

Refrigeration plant room
for compound ammonia
chilling at -70°C

Refrigeration for Manufacture of Cephalosporin

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Introduction

During the processing of Cefroxime Acid Axetil Crystalline, which has three manufacturing steps, low temperature is required for two different steps. Liquid nitrogen is used to achieve the desired temperature. The reactors in both the steps have limpets as well as jackets. Liquid nitrogen used in limpets is vented out and escapes to the atmosphere at a low temperature of -30°C to -80°C resulting in loss of energy; liquid nitrogen gets released to the atmosphere along with solvents and product vapours, resulting in solvent loss as well as corrosion of nearby equipment. Since liquid nitrogen has high cooling capability, the time taken to achieve the desired temperature is low, but the system is inefficient from the energy consumption point of view as well as vapourisation of liquid nitrogen. It was a challenge to attain the low temperature by other means in the time taken by liquid nitrogen, or lesser.

Mechanical Refrigeration System Coupled with Lithium Bromide Vapour Absorption System

In order to achieve -65°C temperature in the process, a system was conceived with -70°C evaporating temperature, keeping in

view the following:

- Energy efficient system with minimum kW/TR.
- Choice of proper refrigerant.
- Since there was no evaporator, the reactor limpet coil and jacket was used as the evaporator with surface area limitation of the reactor. It was not possible to change the geometry and capacity of the reactor, and the available surface area was a constraint.
- For optimum circulation rate fixation in order to achieve the temperature within the stipulated time, the possibility of conventional brine cooling was ruled out.
- Optimal capital cost by opting for a single system, since there were two different steps with different process parameters and using one system to serve both was a challenge.

About the Author

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continued on page 30

continued from page 28

- f) Capacity synchronisation within two stages – high stage and booster – to avoid hunting.
- g) Choice of system – liquid overfeed and evaporative.

Mechanical Vapour Compressor System Using Two Stage Screw Compressor, with Lithium Bromide Vapour Absorption System in Third Stage

In order to optimise energy usage, LiBr vapour absorption chiller was opted for generating chilled water for condensing ammonia gas. In order to take advantage of cogeneration plant, 150TR LiBr chilled water plant was chosen with steam consumption of 650 kg/hr. This quantity of steam is capable of generating 75 kW of power. This 75 kW power generated in the system subsidised the power requirements of two stages, resulting in low consumption of electricity compared to a mechanical screw/reciprocating chiller for chilled water stage, which would add to total power consumption. Considering steam cost at Rs.1.13/kg (from multi-fuel boiler), cost of steam is Rs.675 and power available would be 75 kW (at Rs.7.25), equivalent to Rs.543.75, say Rs.545. Thus, cost per TR is Rs.0.86, compared to screw chiller at 0.8 kW/TR at Rs.7.25/ kW, i.e. Rs.5.80/TR.

The Refrigerant Chosen was Ammonia due to

- I. Low cost of NH₃,
- II. Low capital cost,
- III. Easily detectable during leakage,
- IV. Lower circulation rate.

Ammonia refrigerant systems have smaller mass flow as compared to R-22 and R-134a, e.g. a 100TR plant for -25°C/+40°C requires 1193 kg/hr of ammonia compared to 8317 kg/hr of R-22, hence the pump required for circulation of ammonia would be smaller compared to R-22 or R-134a, resulting in saving of power.

The performance of R134a and ammonia in the liquid overfeed system is compared. As expected, due to the better thermodynamic properties of ammonia, the evaporator refrigerating capacity is higher for ammonia and needs less mass flux than R134a. (Ammonia has a high latent heat of vapourisation so, for equal

heat removal, much less ammonia mass is required to be circulated compared to halocarbons). Please see Figure 1 to 6.

