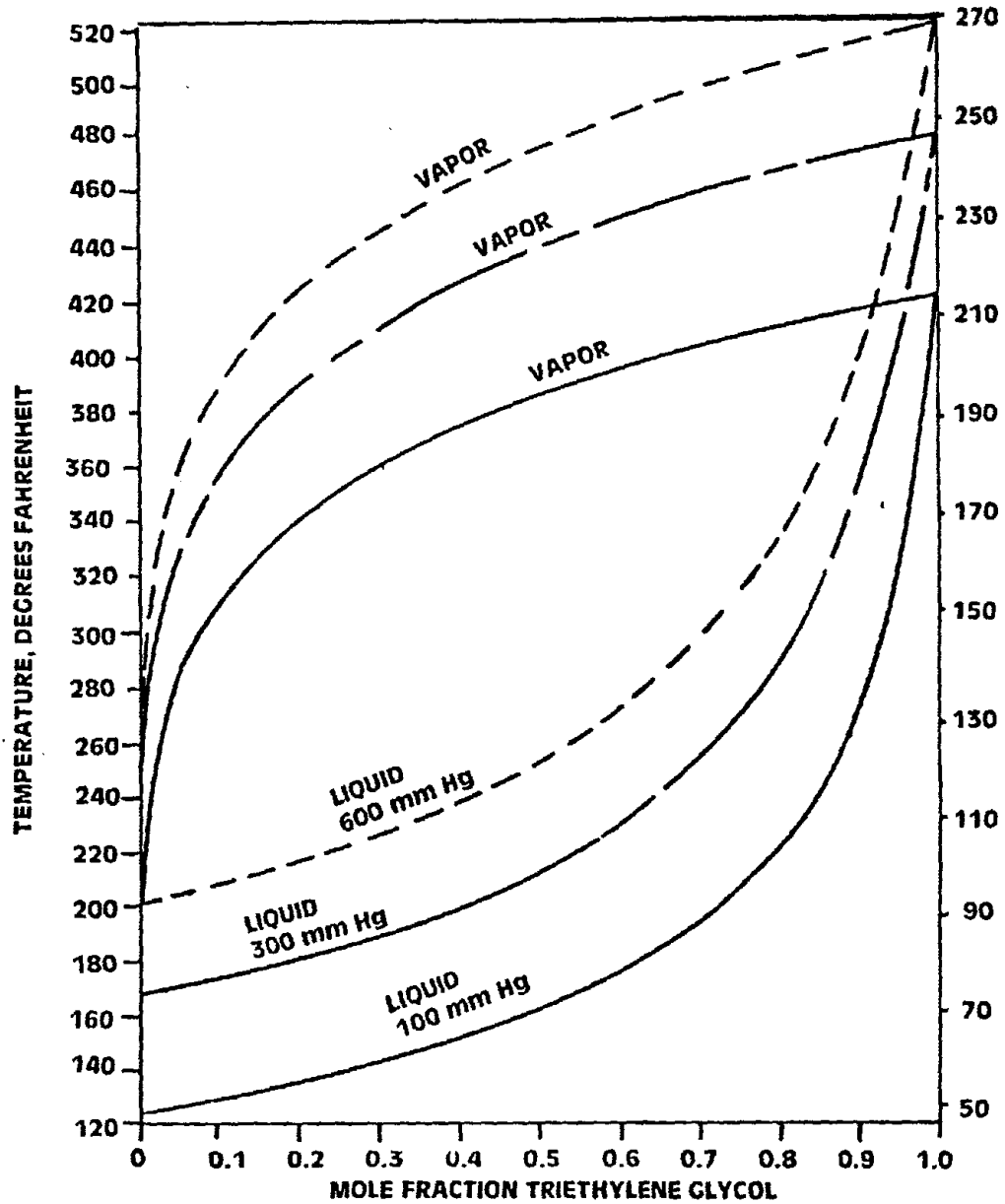


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Figure 20

### VAPOR-LIQUID COMPOSITION CURVES FOR AQUEOUS TRIETHYLENE GLYCOL SOLUTIONS



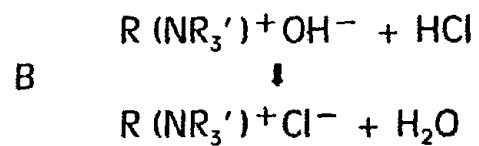
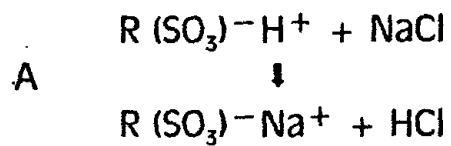
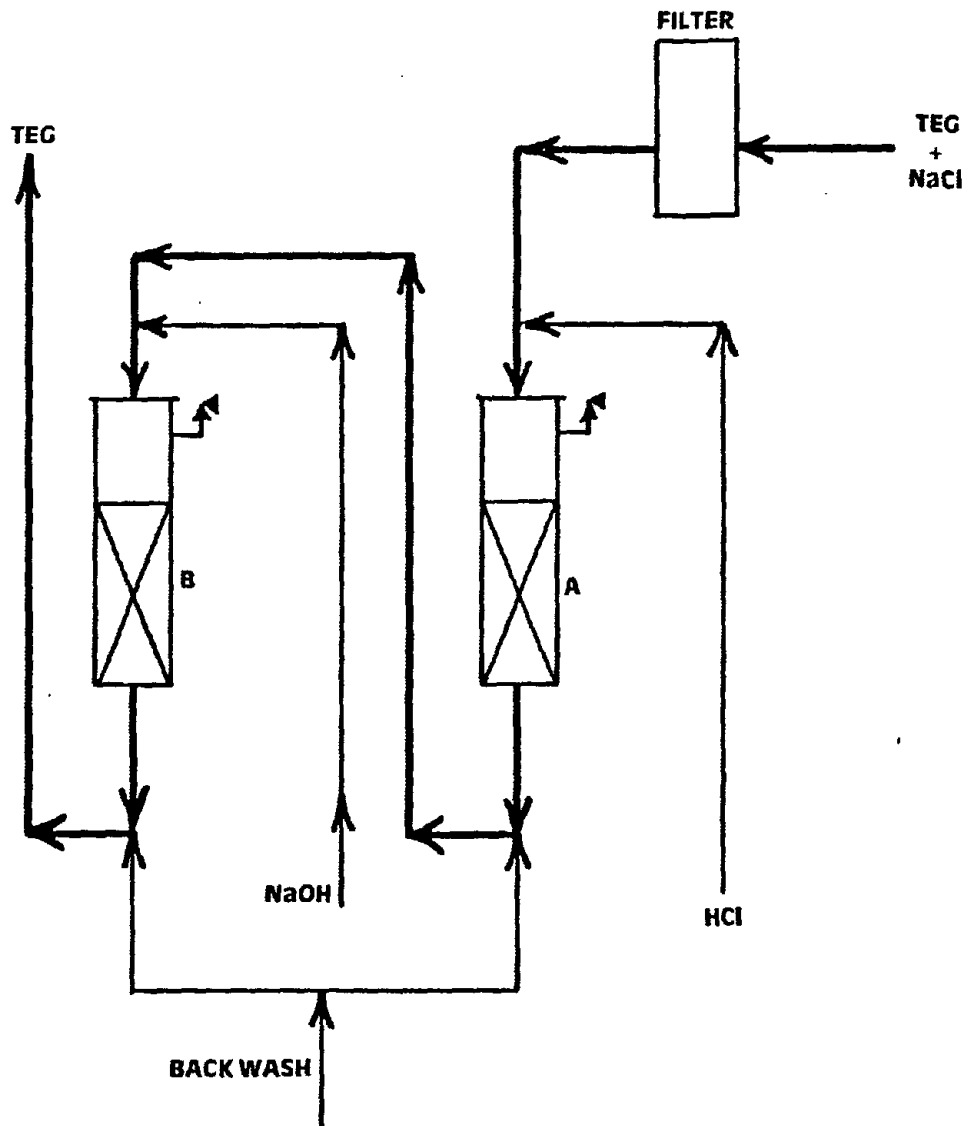


