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SUBJECT: Methods of Tritium Recovery from Molten Lithium

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ABSTRACT

It is important to keep the tritium inventory in a blanket of a thermonuclear reactor at a low level both to eliminate possible hydriding of structural components and to reduce inventory cost. Removing the tritium from a lithium blanket by fractional distillation, flash vaporization, and fractional crystallization was investigated. No definitive data are available either on the vapor-liquid equilibrium between lithium and tritium at low T_2 concentrations, or on the rate of formation and decomposition of lithium tritide. The final distinction between the recovery systems discussed in this report will depend on such data, but presently distillation appears to be the best alternate to the diffusion scheme proposed by A.P. Fraas. The capital cost of equipment necessary to remove tritium by distillation appears to be greater than 10 million dollars for a 5000 Mw system, whereas the capital cost associated with the diffusion process has been estimated to be 4 million dollars.

*Revised in part by R.H. Mayer and P.J. Wood

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1. SUMMARY

It is desirable to maintain a low concentration of tritium in the blanket of a thermonuclear reactor both to prevent hydriding, and thus embrittlement of structural components, and to decrease inventory cost. The size and cost of a system to keep the tritium concentration at an acceptable level is important when considering the feasibility of producing power with fusion reactors.

A diffusional process of modest cost has previously been suggested (2). The purpose of this work was to investigate the possibility of removing tritium and helium (which is also generated in the lithium blanket) by fractional distillation, fractional crystallization, or flash vaporization.

A 5000 Mw (thermal) reactor with a tritium breeding ratio of 1.3 was chosen as the base model (this was the same as used in Ref. (2) for estimating the capital cost for the diffusional method). A tritium inventory level of 8 kg was maintained in the lithium (this corresponds to a mole % of 0.033 tritium in lithium), and the lithium blanket was maintained at a temperature of 1000°C.

A literature survey revealed no definitive data either for the vapor-liquid equilibrium in a lithium-hydrogen (or tritium) system at low concentration of hydrogen, or for the rate of decomposition and formation of lithium hydride (or tritide).

Analysis of the fractional crystallization method showed it to be highly unfavorable from the standpoint of operation control. On the basis of existing vapor-liquid equilibrium and reaction rate data, fractional distillation appears more suitable than flash vaporization in achieving the required separation. However, the capital cost for fractional distillation is greater than 10 million dollars compared to 4 million dollars for the diffusion process. Accurate designs and cost estimates for both the distillation and flash processes will require more reliable rate and equilibrium data.

It is suggested that the rate of formation of lithium tritide in the temperature range of 400-1000°C be determined since flash vaporization might be attractive if this rate is lower than indicated by the few, available data (5).

2. INTRODUCTION

Power production from nuclear fusion may be possible if certain plasma physics and engineering problems are solved. At present, researchers (1) agree that the best fuel for a practical fusion device would be a mixture of tritium and deuterium giving the following fusion reaction:

$$^{2}H + {}^{3}H \xrightarrow{T \cong 10^{8}\,^{o}K} n + {}^{4}He + 17.58 \text{ Mev}$$

It is necessary to produce tritium artificially since, unlike deuterium, it is not available from any natural source.

A possible way of producing tritium would be to bombard lithium with the neutrons produced in the above fusion reaction.

$$^{6}Li + n \longrightarrow {}^{3}H + {}^{4}He$$

$$^{7}Li + n \longrightarrow {}^{3}H + {}^{4}He + n'$$

In addition, the molten lithium could be used as the coolant for the fusion reactor. The feasibility of this scheme requires the separation of the tritium from the molten lithium. It is necessary to maintain a low inventory of tritium in the lithium for the following two reasons:

1. A high concentration of tritium in the lithium would cause hydriding and thus embrittlement of the niobium structural components {a hydrogen concentration in niobium of 980 ppm reduces the elongation in niobium to less than 5% at room temperature (3)}.

2. The high cost of tritium (taken to be $\$10^{6}$/kg) makes inventory costs prohibitive.

Tritium recovery from lithium by diffusion of the tritium through a permeable "window" of niobium has previously been examined (2). The purpose of this project was to examine and evaluate alternative methods of tritium recovery. To make a meaningful comparison, the model of the fusion reactor used in the diffusional separation process was retained (see Table 1).

Three possible recovery systems were examined:

1. Fractional distillation

2. Equilibrium flash vaporization

3. Fractional crystallization

Phase equilibria dictated that fractional crystallization was impractical, hence only fractional distillation and flash vaporization are discussed. Fractional crystallization is discussed further in Appendix 6.1.

Table 1. Reactor Model

Reactor type	magnetic mirror
Thermal power output, Mw	5000
Tritium generation in Li, kg/day	1.0
Tritium breeding ratio	1.3
Material inventories	
Li inventory in blanket, kg	50,000
*K inventory (intermediate heat transfer fluid), kg	90,000
*Surface areas	
Surface area in Li-K boiler, ft^2	75,000
Tube wall thickness in Li-K boiler, mm	1.0
Tritium inventory in lithium, kg	8.0
Mole % tritium in lithium	0.033

*Aspects of model associated with the diffusional process. These are of indirect importance to present study.

3. PROCEDURE

3.1 General System Description

Table 1 summarizes the important operating parameters of the chosen reactor model (1, 2). This is the same model as used by Fraas (2) in determining the feasibility and cost of a diffusional recovery system.

The optimum tritium concentration in the lithium represents a balance between the cost of tritium inventory and the cost of recovery at the corresponding concentration. There is also the constraint that the tritium concentration has to be at a level where the niobium structural damage will not be excessive. This constraint defines the maximum tritium concentration which, for the case of the reactor model chosen, is approximately 0.04 mole % (2).

The tritium generated in the lithium is in a highly reactive, nascent state (4), and in the temperature range 400-1000°C it combines readily with the lithium to form lithium tritide in equilibrium with a solution of tritium in lithium. The existence of tritium in the lithium in these two chemical forms is discussed in Ref. (5).

3.2 Basis for Selecting a Separation Technique

Phase equilibrium data (5) indicate that in the temperature range 800-1000°C much more tritium is present in the vapor phase than in the liquid. Thus this is an acceptable operating range. Little information is available on the rate at which this equilibrium is established. Phase equilibration rate considerations are complicated by the fact that a chemical reaction ($Li + H \rightleftarrows LiH$) can occur in either the gas or liquid phase, and that the rate of establishment of chemical equilibrium may be the controlling step in phase equilibration. Little is known about the rate of attainment of chemical equilibrium. The rate of formation of lithium tritide has been examined to a very limited extent (5, 6). The decomposition of lithium hydride has been reported to be rapid at 1000°C (4, 5, 7). (Obviously, the rate at which equilibrium is attained will strongly affect the size of any separation equipment.)

Recovery of tritium from a stream of lithium by flash evaporation has been suggested by Johnson (4). In his analysis he assumed that vapor liquid equilibrium occurred in a one-stage flash chamber. In this system

large quantities of lithium vapor flashed along with the tritium. To allow separation of the lithium vapor from the tritium in a condenser, Johnson needed to assume that: (1) molecular tritium has a low solubility in lithium and (2) the rate of formation of lithium tritide (in the liquid phase) was very slow when compared to the holdup time in the condenser (6). Johnson concluded that if these assumptions were valid, then flash separation of tritium from lithium would be made possible by a non-equilibrium condensation of lithium from a lithium-tritium vapor stream. If Johnson's assumptions were valid, then use of fractional distillation would be unnecessary.

We can now see that the rate of reaction of lithium with tritium in the liquid phase seems to be the factor which will determine whether flash evaporation or fractional distillation is the better approach to separating tritium from lithium. Rapid reaction will necessitate the use of fractional distillation, while slow equilibration will allow the use of flash evaporation. Insufficient rate data are available for the lithium-tritium reaction, so both separation schemes have been analyzed in this work. Preliminary designs and costs were determined for both methods.

An additional problem in any vapor-liquid tritium separation technique is the separation of tritium from helium. Neutron bombardment of lithium in the blanket of a thermonuclear reactor produces both tritium and helium. Both separation techniques discussed in this report involve the enrichment of the vapor phase with tritium, after which a helium-tritium separation must be carried out. Although this separation has not been studied in depth, possible methods are discussed.