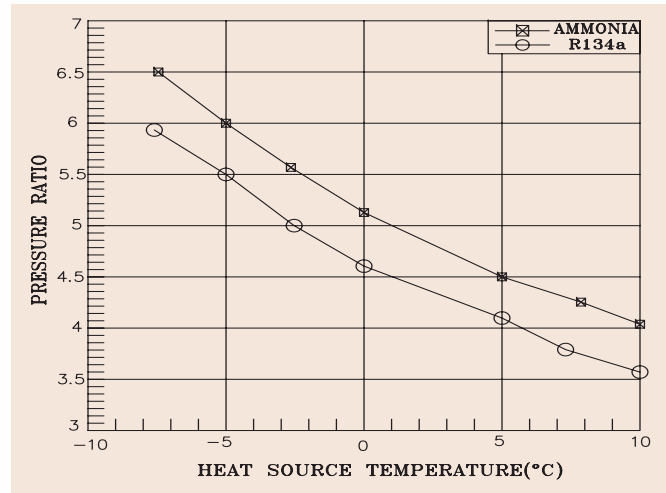


Figure 2: Compressor pressure ratio vs. heat source temperature

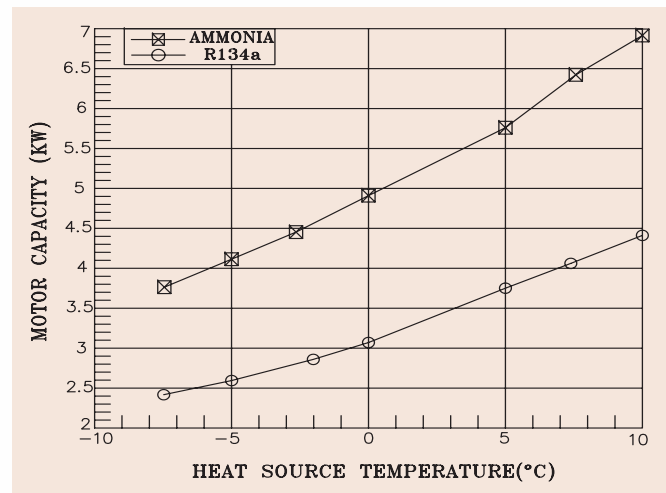


Figure 3: Motor capacity vs. heat source temperature

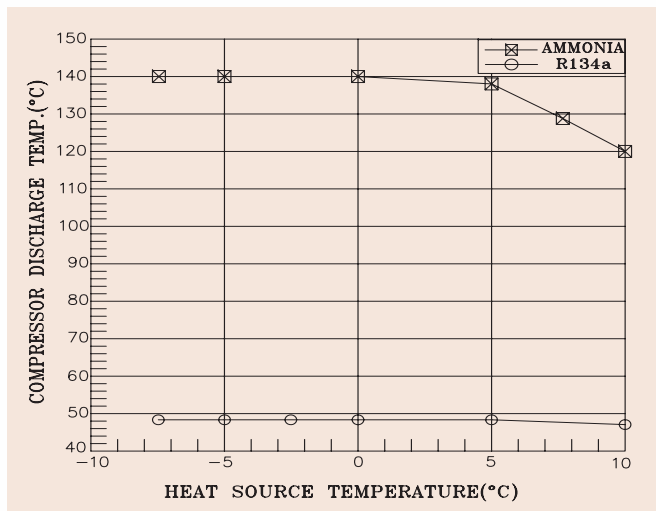


Figure 1: Compressor discharge temperature vs. heat source temperature

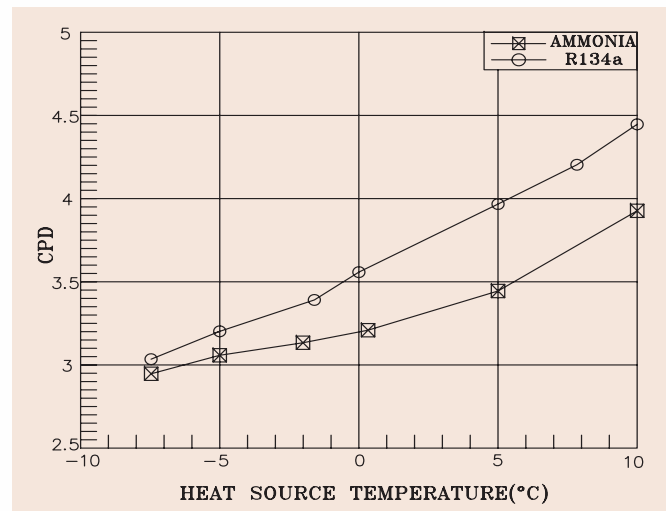


Figure 4: CPD vs. heat source temperature

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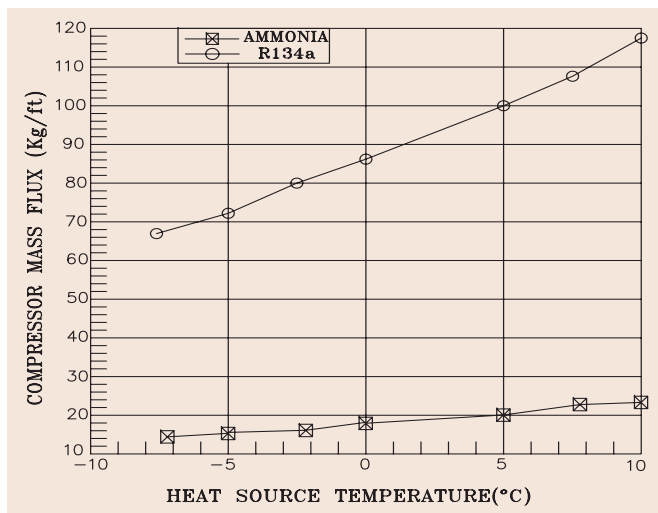


Figure 5: Compressor mass flux vs. heat source temperature

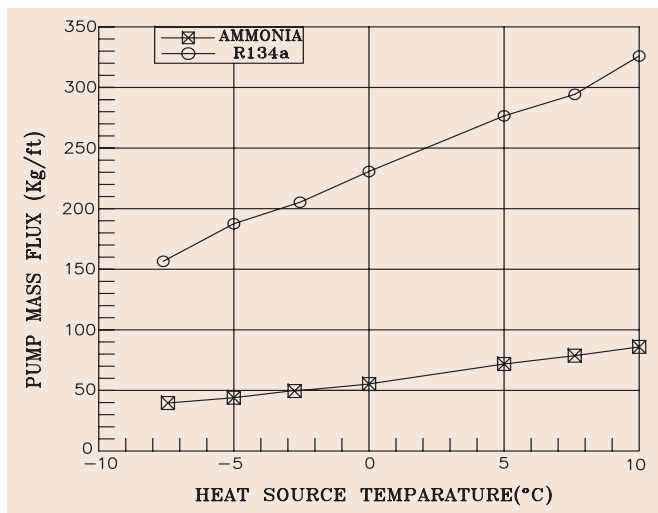


Figure 6: Pump mass flux vs. heat source temperature

Use of brine as a cooling medium to chill the reaction mass in the reactor was ruled out since -70°C evaporating temperature would give chilled brine in the range of -60°C to -65°C , and it would not be able to pull down the temperature of reaction mass to -55°C , or the time taken would be high; hence the possibility of using reactor with limpet coil and jacket for ammonia liquid feed circulation was explored, automating the back pressure of suction line by picking of temperature signal from the reaction mass. LP receiver was integrated in the system to take care of fluctuating loads of two reactors working with different parameters at the same time, and to take care of exothermicity of reactions.