Figure 21. Ion Exchange Purification of Salt  
from Glycol Solutions

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**Figure 22**  
**Removal of NaCl from TEG by Ion Exchange**



GAS CAPACITY CORRECTION FACTORS FOR TRAYED  
GLYCOL-GAS CONTACTORS

TABLE 1A  
TEMPERATURE CORRECTION FACTORS,  $C_t$

Operating Temperature °F	Correction Factor $C_t$
40	1.07
50	1.06
60	1.05
70	1.04
80	1.02
90	1.01
100	1.00
110	0.99
120	0.98

TABLE 1B  
SPECIFIC GRAVITY CORRECTION FACTORS  $C_g$

Gas Specific Gravity	Correction Factor $C_g$
0.55	1.14
0.60	1.08
0.65	1.04
0.70	1.00
0.75	0.97
0.80	0.93
0.85	0.90
0.90	0.88

GAS CAPACITY CORRECTION FACTORS FOR PACKED  
GLYCOL-GAS CONTACTORS

TABLE 2A  
TEMPERATURE CORRECTION FACTORS,  $C_t$

Operating Temperature °F	Correction Factor $C_t$
50	0.93
60	0.94
70	0.96
80	0.97
90	0.99
100	1.00
110	1.01
120	1.02

TABLE 2B  
SPECIFIC GRAVITY CORRECTION FACTORS,  $C_g$

Gas Specific Gravity	Correction Factor $C_g$
0.55	1.13
0.60	1.08
0.65	1.04
0.70	1.00
0.75	0.97
0.80	0.94
0.85	0.91
0.90	0.88

PHYSICAL AND CHEMICAL PROPERTIES  
OF GLYCOLS(16)

TABLE 3A

	Ethylene Glycol	Diethylene Glycol	Triethylene Glycol
Molecular Weight	62.07	106.12	150.17
Specific Gravity @ 68°F	1.1155	1.1184	1.1255
Specific Weight, lb/gal.	9.292	9.316	9.375
Boiling Point @ 760 MMHg, °F	387.7	474.4	550.4
Freezing Point, °F	9.1	18.0	24.3
Surface Tension @ 77°F, dynes/cm	47.0	44.8	45.2
Heat of Vaporization @ 760 MMHg, BTU/lb	364	232	174

TABLE 3B  
100% Diethylene Glycol

Temp. °F	Sp Gr	Viscosity, cps	Sp Heat BTU/lb-°F	Thermal Conductivity BTU/hr-Sq ft-°F/ft
50	1.127	72	0.53	0.146
75	1.117	45	0.54	0.14
100	1.107	18	0.56	0.135
125	1.098	12.7	0.57	0.13
150	1.089	7.3	0.58	0.125
175	1.076	5.5	0.59	0.12
200	1.064	3.6	0.60	0.115
225	1.054	2.8	0.61	0.11
250	1.043	1.9	0.63	0.105
275	1.032	1.6	0.65	
300	1.021	1.3	0.66	

TABLE 3C  
100% Triethylene Glycol

Temp. °F	Sp Gr	Viscosity, cps	Sp Heat BTU/lb-°F	Thermal Conductivity BTU/hr-Sq ft-°F/ft
50	1.134	88	0.485	0.14
75	1.123	56	6.50	0.138
100	1.111	23	0.52	0.132
125	1.101	15.5	0.535	0.130
150	1.091	8.1	0.55	0.125
175	1.080	6.1	0.57	0.121
200	1.068	4.0	0.585	0.118
225	1.057	3.1	0.60	0.113
250	1.046	2.3	0.62	0.110
275	1.034	1.9	0.635	
300	1.022	1.5	0.65	