3.3 Fractional Distillation

3.3.1 General Considerations

In designing the fractional distillation process, we assumed that vapor-liquid phase equilibrium would be established rapidly. Included in this assumption is the further assumption that chemical reaction equilibrium between tritium and lithium in the liquid phase is established rapidly. The data used to estimate size and cost of a distillation system are shown in Sects. 6.3 - 6.9. The phase equilibrium diagram for the Li-T system is given in Fig. 1. This equilibrium plot was calculated for a total pressure of 31 mm Hg and at various mole fractions of helium in the vapor (see Appendices 6.2 and 6.6).

The feed to the distillation column is a small side stream from the lithium blanket. The composition of the liquid feed is:

$$x_T = 0.00033$$

$$x_{Li} = 0.99967$$

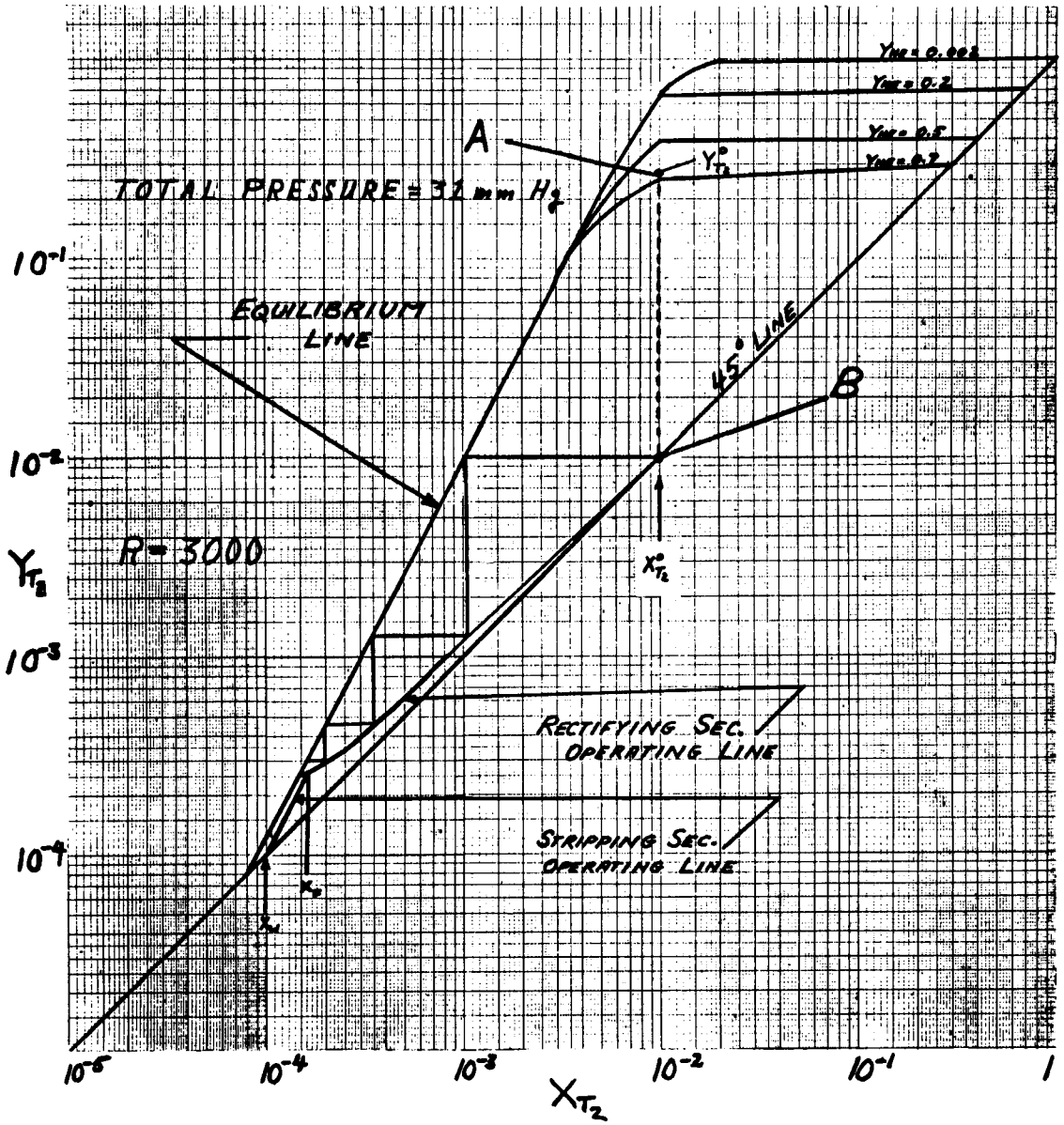


Fig. 1. Vapor-Liquid Equilibrium, T, in Molten Li

The feed was assumed to be introduced to the column as a saturated liquid ($T = 1800^{o}F$, $P = 31$ mm Hg) with helium probably present as bubbles of gas in the liquid lithium. The required production of tritium (T) for the reactor is 1 kg/day and, at the same time, 1.33 kg/day of helium is produced. This gives a mole ratio of $He/T_2 = 2$.

3.3.2 Distillation Design Procedure

The method of McCabe and Thiele with the usual assumptions (e.g., equimolal overflow, etc.) was used (8). The system is shown schematically in Fig. 2. The product tritium, mixed with helium, is removed from the top of the column, and the column bottoms are returned to the main lithium stream. A partial condenser and a total reboiler are used.

The system constraint was that the steady state concentration of helium in the gaseous product had to be twice that of the tritium. Appendix 6.6 shows the calculation of the helium concentration in the column enriching section, and column design details are given in Sect. 6.5.

Figure 2 gives flow rates, heat loads, and heat exchanger areas for a possible distillation process. The total capital cost for the distillation tower, condenser, and reboiler as estimated from the data in Sect. 6.12 is 10 million dollars. This cost estimate neglects a probable additional cost involved in preventing diffusional loss of tritium through the lithium-potassium boiler. Such a cost might be prohibitive.

3.4 Flash Separation

3.4.1 Assumptions

In the design of the flash separator, it was assumed that it is possible to condense lithium from the tritium-lithium vapor rapidly enough to preclude a significant chemical reaction between them. In Sect. 6.1 this assumption is shown to be a poor one, based on limited data. As a result, single stage flash separation may not be possible. Even if the above assumption were valid, other serious problems (discussed in the next section) exist for the flash separation process.

3.4.2 Design Procedure

A schematic diagram for the flash vaporization system and pertinent design details are shown in Fig. 3.

A full design for a $L/V = 10$ is shown in Sect. 6.10. This design indicates that the feed temperature to a single stage flash unit would have to be about $2650^{o}F$. Piping materials which can withstand this temperature are not known and operation at these conditions is impossible.