Most of the designers/users feel that higher the pressure, better is the pump flow to evaporator/cooler and better is the performance. This could be incorrect. The inlet pressure to the evaporator should be just enough to overcome the pressure drop inside the cooler and the wet refrigerant return line up to the LP vessel, e.g. if the plant has been designed for an evaporating temperature of -32°C , saturation pressure corresponding to -32°C

is 1 bar absolute. If the pressure at the inlet is higher, say 2 bar, the corresponding saturated evaporating temperature is -18°C . This means although the liquid supply temperature is -32°C , it would not evaporate till the pressure inside the evaporator drops to 1 bar. Please see Figure 7.

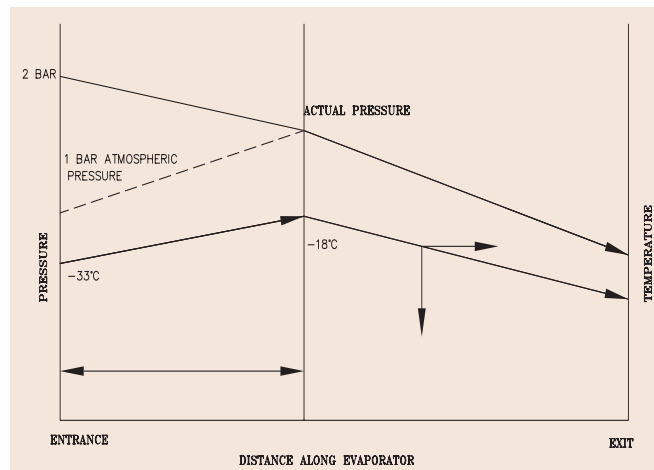


Figure 7: Pressure vs. distance along evaporator

Circulation Rate

Refrigeration system performance is interlinked with the load pattern, and this cannot be met with instantaneously. However, pump circulation decouples the refrigeration system from load variation acting as a buffer. Liquid overfeed means more circulation of ammonia liquid in the evaporator (in our case the reactor jacket/limpet coil) is more than the liquid actually vapourising. Excess liquid means overfeed with liquid overfeeding. Vapours coming out of the evaporator are close to saturated conditions without superheat, resulting in lower temperature at the compressor inlet, which corresponds to lower discharge temperature resulting in better and stable performance.

In this article, we have used the terms overfeed rate, overfeed ratio, recirculation ratio, circulating number, circulating rate, etc. However, despite the lack of agreement on a single term, the definition of the concept remains the same in textbooks and handbooks.

Will Stoeckers, *Industrial Refrigeration Handbook*, p302, defines circulation ratio as

$$\text{Circulation ratio, } n = \frac{\text{refrigerant flow rate supplied to evaporator}}{\text{flow rate of refrigerant vaporized}}$$

ASHRAE *Refrigeration Handbook*, 1990, p2.3, states: "In a liquid overfeed system, the mass ratio of liquid pumped to the amount of vaporized liquid is the circulating number of rate."

The circulation ratio defines the quality of the refrigerant at the outlet of the evaporator. If the vapour quality is 50%, circulation ratio is 2:1. If the vapor quality is 33%, it is 3:1. Note that these definitions do not distinguish whether the work done by the evaporating refrigerant is useful or non-useful. It is a simple ratio between the amount of vapour generated and the amount of liquid fed to the evaporator.

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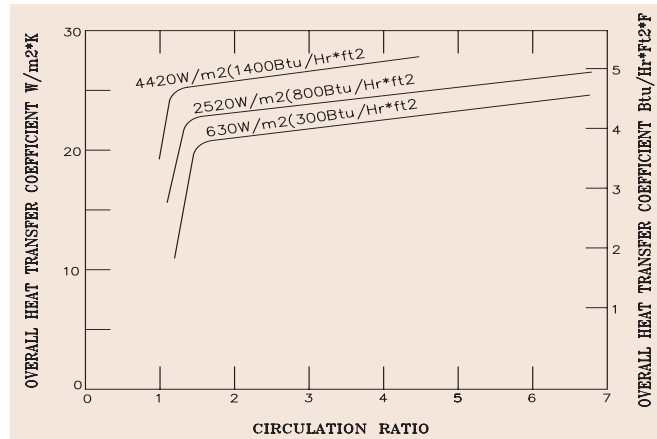


Figure 8: Overall heat transfer coefficient vs. circulation ratio

There are no clear-cut guidelines and explanations as to how to decide the circulation rate, and the designer has to establish it based on his experience and system parameters.