TABLE 4A  
VERTICAL INLET SCRUBBERS

SPECIFICATIONS

Nominal W.P. psig	Size O.D.	Nominal Gas Capacity MMSCFD <sup>1</sup>	Inlet & Gas Outlet Conn	Std Oil Valve	Shipping Weight lb
230	16"	1.8	2"	1"	900
	20"	2.9	3"	1"	1000
	24"	4.1	3"	1"	1200
	30"	6.5	4"	1"	1400
	36"	9.4	4"	1"	1900
	42"	12.7	6"	2"	2600
	48"	16.7	6"	2"	3000
	54"	21.1	6"	2"	3500
500	60"	26.1	6"	2"	4500
	16"	2.7	2"	1"	1000
	20"	4.3	3"	1"	1300
	24"	6.1	3"	1"	2100
	30"	9.3	4"	1"	2700
	36"	13.3	4"	1"	3800
	42"	18.4	6"	2"	4200
	48"	24.3	6"	2"	5000
600	54"	30.6	6"	2"	5400
	60"	38.1	6"	2"	7500
	16"	3.0	2"	1"	1100
	20"	4.6	3"	1"	1400
	24"	6.3	3"	1"	2200
	30"	9.8	4"	1"	2800
	36"	14.7	4"	1"	3900
	42"	20.4	6"	2"	4500
1000	48"	27.1	6"	2"	5100
	54"	34.0	6"	2"	6000
	60"	42.3	6"	2"	8100
	16"	3.9	2"	1"	1100
	20"	6.1	3"	1"	1600
	24"	8.8	3"	1"	2500
	30"	13.6	4"	1"	3200
	36"	20.7	4"	1"	4400
1000	42"	27.5	6"	2"	6300
	48"	36.9	6"	2"	8400
	54"	46.1	6"	2"	9700
	60"	57.7	6"	2"	14500

Nominal W.P. psig	Size O.D.	Nominal Gas Capacity MMSCFD <sup>1</sup>	Inlet & Gas Outlet Conn	Std Oil Valve	Shipping Weight lb
1200	16"	4.2	2"	1"	1150
	20"	6.5	3"	1"	1800
	24"	10.0	3"	1"	2600
	30"	15.3	4"	1"	3400
	36"	23.1	4"	1"	4700
	42"	31.0	6"	2"	6700
	48"	40.5	6"	2"	8500
	54"	51.4	6"	2"	11300
1440	16"	4.8	2"	1"	1500
	20"	6.7	3"	1"	2100
	24"	11.2	3"	1"	2800
	30"	17.7	4"	1"	3900
	36"	25.5	4"	1"	5400
	42"	34.7	6"	2"	7800
	48"	45.3	6"	2"	9200
	54"	56.1	6"	2"	12900
60"	69.6	6"	2"	16000	

1. Gas capacity based on 100°F, 0.7 sp gr, and vessel working pressure.

TABLE 4B

Size O.D.	Two Phase Scrubber			Three Phase Scrubber			
	Shell Height	Settling Volume, Bbl <sup>1</sup>	Liquid Capacity Bbl/day <sup>2</sup>	Shell Height	Settling Volume Bbl <sup>1</sup>	Liquid Capacity Bbl/day <sup>3</sup>	
						Oil	Water
16"	5'	0.27	340	7-1/2'	0.72	100	100
20"	5'	0.44	530	7-1/2'	1.15	160	160
24"	5'	0.66	760	7-1/2'	1.68	240	240
30"	5'	1.13	1180	7-1/2'	2.78	400	400
36"	7-1/2'	1.73	2000	10'	4.13	590	590
42"	7-1/2'	2.52	3000	10'	5.80	830	830
48"	7-1/2'	3.48	4000	10'	7.79	1120	1120
54"	7-1/2'	4.65	5000	10'	10.12	1450	1450
60"	7-1/2'	6.01	6000	10'	12.73	1830	1830

1. Based on nominal 1000 psig W.P. scrubber.
2. Based on 1.0 minute retention time.
3. Based on 5.0 minute retention time.



TABLE 5A  
TRAY TYPE GLYCOL/GAS CONTACTORS

SPECIFICATIONS

Nominal W.P. psig	Size O.D.	Nominal Gas Capacity MMSCFD <sup>1</sup>	Gas Inlet & Outlet Size	Glycol Inlet & Outlet Size	Glycol Cooler Size	Shipping Weight lb
250	12-3/4"	1.5	2"	1/2"	2" x 4"	800
	16"	2.4	2"	3/4"	2" x 4"	900
	18"	3.2	3"	3/4"	3" x 5"	1100
	20"	4.0	3"	1"	3" x 5"	1400
	24"	6.1	3"	1"	3" x 5"	2000
	30"	9.9	4"	1"	4" x 6"	2400
	36"	14.7	4"	1-1/2"	4" x 6"	3200
	42"	19.7	6"	1-1/2"	6" x 8"	4400
	48"	26.3	6"	2"	6" x 8"	6300
	54"	32.7	6"	2"	6" x 8"	7700
	60"	40.6	6"	2"	6" x 8"	9600
500	12-3/4"	2.0	2"	1/2"	2" x 4"	1000
	16"	3.4	2"	1/2"	2" x 4"	1300
	18"	4.3	3"	3/4"	3" x 5"	1500
	20"	5.3	3"	1"	3" x 5"	1700
	24"	8.3	3"	1"	3" x 5"	2900
	30"	13.1	4"	1"	3" x 5"	3900
	36"	19.2	4"	1-1/2"	4" x 6"	6000
	42"	27.4	6"	1-1/2"	6" x 8"	7700
	48"	35.1	6"	2"	6" x 8"	10000
	54"	44.5	6"	2"	6" x 8"	12000
	60"	55.2	6"	2"	6" x 8"	15300
600	12-3/4"	2.2	2"	1/2"	2" x 4"	1100
	16"	3.4	2"	3/4"	2" x 4"	1300
	18"	4.5	3"	3/4"	3" x 5"	1600
	20"	5.5	3"	1"	3" x 5"	1800
	24"	8.5	3"	1"	3" x 5"	3000
	30"	14.3	4"	1"	3" x 5"	4000
	36"	21.2	4"	1-1/2"	4" x 6"	6300
	42"	29.4	6"	1-1/2"	6" x 8"	8400
	48"	39.2	6"	2"	6" x 8"	11300
	54"	49.3	6"	2"	6" x 8"	13400
	60"	61.3	6"	2"	6" x 8"	16500