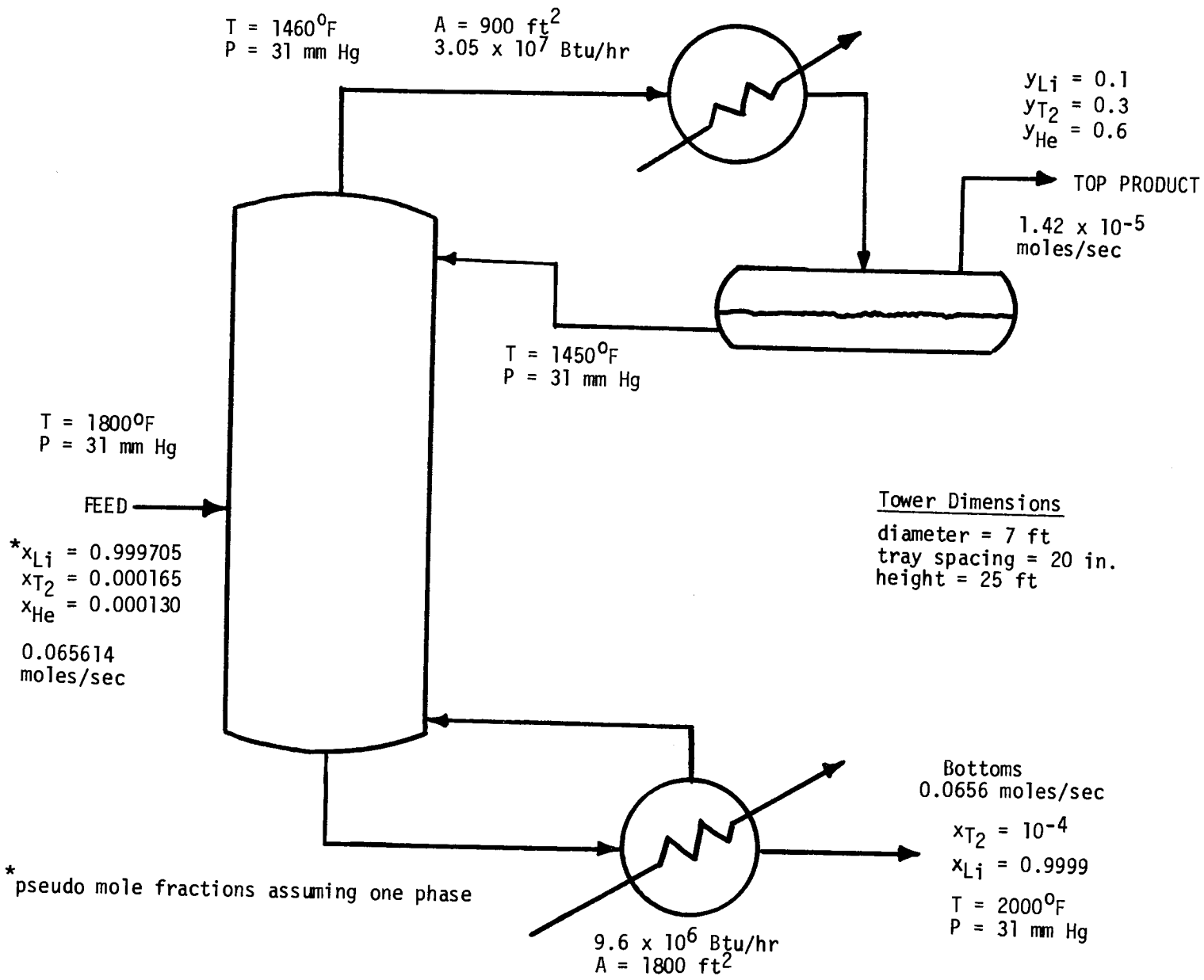
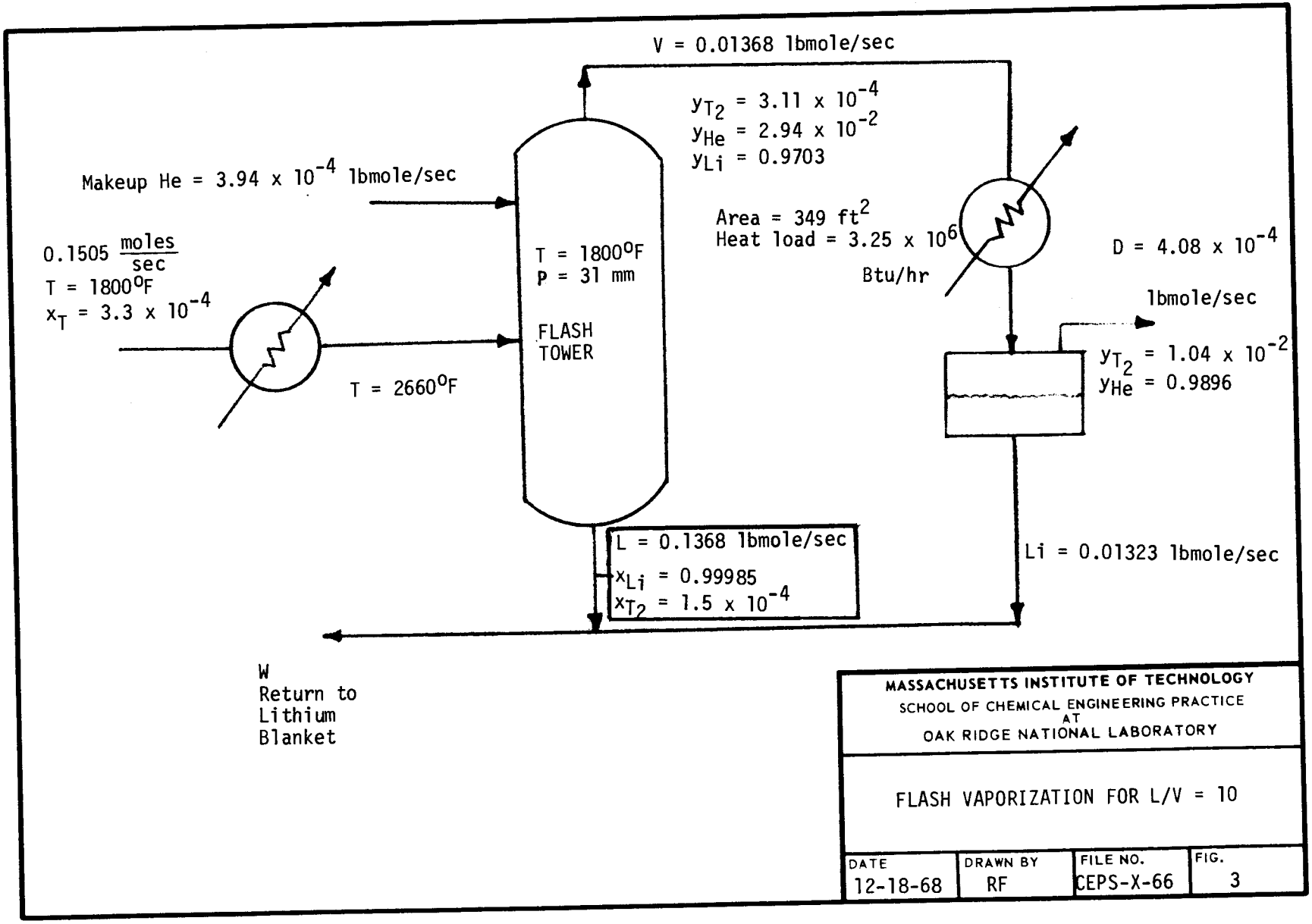


Fig. 2. Schematic Diagram of Proposed Distillation System



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FLASH VAPORIZATION FOR L/V = 10

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Figures 4 and 5 show that at higher L/V ratios both the heat load and vapor load on the separator decrease. At high L/V ratios higher feed rates are necessary. This has the advantage that the required temperature of the feed is lower. Lower feed temperature is necessary from a metallurgical standpoint, but at high L/V ratios it is necessary to recirculate greater amounts of helium to insure tritium separation. This will lead to a larger helium inventory. Even at an L/V = 10 the problem of helium-tritium separation has been greatly increased over that for the distillation process.

No attempt was made to optimize this design. The total cost of the separator and the condenser as estimated from the data in Sect. 6.12 is 2 million dollars. This estimate does not include the cost of preventing tritium losses through the Li-K boiler. This cost, as in the distillation process, may be prohibitive.

3.5 Helium-Tritium Separation

A literature search showed that the two techniques which might be used to separate tritium from helium are adsorption and diffusion. A method of tritium removal from a gas mixture consisting primarily of helium was studied on a laboratory scale (9) using activated charcoal as an adsorbent. The tritium enriched gas was then desorbed from the charcoal by heating, reacted with oxygen using a catalyst (hopcalite) and the tritiated water was separated from the helium using a molecular sieve. The tritium could then be removed from the water by electrolysis.