The heat transfer coefficient increases as the circulation rate increases. Please see Figure 8.

The largest gain in heat transfer is when the entire evaporator inside area remains completely wet from inlet to outlet, as it is the latent heat transfer or phase change process which absorbs the maximum heat from the fluid or product. Circulation rate of 1:6 was designed, with a bypass line for adjustment of circulation rate. This resulted in better heat transfer, optimum power consumption and the desired timing for temperature attainment.

The liquid recirculation system is the most advanced and energy efficient technique in ammonia liquid, through liquid-feed-flow rate of refrigerant vapourised pumps. For multiple uses, it saves electric energy and results in the highest efficiency of refrigeration systems. The advantages of ammonia liquid recirculation are as under:

- Increase in efficiency: The internal surface of cooling coils (evaporator) is the completely wetted efficiency of the refrigeration system.
- Compressor protection: The compressors are protected from liquid slugs resulting from malfunctioning of multiple units.
- Lower discharge pressure: Low suction superheat causes minimum discharge temperature and minimum condenser fouling.
- No need of accumulator: There is no need for individual accumulators for every evaporator.
- Increase in compressor life: Because of ideal entering suction gas conditions, lower compressor maintenance and fewer breakdowns, compressor life increases. Oil circulation rate to the

evaporators, discharge superheat and separation at the surge drum are reduced.

- Overall benefit: Liquid recirculation systems are most useful for multiple users, e.g. two reactors working at different chilling parameters. In the present case, higher velocities have been maintained in limpet coils resulting in better heat transfer and better temperature attainment timings compared to liquid nitrogen, due to less time taken to cool the mass in the reactor by ammonia circulating in limpet and jacket with higher velocity. Circulation ratio of 6:1 was less than the liquid nitrogen utility, since liquid nitrogen was vapourising in the limpet coil and heat transfer was inferior to liquid over-feed. There was saving of nearly 1.30hr for 6kl reactor mass chilling for the same process conditions.

In order to synchronise and take care of fluctuations as well as part loading and energy conservation, in addition to capacity control through the slide valve getting a signal from PLC, VFDs with compressor motors were also employed resulting in better synchronisation of high stage compressor with booster compressor and lower energy consumption as well as no hunting on part load.

Brief Working Principal

HP receiver acts as a storage vessel in which ammonia gas is charged, which works at 15°C temperature and 6.39kg/cm²g pressure. High pressure ammonia liquid from HP receiver feeds LP receiver after subcooling through the intercooler (liquid and gas cooler). The subcooling is done by the partially filled liquid in the intercooler shell, which enters from the HP receiver after expansion at -40°C.

From LP receiver, liquid ammonia pump delivers ammonia liquid to the reactor vessels with a circulation ratio of 6:1. The LP vessel performs two major roles in the system:

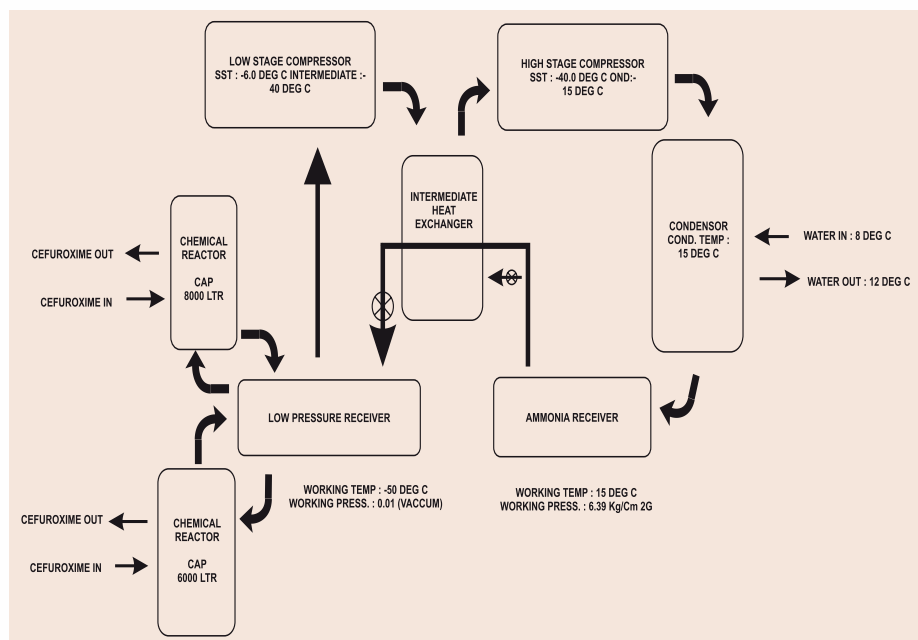


Figure 9: Flow diagram for -60°C NH₃/H₂O cascade system

continued from page 34

- I. Liquid vapour separation to ensure only vapour is sucked by the compressor.
- II. Liquid refrigerant storage at the required temperature so that it is available as a utility to meet the requirement of either one or more operating evaporators.

In the reactor vessel, heat exchange occurs between ammonia liquid and the chemical, due to which the liquid converts into vapour, and the vapour containing liquid and gas molecules of ammonia returns to LP receiver.

The low stage compressor sucks the dry gas from LP receiver at 0.8 bar (vacuum) pressure and delivers the superheated gas at 0.28 bar (vacuum).

This superheated gas goes to the cooler where desuperheating occurs to reduce the compressor work done.

The high stage compressor sucks the dry saturated gas from the cooler at 0.28 bar (vacuum), and delivers the high temperature gas at 6.28 bar pressure.

High temperature ammonia gas condenses to ammonia liquid at 15°C temp by exchanging heat with the chilled water, which

enters in the condenser at 8°C and leaves at 12°C, with flow rate of 90.8 cmh.

Now, this condensed ammonia liquid at 15°C goes to the HP receiver, and the cycle restarts. Please see *Figure 9* for a flow diagram for -70°C.

Optimum Flow Rate Adjustment

The system is provided with flow regulating valves at the inlet of jacket and limpet coil of reactor, and automatic flow regulation serves two functions. It adjusts the flow of liquid ammonia, and also acts as a check valve for both of the reactors under positive temperature and under pull-down conditions. In order to adjust the flow temperature, probe Pt 1000 is provided in the liquid mass inside the thermowell. The flow regulating valve is adjusted as per $4 \cong 20$ MA Signal from Pt 1000 temperature controller. The inlet pressure to the coil/jacket is adjusted to overcome the pressure drop across the reactor jacket/limpet and wet refrigerant line up to the LP receiver. Please see *Figure 10* and *11*.

Feed Section (Top or Bottom)

When feed rate increases, velocity increases. In order to get better efficiency, bottom feed is chosen since with top feed higher velocity would cause a lot of splashing and may result in some un-wetted surface in the total evaporation area. Liquid separation is unlikely to occur in bottom feed, since liquid is being pushed up violently by gas molecules vibrating at sonic speed. Although ASHRAE recommends circulation rate of 1:2 to 4 for bottom feed, the circulation rate 1:6 was opted since the application was typical with limitation of fixed surface area, reaction to be completed within specific time frame and removal of heat of reaction (exothermicity).