Nominal W.P. psig	Size O.D.	Nominal Gas Capacity MMSCFD <sup>1</sup>	Gas Inlet & Outlet Size	Glycol Inlet & Outlet Size	Glycol Cooler Size	Shipping Weight lb
1000	12-3/4"	2.7	2"	1/2"	2" x 4"	1300
	16"	4.3	2"	3/4"	2" x 4"	1600
	18"	5.5	3"	3/4"	3" x 5"	2100
	20"	7.3	3"	1"	3" x 5"	2600
	24"	11.3	3"	1"	3" x 5"	4200
	30"	18.4	4"	1"	3" x 5"	5500
	36"	27.5	4"	1-1/2"	4" x 6"	8500
	42"	37.1	4"	1-1/2"	6" x 8"	11800
	48"	49.6	6"	2"	6" x 8"	16200
	54"	62.0	6"	2"	6" x 8"	20200
60"	77.5	6"	2"	6" x 8"	26300	
1200	12-3/4"	3.0	2"	1/2"	2" x 4"	1500
	16"	4.7	2"	3/4"	2" x 4"	1900
	18"	6.0	3"	3/4"	3" x 5"	2300
	20"	7.8	3"	1"	3" x 5"	3000
	24"	12.0	3"	1"	3" x 5"	4900
	30"	20.1	4"	1"	3" x 5"	6400
	36"	29.8	4"	1-1/2"	4" x 6"	10000
	42"	41.4	6"	1-1/2"	6" x 8"	13100
	48"	54.1	6"	2"	6" x 8"	18400
	54"	68.4	6"	2"	6" x 8"	23500
60"	85.0	6"	2"	6" x 8"	29000	
1440	12-3/4"	3.1	2"	1/2"	2" x 4"	1800
	16"	4.9	2"	3/4"	2" x 4"	2200
	18"	6.5	3"	3/4"	3" x 5"	2800
	20"	8.3	3"	1"	3" x 5"	3500
	24"	13.3	3"	1"	3" x 5"	5800
	30"	22.3	4"	1"	3" x 5"	7500
	36"	32.8	4"	1-1/2"	4" x 6"	11700
	42"	44.3	6"	1-1/2"	6" x 8"	14400
	48"	58.3	6"	2"	6" x 8"	20000
	54"	74.0	6"	2"	6" x 8"	25800
60"	91.1	6"	2"	6" x 8"	32000	

1. Gas capacity based on 100°F, 0.7 sp gr and contactor working pressure.

TABLE 5B  
TRAY TYPE GLYCOL/GAS CONTACTORS

OTHER SPECIFICATIONS

Size O.D.	Standard Shell Height <sup>1</sup>	Standard Glycol Cooler Height <sup>1</sup>	Add to Height For Add. Tray, Ea.	Glycol Charge, gal.	
				Standard <sup>1</sup>	For Each Add. Tray
12-3/4"	13'	9'	2'	10	1.5
16"	13'	9'	2'	13	2.2
18"	13'	9'	2'	16	2.8
20"	13'	9'	2'	19	3.5
24"	13'	9'	2'	25	5.0
30"	13'	9'	2'	38	8.2
36"	13'	9'	2'	53	11.8
42"	13'	9'	2'	73	16.8
48"	13'	9'	2'	90	20.9
54"	13'	9'	2'	112	26.6
60"	13'	9'	2'	137	32.6

1. For Standard Four Tray Contactor

TABLE 6A  
PACKED COLUMN GLYCOL/GAS CONTACTORS

SPECIFICATIONS

Nominal W.P. psig	Size O. D.	Nominal Gas Capacity MMSCFD <sup>1</sup>	Gas Inlet & Outlet Size	Glycol Inlet & Outlet Size	Glycol Cooler Size	Shipping Weight lb
250	10-3/4"	1.1	2"	1/2"	2" x 4"	500
	12-3/4"	1.6	2"	1/2"	2" x 4"	600
	14"	1.9	2"	1/2"	2" x 4"	650
	16"	2.5	2"	1/2"	2" x 4"	800
	18"	3.4	3"	3/4"	3" x 5"	900
	20"	4.0	3"	3/4"	3" x 5"	1100
	24"	5.5	3"	1"	3" x 5"	1800
500	10-3/4"	1.5	2"	1/2"	2" x 4"	600
	12-3/4"	2.2	2"	1/2"	2" x 4"	700
	14"	2.6	2"	1/2"	2" x 4"	750
	16"	3.4	2"	1/2"	2" x 4"	900
	18"	4.4	3"	3/4"	3" x 5"	1000
	20"	5.5	3"	3/4"	3" x 5"	1500
	24"	7.5	3"	1"	3" x 5"	2500

Nominal W.P. psig	Size O.D.	Nominal Gas Capacity MMSCFD <sup>1</sup>	Gas Inlet & Outlet Size	Glycol Inlet & Outlet Size	Glycol Cooler Size	Shipping Weight lb
600	10-3/4"	1.7	2"	1/2"	2" x 4"	650
	12-3/4"	2.4	2"	1/2"	2" x 4"	750
	14"	2.9	2"	1/2"	2" x 4"	800
	16"	3.8	2"	1/2"	2" x 4"	950
	18"	4.8	3"	3/4"	3" x 5"	1100
	20"	6.0	3"	3/4"	3" x 5"	1700
	24"	8.1	3"	1"	3" x 5"	2700
1000	10-3/4"	2.3	2"	1/2"	2" x 4"	900
	12-3/4"	3.3	2"	1/2"	2" x 4"	1000
	14"	4.0	2"	1/2"	2" x 4"	1100
	16"	5.2	2"	1/2"	2" x 4"	1300
	18"	6.6	3"	3/4"	3" x 5"	1800
	20"	8.2	3"	3/4"	3" x 5"	2300
	24"	11.8	3"	1"	3" x 5"	3500
1200	10-3/4"	2.5	2"	1/2"	2" x 4"	1200
	12-3/4"	3.6	2"	1/2"	2" x 4"	1300
	14"	4.1	2"	1/2"	2" x 4"	1500
	16"	5.4	2"	1/2"	2" x 4"	1700
	18"	6.9	3"	3/4"	3" x 5"	2200
	20"	8.5	3"	3/4"	3" x 5"	2800
	24"	12.3	3"	1"	3" x 5"	4000
1440	10-3/4"	2.6	2"	1/2"	2" x 4"	1300
	12-3/4"	3.7	2"	1/2"	2" x 4"	1400
	14"	4.5	2"	1/2"	2" x 4"	1600
	16"	5.9	2"	1/2"	2" x 4"	1900
	18"	7.5	3"	3/4"	3" x 5"	2500
	20"	9.3	3"	3/4"	3" x 5"	3100
	24"	12.7	3"	1"	3" x 5"	4500