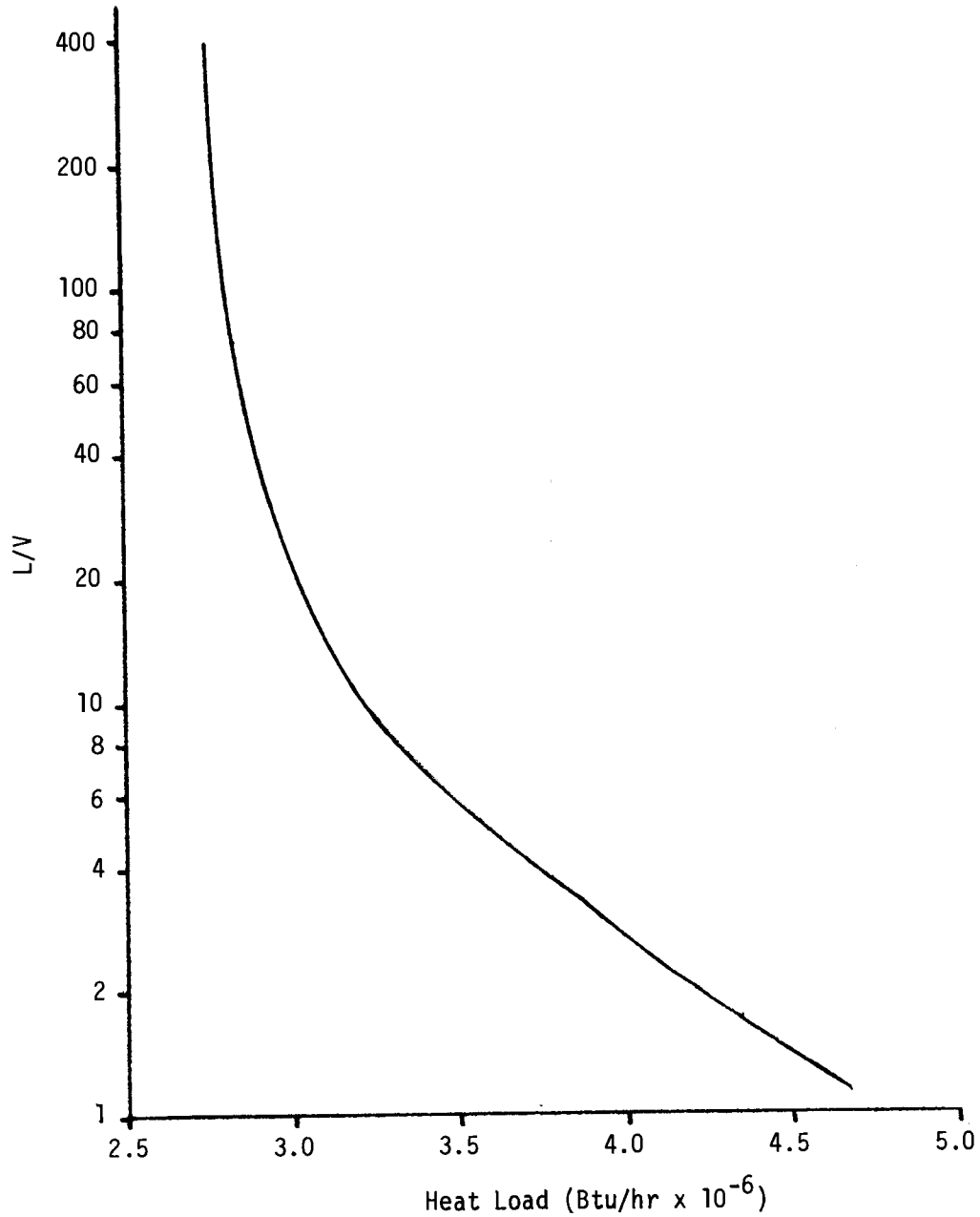
Chemisorption of tritium in a helium stream on palladium is another possible method of separation. Although helium will also be adsorbed by the palladium, use of several palladium adsorption stages should permit a good separation. The drawback of such a process would be that it is batchwise since desorption of the tritium would have to be performed by heating the palladium. Using a fluidized bed of palladium pellets, operated as shown in Fig. 6, may allow continuous operation. This process has not been evaluated.

Liquid distillation separation of tritium and helium has been studied (10), but the low operating temperatures involved ($1^{o}K$) make this system unattractive.

4. DISCUSSION OF RESULTS

4.1 General Discussion

Table 2 summarizes the operating parameters considered and the results obtained for the two tritium recovery systems studied. From an economic standpoint the recovery of tritium by flash vaporization appears to be more



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HEAT LOAD AS A FUNCTION OF L/V
FOR FLASH SEPARATOR

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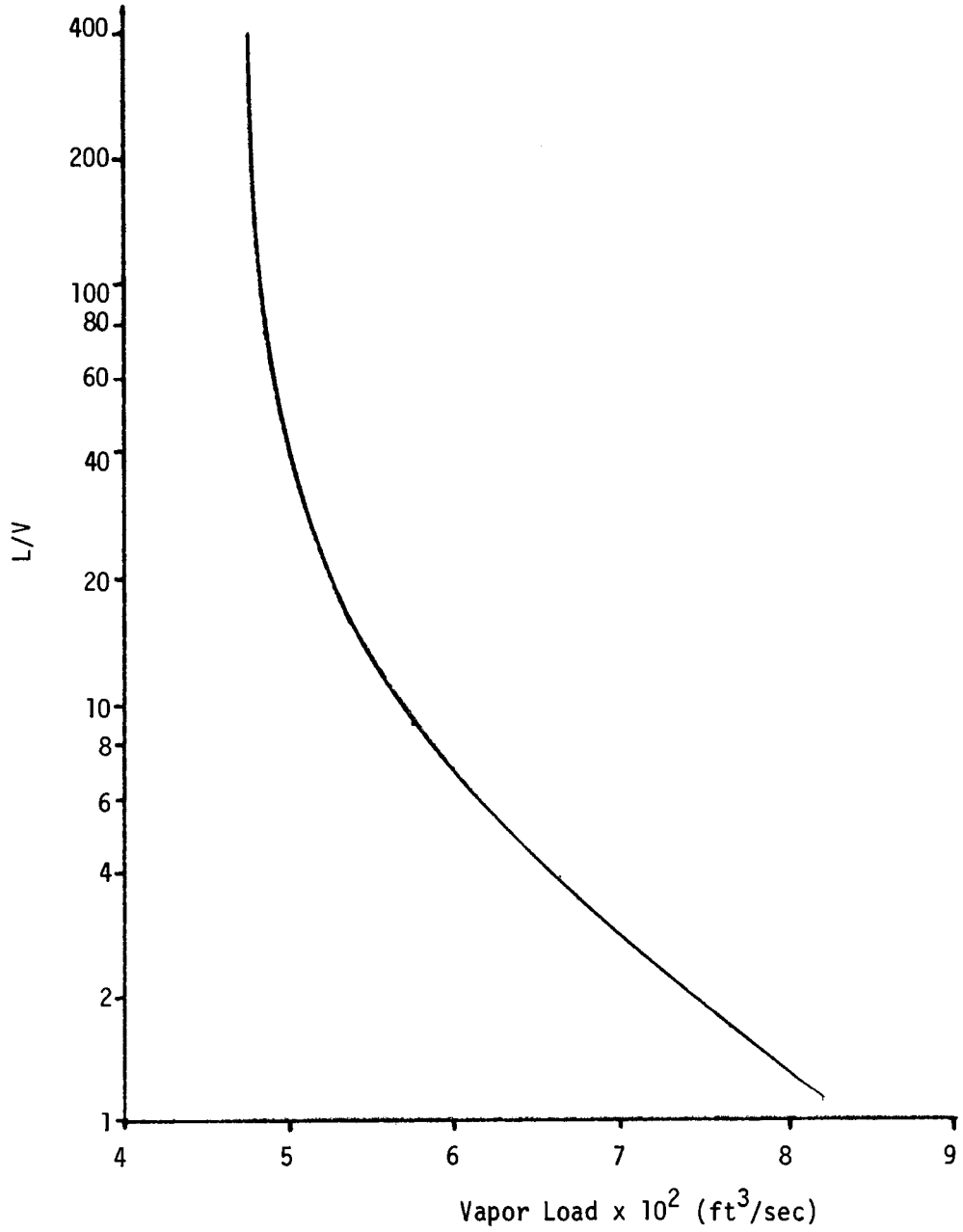