Some Useful Tips

1. Reactor vessel should be designed for 300 psi for shell side as well as jacket side.
2. Reactor shell should be fabricated from SS316 grade; for limpet, SS316/SS304 should be used.
3. Jacket should be designed for 300 psi, with material of construction to be chosen for low temperature application.
4. Safety valves should be provided in jacket and limpet side to vent out excess pressure being developed during long shutdowns.
5. LP vessel should be designed for 300 psi since long or accidental shutdown may lead to increase in pressure.
6. High-level alarm should be set at 50% level and shut down at 55% level.
7. Suction pipe to pump should be sized with 3 fps velocity. Ball valves in pump suction are best to give full flow.
8. Pump should be at least 6 pipe diameters away from elbow, and eccentric reducers at the pump flange are recommended when direct vertical inlet to pump is not possible. Preferred piping is always vertical direct inlet to pump suction.
9. Pump mounting places should be supported on flexible mounts to accommodate piping length variation due to temperature variation.

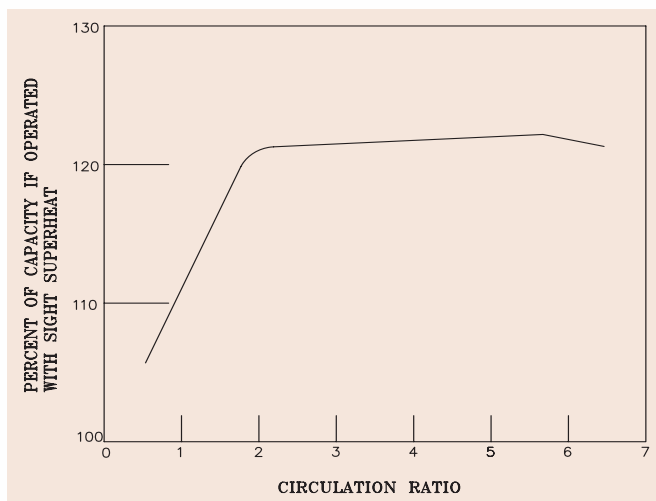


Figure 10: Percent of capacity vs. circulation ratio

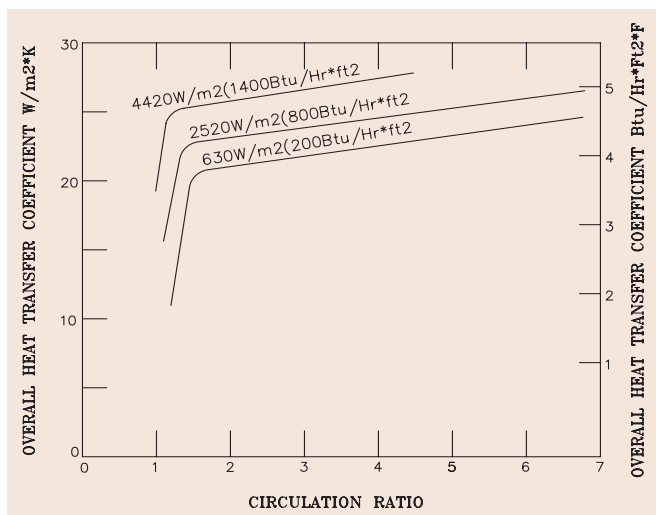


Figure 11: Overall Heat transfer coefficient vs. circulation ratio

continued from page 36

10. Oil drain pot should not be insulated and a pressure relief valve must be provided for the drain pot.
11. Vortex breaker should be provided in the inlet line of liquid to pump in the LP receiver.
12. Pumps should be selected for low NPSH (1.5 to 2.5 ft head). Liquid level from centre-line of pump suction to ammonia operating level should be twice the pump NPSH.
13. Provide independent suction pipe to each pump and not from common drop leg, if possible, to ensure that vapour bubbles caused by heat transfer from standby idle pump to suction of operating pump are avoided.

Acknowledgements and References

1. Refrigeration plant designed and supplied by Frick India Limited with the above considerations.
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