1. Gas capacity based on 100°F, 0.7 sp gr and contactor working pressure.

TABLE 6B  
PACKED GLYCOL/GAS CONTACTORS

OTHER SPECIFICATIONS

Size O.D.	Standard Shell Height	Standard Glycol Cooler Height	Standard Contacting Element <sup>1</sup>	Glycol Charge gal.
10-3/4"	9'	7'	1" x 4"	6
12-3/4"	9'	7'	1" x 4"	7
14"	9'	7'	1" x 4"	8
16"	9'	7'	1" x 4"	10
18"	9'	7'	1" x 4"	12
20"	9'	7'	1" x 4"	14
24"	9'	7'	1" x 4"	18

1. Standard contacting element is carbon steel metal pall rings of size listed in Table.

TABLE 7  
GLYCOL RECONCENTRATORS

SPECIFICATIONS

Reboiler Capacity Btu/hr	Glycol Capacity gph*	Reboiler Size Dia. x Len.	Heat Exchanger Surge Tank, Size Dia. x Len.	Stripping Still Size, Dia. x Ht.	Reflux Condenser Size, Dia. x Ht.	Flash Separator Size Dia. x Ht.
75,000	20	18" x 3'-6"	18" x 3'-6"	6-5/8" x 4'-6"	6-5/8" x 2'-0"	12" x 48"
75,000	35	18" x 3'-6"	18" x 3'-6"	6-5/8" x 4'-6"	6-5/8" x 2'-0"	12" x 48"
125,000	40	18" x 5'	18" x 5'	6-5/8" x 4'-6"	6-5/8" x 2'-0"	16" x 48"
125,000	70	18" x 5'	18" x 5'	6-5/8" x 4'-6"	6-5/8" x 2'-0"	16" x 48"
175,000	90	24" x 5'	24" x 5'	8-5/8" x 4'-6"	8-5/8" x 2'-0"	16" x 48"
175,000	100	24" x 5'	24" x 5'	8-5/8" x 4'-6"	8-5/8" x 2'-0"	16" x 48"
250,000	150	24" x 7'	24" x 7'	8-5/8" x 5'-0"	8-5/8" x 2'-0"	16" x 48"
350,000	210	24" x 10'	24" x 10'	10-3/4" x 5'-0"	10-3/4" x 2'-6"	20" x 48"
400,000	250	30" x 10'	30" x 10'	10-3/4" x 6'-0"	10-3/4" x 2'-6"	20" x 48"
500,000	315	36" x 10'	36" x 10'	12-3/4" x 7'-0"	12-3/4" x 2'-6"	24" x 48"
750,000	450	36" x 15'	36" x 10'	14" x 8'-0"	14" x 3'-0"	30" x 48"
850,000	450	42" x 15'	36" x 10'	14" x 8'-0"	14" x 3'-0"	30" x 48"
1,000,000	450	48" x 16'	36" x 10'	16" x 8'-0"	16" x 3'-0"	30" x 48"

69T

Heat Exchange Coil		Glycol Pump Model	High Pressure Glycol Filter Size	Glycol Charge gal.	Shipping Wt lb
Size	Coil Area, sq ft				
1/2"	12.9	1715PV	1"	75	2100
1/2"	12.9	4015PV	1"	75	2100
1/2"	23.3	4015PV	1"	105	2200
1/2"	23.3	9015PV	1"	105	2250
1/2"	31.1	9015PV	1"	190	3200
1/2"	31.1	21015PV	1-1/2"	190	3200
3/4"	44.6	21015PV	1-1/2"	260	3700
3/4"	64.8	21015PV	1-1/2"	375	4000
3/4"	64.8	45015PV	2"	445	4500
1"	82.1	45015PV	2"	680	6500
1"	102.6	45015PV	2"	990	7000
1"	102.6	45015PV	2"	1175	7500
1"	102.6	45015PV	2"	1425	1000

\*Glycol capacity is based on circulating 2.5 gal. TEG/lb H<sub>2</sub>O and is controlled by the reboiler capacity or pump capacity whichever is smaller.

APPENDIX C  
TABLE 8  
GLYCOL PUMPS (3)

STANDARD HIGH PRESSURE PUMPS

CIRCULATION RATE – Gallons/Hour

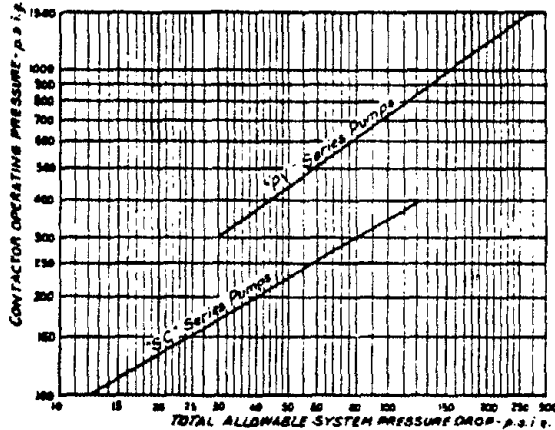
Model Number	*Pump Speed – Strokes/Minute. Count one stroke for each discharge of pump.																			
	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40			
1715 PV	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40			
4015 PV	..	..	..	..	..	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40
9015 PV	..	..	..	..	..	27	31.5	36	40.5	45	49.5	54	58.5	63	67.5	72	76.5	81	85.5	90
21015 PV	..	..	..	..	66	79	92	105	118	131	144	157	171	184	197	210	..	..	..	..
45015 PV	..	..	..	..	166	200	233	266	300	333	366	400	433	466	..	..	..	..	..	..

\*It is not recommended to attempt to run pumps at speeds less or greater than those indicated in the above table.