T = 1800°F

P = 31 mm Hg

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VAPOR LOADING VERSUS L/V RATIO IN FLASH SEPARATOR

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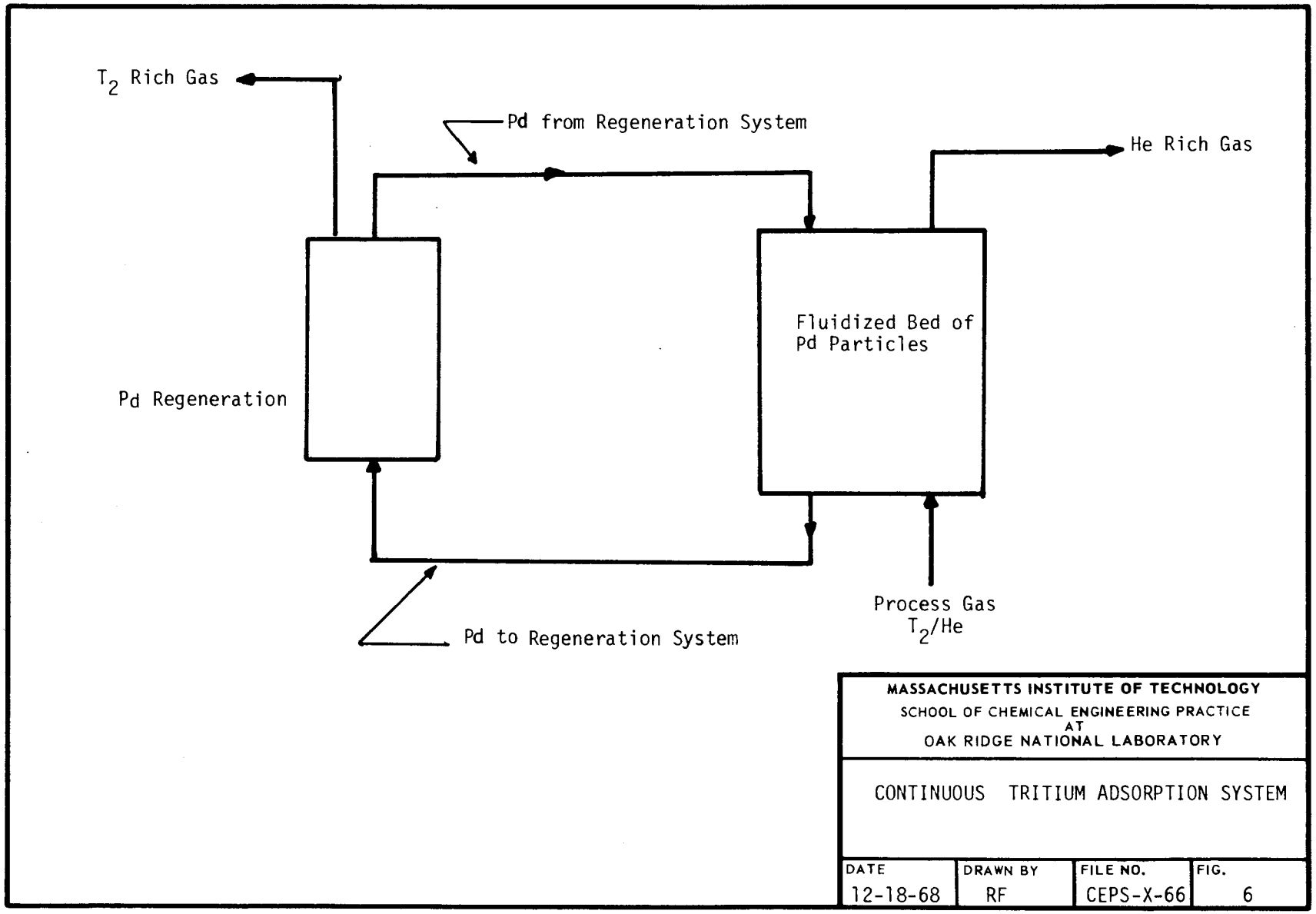
T_2 Rich Gas
Pd from Regeneration System
He Rich Gas
Fluidized Bed of Pd Particles
Pd Regeneration
Process Gas T_2/He
Pd to Regeneration System
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CONTINUOUS TRITIUM ADSORPTION SYSTEM
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FIG. 6

Table 2. Operating Parameters and Costs

System of Parameters	Method of Separation: Distillation	Method of Separation: Flash Separation
Feed rate	0.06562 moles/sec	0.1505 moles/sec
Feed pressure	31 mm Hg	1 atm
Feed temperature	1800°F	2660°F
Heat load	9.6×10^6 Btu/hr	-
Condenser load	10.5×10^6 Btu/hr	3.25×10^6 Btu/hr
Condenser area	900 ft^2	349 ft^2
Reboiler area	1800 ft^2	-
Distil., O/D; Flash Sep., L/V	3000	10
Tower pressure	31 mm Hg	31 mm Hg
Approximate tower temperature	1800°F	1800°F
Tritium inventory in equipment	∿ $140,000	∿ $16,000
Cost	∿ $10,000,000	∿ $2,000,000

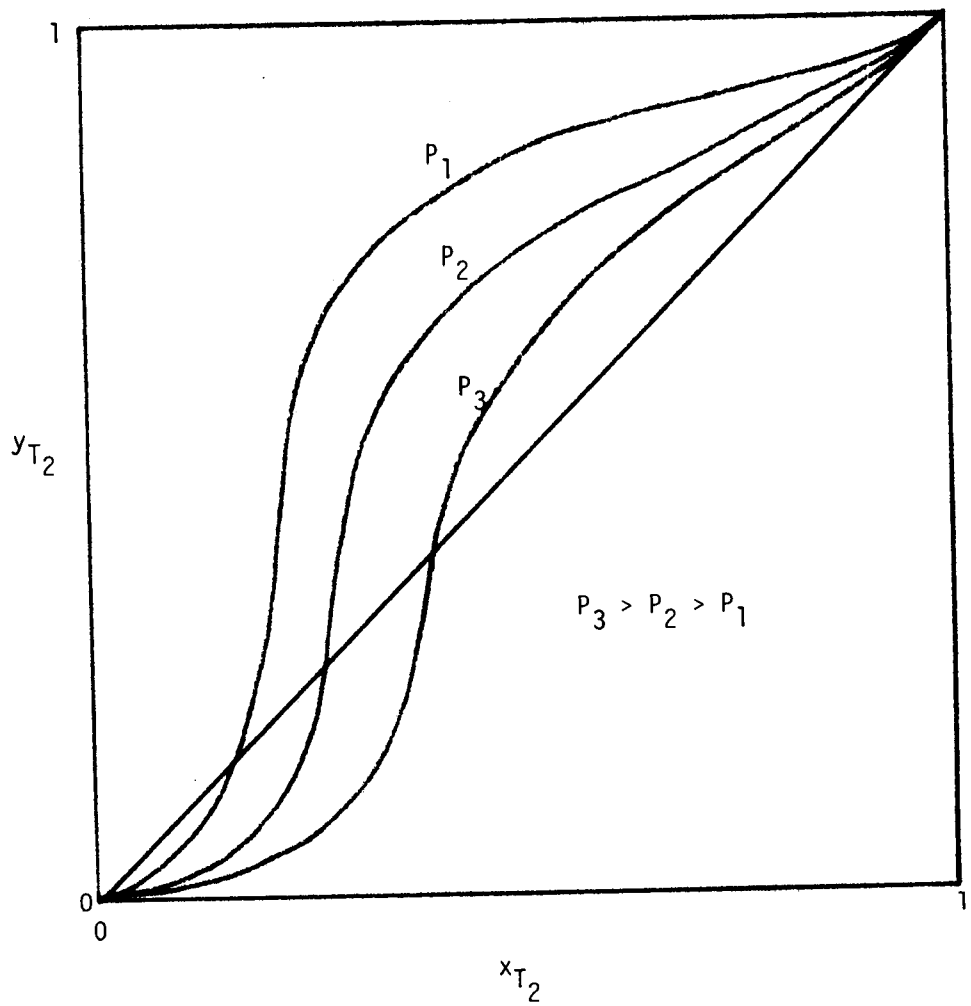
attractive than recovery by fractional distillation. However, three points relating to the validity of this economic comparison should be made. First, neither cost estimate includes the important cost of coating the niobium tubes between the lithium and boiling potassium to prevent loss of tritium by diffusion from the lithium through the walls to the potassium. Second, the feasibility of flash vaporization as a tritium recovery technique is predicated on the assumption that a mixture of lithium and tritium vapor can be separated by a non-equilibrium condensation in which no tritium reacts with the condensed lithium. In the present flash evaporator design this assumption is not valid because the very large surface area of the condenser (350 ft^2) gives too much surface on which the reaction between tritium vapor and liquid lithium can occur. {This large condenser area, which is considerably more than is required based on heat transfer considerations (30 ft^2), is needed because of a vapor load limitation resulting from our use of 5-ft-long condenser tubes (see Appendix 6.7).} For a condenser of area 350 ft^2 operated at conditions similar to those in our condenser, the data of Albert and Mahé (6) indicate that more than one third of the tritium entering the condenser would react with liquid lithium to form lithium tritide in the liquid phase (see Appendix 6.11.1). A decrease in the vapor load on the condenser (allowing a smaller condenser) could be achieved by increasing the flash tower pressure. However, as shown schematically in Fig. 7, as tower pressure is increased, the vapor-liquid equilibrium is adversely affected, and the separation becomes worse.

The third consideration which should be noted is that in the proposed flash evaporator design, the feed must be introduced at a temperature of 2660°F, which is unacceptably high from a metallurgical standpoint. As indicated in Sect. 3.4, a reasonable feed temperature can be achieved only by introducing a much greater amount of recycle helium to the flash separator. This would result in a vapor loading problem as well as possibly a concentration polarization problem in the condenser. These factors would all tend to increase the capital cost substantially.

For a distillation system, the problems outlined for the flash evaporator are overcome because of the high enrichment that is obtained in the top of the column. Based on present knowledge, distillation is the only alternative to the diffusion system (2). The approximate capital cost for the distillation system (reported in Table 2) of 10 million dollars is dependent on the rate at which vapor-liquid equilibrium can be attained. This is an area of uncertainty which must be more clearly defined before distillation can be compared meaningfully with the diffusional recovery scheme.

4.2 The Effects of Operating Parameters on Distillation

In the design of the distillation recovery system, no effort was made to obtain optimum operating conditions (a balance between tritium inventory and cost of separation). An examination of the effects of the system parameters on the McCabe Thiele diagram (Fig. 1) will show the direction in which to proceed in this optimization.



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EFFECT OF PRESSURE ON VAPOR-LIQUID EQUILIBRIUM OF Li-T_2 SYSTEM

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4.2.1 Pressure

A pressure increase will decrease the vapor load on the overhead condenser and will reduce the size of this unit. However, the equilibrium becomes less favorable (the curve in Fig. 7 moves to the right and down), and thus the azeotrope is formed at higher tritium mole fractions. The maximum pressure possible is thus the pressure at which the equilibrium line intersects the 45° line at the feed composition. At this pressure an infinite number of stages would be needed to achieve the desired separation. For a pressure increase the decrease in condenser costs would have to be balanced against the cost of a larger tower.

A pressure decrease will increase the vapor load on the condenser. However, the equilibrium becomes more favorable (see Fig. 7), so that for the same degree of separation, fewer stages will be needed. At low pressure, tower operating temperature will have to be decreased to eliminate lithium from the final product. At lower temperatures, the rate of decomposition of lithium tritide will be lower, perhaps necessitating an increase in tower height. For a desired degree of separation, as the concentration of T_2 in the feed stream is decreased, the pressure and temperature in the column must be decreased.

4.2.2 Temperature

The rate of formation and decomposition of lithium tritide will be affected by temperature. Operation at higher temperature will cause an increase in the heat load on the condenser and the vapor load in the column. At low enough temperatures much less expensive construction materials could be used (see Sect. 6.12).

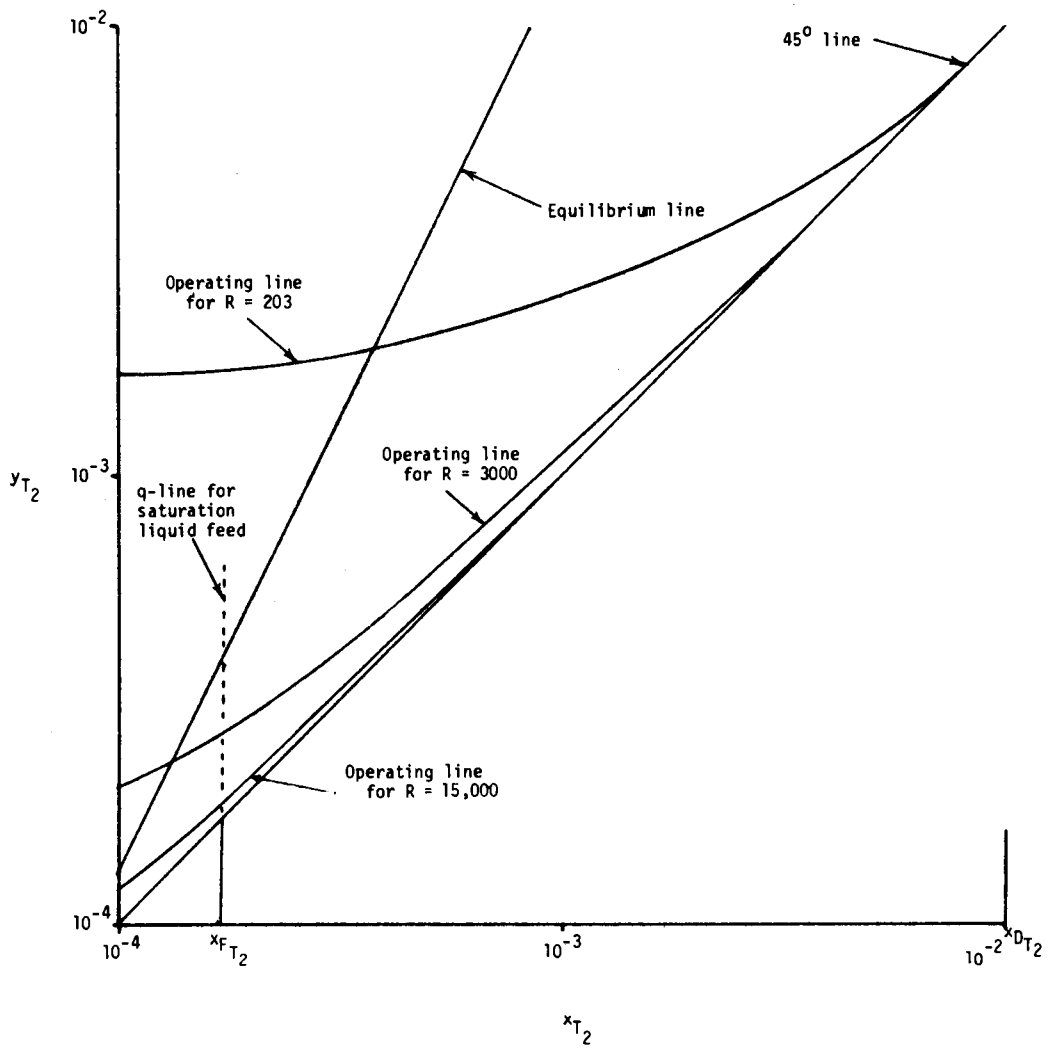
4.2.3 Reflux Ratio

An increase in reflux ratio will cause a decrease in the required tower size, but the column vapor loading will increase. Increased reflux ratio will, therefore, increase the heat load on both the reboiler and condenser, while simultaneously decreasing the capital cost of the column.

If it is assumed that the feed stream is a saturated liquid at its bubble point, then there is a lower limit on the reflux ratio. Figure 8 shows that this lower limit must be between R = 200 and R = 3000 since the q-line and the operating line must intersect below the equilibrium line (8).