GAS CONSUMPTION

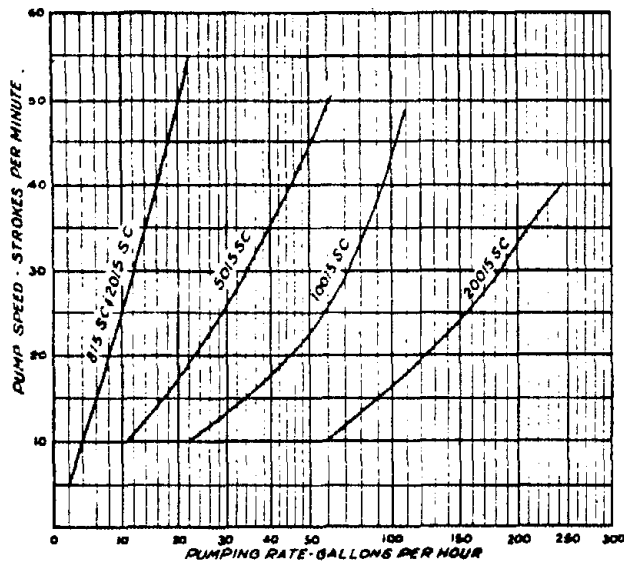
Operating Pressure—p.s.i.g.*	300	400	500	600	700	800	900	1000	1100	1200	1300	1400	1500
Cu. Ft./Gallon @ 14.4 & 60°F.	1.7	2.3	2.8	3.4	3.9	4.5	5.0	5.6	6.1	6.7	7.2	7.9	8.3

PRESSURE DROP



Pump Model	Pump Conn.	Size Strainer	High Press. Filter		Low Press. Filter	
			Size	Elements	Size	Elements
315PV	1/4"	1/2"	1"	1-2-3/4" x 9-3/4"	1/2"	1-3" x 18"
1715PV & 815SC	1/2"	3/4"	1"	1-2-3/4" x 9-3/4"	1/2"	1-3" x 18"
4015PV & 2015SC	1/2"	3/4"	1"	1-2-3/4" x 9-3/4"	1/2"	1-3" x 18"
9015PV & 5015SC	3/4"	1"	1"	2-2-3/4" x 9-3/4"	3/4"	1-3" x 36"
21015PV & 10015SC	1"	1-1/2"	1-1/2"	4-2-3/4" x 9-3/4"	1"	4-3" x 18"
45105PV & 20015SC	1-1/2"	2"	2"	8-2-3/4" x 9-3/4"	1-1/2"	4-3" x 36"

## SMALL BORE PUMPS FOR LOW PRESSURES



### GAS CONSUMPTION

Operating Pressure—p.s.i.g.	100	200	300	400
Cu. Ft./Gal. @ 14.4 & 60°F	1.0	1.9	2.8	3.7

## MODEL 315PV SMALL PUMP

### CIRCULATION RATE—Gallons/Hour

*Pump Speed — Strokes / Minute. Count one stroke for each discharge of pump.																
20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100
2.5	3.2	3.9	4.5	5.2	5.8	6.5	7.1	7.8	8.4	9.1	9.7	10.4	11.0	11.7	12.3	13.0

\* Do not operate at speeds in excess of 100 strokes per minute. Count ONE stroke for each discharge of pump.

### GAS CONSUMPTION

Operating Pressure — p.s.i.g.	100	200	300	400	500	600	700	800	900	1000	1100	1200	1300	1400	1500
Cu. Ft./Gallon @ 14.4 & 60°F	0.9	2.0	2.9	3.9	4.8	5.7	6.7	7.6	8.5	9.5	10.3	11.2	12.2	13.1	14.0

APPENDIX C

CHEMICAL ANALYSIS - PROCEDURES

Distillation Range	C-1
Gas Chromatographic Analysis of Glycols	C-2-C-3
Determination of Hydrocarbons in Aqueous Glycol Solutions	C-4
Determination of Foaming in Glycol Solutions	C-5
Iron Determination	C-6-C-7



## GAS CHROMATOGRAPHIC ANALYSIS OF GLYCOLS

### . Apparatus

1. Gas chromatograph capable of operating with temperature programming. Thermal conductivity detector
2. Microsyringe, 5 to 10  $\mu$ l capacity
3. Integrator

### . Column

Glass or stainless steel, 2 mm I.D. x 3-4' length. (For ss tubing, use 1/8 in. O.D. x 0.012 in. average wall thickness)

### . Packing

Tenax GC, 60/80 mesh. (Available from Alltech Associates, Inc., Houston, or any GC supply house) (See note (1))

### . Reagents

1. Glycols, EG through T<sub>4</sub>EG, and 1,3-butanediol for standards
2. Helium, commercial grade, for carrier gas

### . Operating Conditions

1. Column temperature (oven temp.), temperature programmed from 100°C to 285°C at 15°C/min., with approximately seven min. hold at maximum temperature
2. Thermal conductivity detector temperature, 300°C
3. Injector port temperature, 250°C
4. Dual column operation
5. He flow, 30 ml/min

### . Procedure

Prepare a five to ten gram sample of a standard by adding the various components, H<sub>2</sub>O, glycols and internal standard, in the approximate amounts shown in the attached sample chromatogram. Weigh each component to the nearest milligram (2). This standard will serve to calibrate the instrument if the internal standard method of analysis is to be used.

## DISTILLATION RANGE

### . Principle

The distillation range is determined essentially by ASTM Designation D-1078. The procedure is empirical and will give reproducible results only if the specified conditions are employed.

### . Apparatus

The apparatus as specified under ASTM Designation D-1078 is used with the following modifications:

Use ASTM E-202 Method D-1078-70 200-ml flash for all glycols. For all glycols except triethylene and tetraethylene, use the thermometers specified in Table 1 ASTM D-1078.

For triethylene and tetraethylene glycols, a thermometer such as ASTM 3C, 76 mm immersion, temperature range to 400°C, graduated in 1°C should be used.

### . Procedure

Follow ASTM Designation D-1078 with two modifications:

1. Source of heat. When a burner is used, the flame shall never be so large that it spreads over a circle greater than 3-1/2 inches on the under surface of the asbestos board.
2. Dry point. Adjust the heat input if necessary so that the time required to reach the dry point after the liquid residue in the flask is approximately five ml does not exceed five minutes.

### . Note

For correction of the boiling point for barometric pressure, the value of K is as follows:

Ethylene glycol . . . . .	0.042°C per mm
Diethylene glycol . . . . .	0.049°C per mm
Triethylene glycol . . . . .	0.054°C per mm
Tetraethylene glycol. . . . .	0.043°C per mm
Propylene glycol . . . . .	0.042°C per mm
Dipropylene glycol. . . . .	0.050°C per mm
Tripropylene glycol . . . . .	0.055°C per mm

Run the standard sample and calculate the response factor (amount/area) for each peak. The wt % of each component is then obtained by:

$$\% y = \frac{\text{Area } y \times \text{Response } y}{\text{Area int. std.} \times \text{Response int. std.}} \times \frac{\text{Amt. int. std.}}{\text{Amt. spl.}} \times 100$$

Prepare the unknown for analysis by adding a weighed amount of 1,3-butenediol (int. std.) to a weighed amount of unknown in the same proportion as in the standard sample. Calculate the amount of each glycol as shown above.