4.2.4 Feed Conditions

At a fixed feed composition of $x_T = 3.3 \times 10^{-4}$ (see Sect. 2), variations in feed pressure offer no advantage so the feed should be introduced at the tower pressure. The state of the feed has a strong influence on tower and auxiliary unit sizes. If the feed is introduced as cold liquid,



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EFFECT OF DIFFERENT REFLUX RATIO ON POSITION OF OPERATING LINE (with $x_{D_{T_2}} = 10^{-2}$)

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lower reflux ratios are possible but the vapor load on the reboiler is increased. Introduction of feed as a saturated or superheated vapor will cause an increase in condenser load.

From the discussion in Sect. 4.2, an algorithm can be developed for cost versus operating conditions. Optimization of the tower can then be accomplished.

4.3 Comparison of Distillation and Diffusional Recovery System

Fraas (2) determined the cost of a system for recovering tritium by diffusion through a niobium window. Assuming that the capital cost of the system was that of the niobium tube bank, he calculated it to be approximately $\$4 \times 10^6$. The tritium inventory in the lithium is approximately 8 kg, valued at approximately $\$10 \times 10^6$. Assuming the same inventory maintained for distillation as for diffusion, the capital cost for the distillation system is approximately $\$10 \times 10^6$. This cost includes the cost of the tower, reboiler, and condenser, but neglects the cost of coating the tubes of the potassium boiler to prevent loss of tritium by its diffusion from the lithium to the potassium. A tritium inventory exists in the condenser, reboiler, and distillation tower which is valued at about $140,000.

Based both on capital cost and tritium inventory, the distillation system is more expensive than the diffusional process. In addition, the diffusion process appears more economical than the distillation process when they are compared for a lower tritium inventory in the lithium blanket (see Appendix 6.13). However, any improvement in the design of the condenser and reboiler for the distillation system to allow reduction of their size would make this process more competitive with the diffusion scheme. A further consideration which makes both the distillation and flash processes less attractive is the cost of keeping the tritium from diffusing out of the lithium blanket through the lithium-potassium boiler. This will represent an additional capital cost to the flash and distillation processes. No consideration has been given to the extra cost which this would entail.

5. CONCLUSIONS AND RECOMMENDATIONS

1. With the assumptions made regarding vapor-liquid equilibrium and formation and decomposition rates of lithium tritide, the cost of a distillation system is more than twice that of a diffusion recovery system.

2. The diffusion process is more versatile than the distillation process since lower tritium inventories can be achieved with lower capital expenditures.

3. Before an accurate design and cost analysis of the distillation system can be performed, reliable data on rate of formation and decomposition

of lithium tritide (or hydride) are needed. The temperature and pressure range of interest are 400-1000°C and 20-760 mm Hg.

4. Equilibrium data for the low concentration range ($x_T = 3.3 \times 10^{-4} \rightarrow 0.10$) of tritium in lithium are necessary before a definitive design for the distillation tower may be attempted.

5. The condenser represents a considerable proportion of the cost of the distillation system.

6. A problem exists in fabricating the distillation equipment with tungsten coated niobium.

7. Based on presently available lithium-tritium equilibrium and rate data, flash separation is not feasible.

It is recommended that phase equilibrium and reaction rate data be obtained for the lithium-tritium system to verify the above conclusions. Cost estimates for fractional and flash distillation should be made for low enough temperatures that tungsten-coated niobium construction materials are not needed, since these represent a major cost.

6. APPENDIX

6.1 Fractional Crystallization

Figure 9 is a phase diagram for a lithium-lithium hydride system. We assumed that the lithium tritide phase diagram was the same as that for lithium hydride. Since the lithium stream from the reactor is only 0.033 mole % tritium, the first material to crystallize out as temperature is decreased would be pure lithium (at $T \sim 186^{o}C$). However, pure lithium could only be removed until the eutectic composition is obtained at 3% LiH. Other techniques would then be needed to separate the 3% LiH. Also, the difference in melting point between pure lithium and the 3% LiH eutectic is only $1.8^{o}C$ (11). Consequently, temperature control accurate enough to prevent crystallization of the 3% LiH eutectic mixture would be impossible. Even if accurate enough temperature control were possible, the crystals of lithium would contain 10^2-10^3 ppm of occluded tritium (11).

For these reasons it is not apparent that any significant separation could be achieved between lithium and lithium tritide by fractional crystallization.

6.2 Vapor-Liquid Equilibrium Data

6.2.1 Calculational Procedure

Phase equilibrium data were obtained from the dissociation pressure isotherms of the lithium-lithium hydride system given by C.E. Messer (5). Since the mole fraction of tritium in the lithium is required to be very small (0.00033), it was necessary to extrapolate the data into the region of interest. The data in Ref. (5) for pressures of 10 to 100 mm Hg indicate that the concentration of dissolved tritium varies at the square root of the tritium partial pressure. It was assumed that this relationship holds at the low pressures of interest in this study. The justification for this assumption is discussed at the end of this section.

The system under consideration contained two phases and three compounds (T_2, Li, He), and thus from the phase rule there are $\psi = C + 2 - P = 3 + 2 - 2 = 3$ degrees of freedom.

Using the data of Ref. (5) and the dependence of the tritium concentration in the liquid on the square root of the tritium partial pressure, equilibrium curves for different values of y_{He} (mole fraction of helium in the vapor phase) can be obtained. By choosing x_{T_2} (mole fraction of tritium in the liquid), π (the system pressure), and y_{He}, the system is fully defined, and the equilibrium temperature and y_{T_2} (mole fraction of tritium in the gas) can be calculated.