For most analyses, results in area % should be good to  $\pm 10\%$  of the value obtained by:

$$\% y = \frac{\text{Area } y}{\text{Area}} \times 100$$

Addition of 1,3-BD is still recommended since it will serve as a check.

. Notes

1. This type of packing is used because of its versatility for other analyses (alkanolamines). Other recommended packings for glycol analysis are CHROMOSORB 101, A Johns-Manville product, and PORAPAK PS 80/100.

Condition all packing according to the procedure suggested by the manufacturer.

2. Most glycols are not pure compounds, obtain analysis of each component to calculate the exact composition.

DETERMINATION OF HYDROCARBONS IN  
AQUEOUS GLYCOL SOLUTIONS

. Scope

This method is intended for the determination of volatile hydrocarbons up to the hexane series in aqueous glycol solutions.

. Principle

The hydrocarbons are distilled and measured volumetrically using a modified Dean-Stark apparatus. This test is empirical in nature.

. Apparatus

1. Distilling receiver, modified Dean-Stark trap with 24/40 T<sub>S</sub> Joints, graduated in milliliters
2. Condenser, water, 500-mm with 24/40 T<sub>S</sub> Joint
3. Boiling flask, 500-ml round bottom with 24/40 T<sub>S</sub> Joint
4. Heating mantle, to fit 3 (c)
5. Distillation column, 6-in by 30-mm, packed with glass beads, mounted between the flask and the trap
6. Thermometer

. Reagents

Hydrochloric acid, 1:1 solution. Mix equal volumes of water and concentrated hydrochloric acid.

. Procedure

Place 100 ml of sample in the boiling flask and 5 ml of 1:1 hydrochloric acid. Connect the flask to the apparatus and turn on the cooling water and heating mantle. Reflux the sample for two hours, collecting the distillate in the distilling receiver. Record the volume of the hydrocarbon fraction as the percent hydrocarbons.

DETERMINATION OF FOAMING IN  
GLYCOL SOLUTIONS

. Scope

This test is intended for the determination of the foaming characteristics of glycol solutions.

. Principle

Air is bubbled through the sample at a definite rate for five minutes, and the foam height and stability are measured.

. Apparatus

1. Stop watch, calibrated in seconds
2. Foaming apparatus (see attached figure)

. Procedure

1. Pour 200 ml. of sample into the 1,000-ml cylinder. Connect the air delivery tube and introduce oil-free air at four liters per minute.
2. Allow the bubbling to continue for five minutes, stop the air flow and start the stopwatch. Immediately record the height of the foam (cf. 5) and also the time in seconds for foam to break completely after the air supply is shut off.

. Note

Foam height, in milliliters, is the difference between the height of the foam and the initial height of the liquid (200 ml)

. Comments

1. This method can be used to evaluate the effects of anti-foam agents on a sample. Care should be exercised in cleaning the equipment since a very small amount of anti-foam agent may affect the test.

Foaming is sometimes caused by materials which can be removed by activated carbon treatment. The effect of activated carbon filtration can be evaluated by running foam tests on treated and untreated samples. The sample is treated by mixing with enough carbon (12 x 20 mesh) to completely remove the contaminant, and filtering the mixture through Whatman No. 41 filter paper.

## IRON DETERMINATION

### . Principle

The iron is reduced with hydroxylamine to the ferrous state, and reacted with 1,10-phenanthroline in an acetate buffered solution (pH 5) to form an orange-red complex. Photometric measurement is made at a wavelength of approximately 510 m $\mu$ . The milligrams of iron are determined from a standard curve.

### . Apparatus

1. Colorimeter, photoelectric, capable of measuring the absorbance at 510 m $\mu$
2. Optical cell, 50-mm (see Note)

### . Reagents

1. Iron, standard solution. Dissolve 0.1000 gram of primary standard iron wire in 50 ml of distilled water, 25 ml of concentrated reagent grade hydrochloric acid, and 1 ml of concentrated reagent grade nitric acid. Cool the solution, transfer to a one liter volumetric flask and dilute to volume with distilled water. Pipet 100 ml of this solution into another one liter volumetric flask. Add 10 ml of concentrated reagent grade hydrochloric acid and dilute to volume with distilled water. One milliliter of this solution contains 0.010 milligrams of iron.
2. Hydrochloric acid, concentrated ACS reagent grade
3. Phenolphthalein indicator, 0.5% solution. Dissolve 0.5 gram of the disodium salt of phenolphthalein in 100 ml of distilled water.
4. Hydroxylamine hydrochloride, 10% solution. Dissolve 10 grams of reagent grade hydroxylamine hydrochloride, NH<sub>2</sub>OH.HCl, in distilled water and dilute to 100 ml.
5. Acetate buffer solution, pH 5. Dissolve 272 grams of reagent grade sodium acetate trihydrate, NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>.3H<sub>2</sub>O, in 500 ml of distilled water. Add 240 ml of glacial acetic acid; cool and dilute to one liter with distilled water.
6. 1,10-phenanthroline, 1% solution. Dissolve 2.0 grams of 1,10-phenanthroline in methyl alcohol and dilute to 250 ml with alcohol.

. Preparation of Standard Curve

1. Using a buret, transfer 0, 0.5, 1.0, 2.0, 3.0 and 5.0 ml of the standard iron solution into a series of 100-ml volumetric flasks. Dilute each flask to about 50 ml with water.
2. Add to each flask with mixing after each addition, 5 ml of hydroxylamine hydrochloride solution, 10 ml of acetate buffer solution and 5 ml of the phenanthroline solution. Dilute to volume with water and allow to stand for 15 minutes for complete color development.
3. Obtain the colorimeter reading at 510 m $\mu$  using the blank as a reference.
4. Plot the number of milligrams of iron versus colorimeter readings.

. Procedure

1. Weight a 1.0-g sample to the nearest 0.001 gram into a 100 ml flask. Add 50 ml of water and concentrated hydrochloric acid in increments until just acid to phenolphthalein. Add 1 ml concentrated hydrochloric acid in excess. Cool, dilute to volume and mix.
2. Prepare a blank of 50 ml of distilled water in a 100-ml volumetric flask and develop the color as in 3 and 4 above in both flasks.
3. Find the number of milligrams of iron per 100 ml of solution from the standard curve. Dilute the sample approximately if the reading is off-scale.

. Calculation

Where

A = milligrams of iron per 100 ml of final solution

B = grams of sample in aliquot

Then

$$\frac{A \times 1000}{B} = \text{ppm iron (actual basis)}$$

. Note

This method has been written for cells having a 50-mm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

APPENDIX D

CHEMICAL ANALYSIS - CASE STUDIES

Two examples will serve to illustrate how analysis of the glycol helps to identify and solve plant problems.

Case 1. Contaminated Glycol

A sample of TEG was received with a request for analysis. The unit in question had a history of fire tube failure and excessive losses of glycol. A complete analysis was performed and the results are shown in Table 1.

Three factors indicate that the glycol is being thermally degraded:

1. Unusually high amounts of lower glycols, EG and DEG.
2. Very low pH.
3. Sweet odor typical of burned glycol

Salt contamination is at the root of the problem in this unit. Some hydrocarbons are also present as indicated by the formation of a stable emulsion with water. Very likely, these are heavy hydrocarbons since the distillation shows only less than half a percent of the low boiling components.

Recommendations were made to remove the contaminated glycol from this unit and to recharge it with fresh TEG after thorough cleaning of the reboiler and fire tube. The decision whether to discard this material or to reclaim it is an economic one and should be made on the basis of 1) availability of reclaiming equipment, 2) volume of the solution, 3) shrinkage due to the presence of the lower glycols and contaminants.

TABLE 1. Analysis of Contaminated Field  
Sample of Triethylene Glycol. Case 1

Appearance	Clear, dark brown sweet smelling odor
pH (50% aq. solution)	4.2
Emulsion with water	Yes
% wt H <sub>2</sub> O (K-F titration)	4.0
<u>Gas Chromatography, % wt</u>	
Ethylene glycol	0.6
Diethylene glycol	4.2



Triethylene glycol	89.3
Tetraethylene glycol	<u>1.1</u>
Total glycols	95.9
Hydrocarbons (C <sub>6</sub> or lighter), % vol.	<0.5
Inorganic chlorides, ppm Cl <sup>-</sup>	7400
Salt, calculated as NaCl, % wt	1.2

Foaming Characteristics

	Treatment		Carbon #
	None	Antifoam*	
Foam height (ml)	150	30	50
Stability (sec)	>120	5	3

\* 200 ppm, Dow Corning DB-31

# Slurried with 3% granular carbon for 45 minutes

TABLE 2. Plant Conditions. Case 2

Gas flow rate	55 MMSCFD
Inlet pressure	750 psig
Inlet temperature	90°F
TEG circulation	8 gpm
Lean TEG temp.	76°F
Water in	58 lb/MMSCF

TABLE 3. Analysis of Lean and Rich  
TEG Samples. Case 2

	<u>Rich</u>	<u>Lean</u>
pH	9.1	9.5
% wt H <sub>2</sub> O (K-F)	3.4	1.6
<u>Gas Chromatography, % wt</u>		
Ethylene glycol	0.24	0.18
Diethylene glycol	1.9	2.0
Triethylene glycol	91.1	95.4
Tetraethylene glycol	<u>0.6</u>	<u>0.8</u>
Total Glycols	93.8	98.4
Hydrocarbons, % vol.	2.8	Trace
Foaming characteristics	Emulsion formed with air flow	

Contamination by the salt was traced to a one-time occurrence of salt water carryover. The inlet separator was deemed of sufficient size. Installation of a full-flow activated carbon filter (one was not in existence) was recommended based on the results of the foam tests. Excessive losses of the glycol were also traced to foaming. This problem was controlled, as a temporary measure, by the addition of a silicone type antifoam.

#### Case 2. Insufficient Dew Point Depression

A gas dehydrator operating at the conditions shown in Table 2 was not effecting the desired dew point depression. Samples of lean and rich TEG were analyzed with the results shown in Table 3.

From equilibrium data, it can be shown that this dehydrator should give a dew point depression of at least 70°F with 1.6% H<sub>2</sub>O in the lean glycol. The water removed, however, as calculated below,

$$8.0 \text{ gpm} \times 60 \times 24 \times \frac{9.3 \text{ lb}}{\text{gal}} \times \frac{(3.4-1.6)}{100} = 1,928 \frac{\text{lb H}_2\text{O}}{\text{day}}$$

$$1,928 \div 55 \text{ MMSCFD} = 35.1 \frac{\text{lb H}_2\text{O removed}}{\text{MMSCF}}$$

is only sufficient to give an outlet dew point of 60°F. This corresponds to a dew point depression of 30°F.

The main problem in this unit was due to the presence of hydrocarbons which had probably coated the contact surfaces in the absorber and prevented good contact between the gas and the glycol. Plant personnel deemed impractical to clean out this unit and provide for removal of hydrocarbons. The low temperature of the lean TEG going to the top of the absorber was also contributing to the problem. Hydrocarbons from the gas will condense into the glycol if the temperature of the latter is not maintained 10-15°F above the gas temperature.

Raising the temperature of the lean TEG to approximately 100°F and increasing the glycol circulation to the maximum allowed by the pump capacity affected an improvement in the dehydration which was sufficient to satisfy the operational requirement of the plant.

#### CONCLUSIONS

The two case studies shown above are classical examples of problems and solutions encountered in the day-by-day operation of glycol dehydrators. Not all field problems are as straightforward as the ones discussed. The examples have been selected for didactic convenience, but serve well to illustrate the fact that applications of the analytical techniques described in this paper can pinpoint the problem and indicate the solution. Not all the analyses shown should be performed on all samples, of course. The judgement of the analyst and his communication with plant personnel will determine what type of analysis is best indicated. Most of the methods described, here and in Reference 7, can, to a certain extent, be adapted for field use.