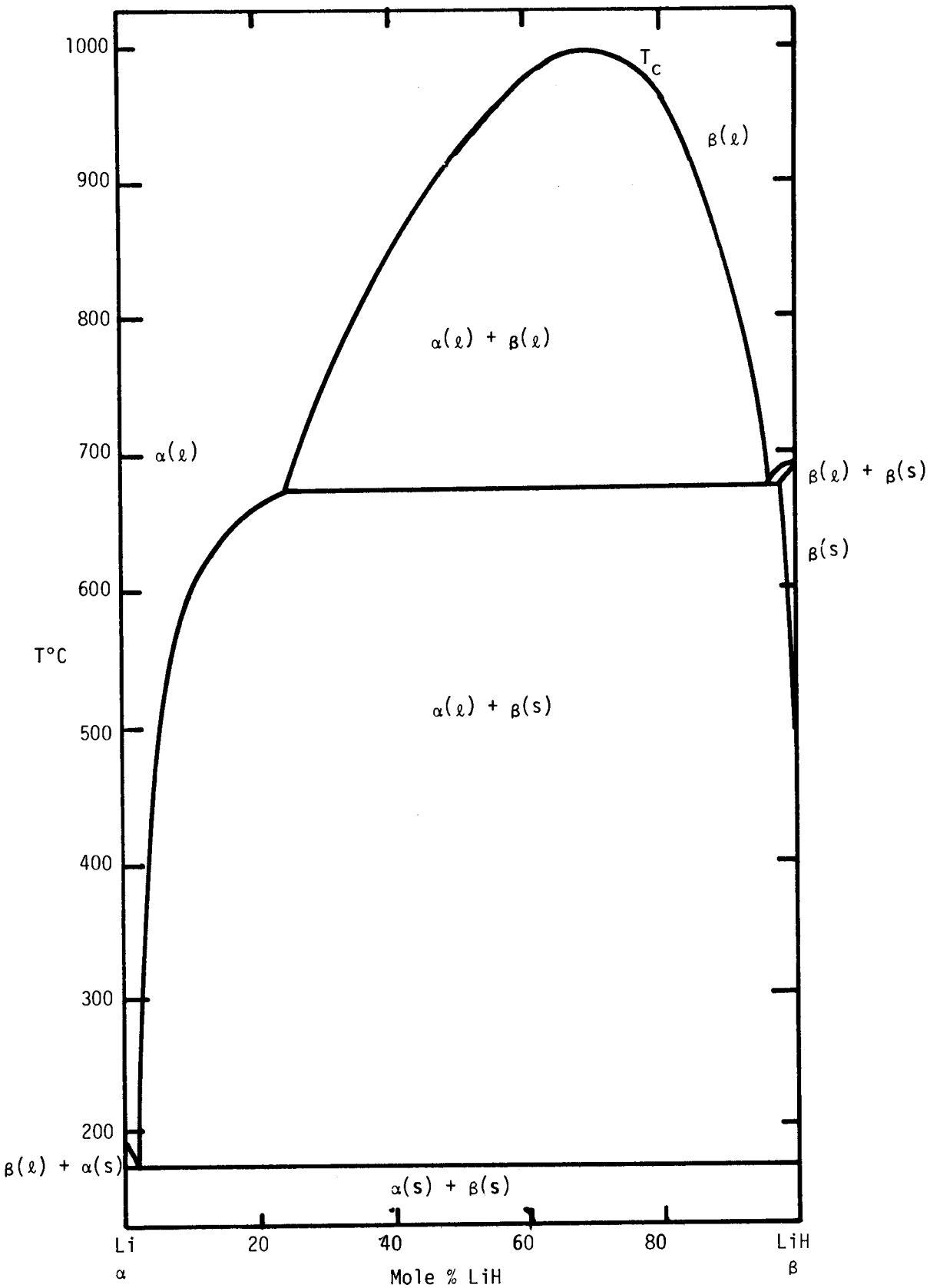


Fig. 9 . Phase Diagram (Schematic) Lithium-Lithium Hydride System (8)

A vapor-liquid equilibrium diagram was constructed for a system pressure of π = 31 mm Hg and for a range of y_{He} from 0.002 to 0.7. This is shown in Fig. 2. A schematic diagram showing the effect of system pressure on the equilibrium curves is given in Fig. 9.

The variation of y_{He} had a noticeable effect only at high values of x_{T_2} (>3 x 10^{-3}), since the accuracy of the extrapolated data was not sufficient to permit different equilibrium lines to be drawn at low values of x_{T_2}.

The equilibrium constant K (which is a function of temperature) in the relationship $x_T = K\sqrt{p_{T_2}}$ could not be accurately determined and was assumed to be constant with temperatures in the range 900-1000°C. In practice the same K (at 1000°C) was used until the x_T value was high enough to enable the partial pressure of T_2 to be read from the dissociation isotherms of Ref. (5).

6.2.2 Sample Calculation

system pressure = 31 mm Hg

mole fraction helium, y_{He} = 0.002

Assume that,

$$x_T = K\sqrt{p_{T_2}} = K\sqrt{\pi y_{T_2}} \qquad (1)$$

at low tritium concentrations. {Dissociation isotherms (5) were used at high tritium concentrations.} The tritium concentration in the lithium at 1000°C is about 1 mole % for a tritium partial pressure of 10 mm Hg (5).

$$0.01 = K\sqrt{10}$$

$$K = 3.16 \times 10^{-3} \text{ mm Hg}^{-1} \text{ at } 1000^{\circ}\text{C}$$

From Eq. (1) with $x_T = 2x_{T_2}$,

$$y_{T_2} = \frac{4(x_{T_2})^2}{\pi K^2}$$

for

$$x_{T_2} = 10^{-4}$$

Assume T = 1000°C,

$$y_{T_2} = \frac{4 \times 10^{-8}}{31 \times 10^{-5}} = 1.29 \times 10^{-4}$$

$\pi y_{Li} = x_{Li} P_{Li}^{vp}$ {assuming Raoult's law at this high lithium concentration; at lower lithium concentrations the dissociation isotherms of Ref. (5) were used.}

$$y_{Li} = \frac{P_{Li}^{vp}}{\pi} x_{Li} \text{ (see Fig. 10 for vapor pressure curve)}$$

$$= \frac{31}{31}(0.9999)$$

$$= 0.9999$$

$$\Sigma y = 0.9999 + 0.0020 + 0.0001 \simeq 1.0$$

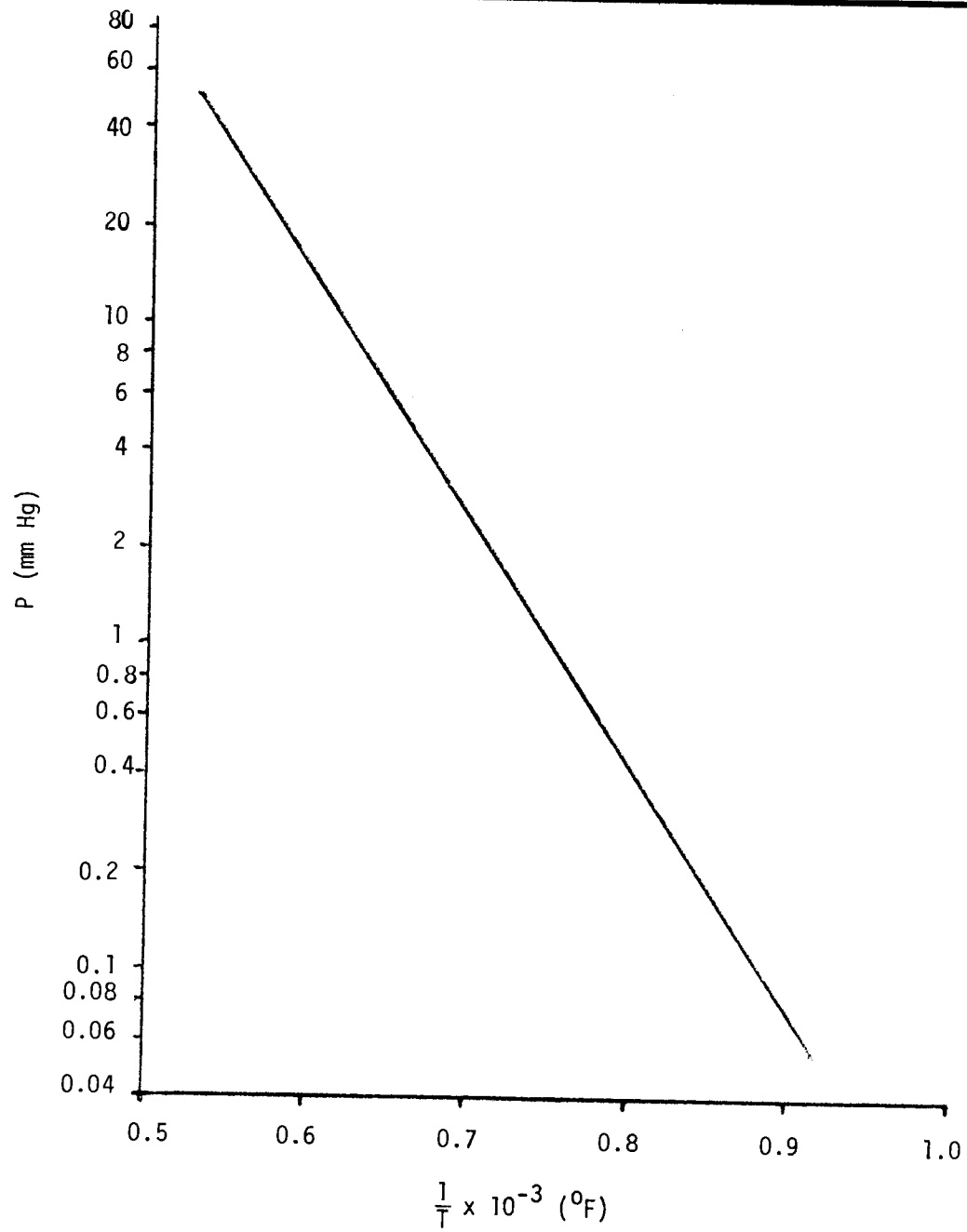
In summary, at T = 1000°C and $x_{T_2} = 10^{-4}$, $y_{T_2} = 1.29 \times 10^{-4}$.

The equilibrium results for y_{He} are summarized below

π = 31 mm Hg y_{He} = 0.002

x_{T_2}	y_{T_2}	T°C
10^{-4}	1.29×10^{-4}	1000
10^{-3}	1.3×10^{-2}	1000
3×10^{-3}	0.116	990
5×10^{-3}	0.322	950
10^{-2}	0.805	850
5×10^{-2}	0.967	825

The procedure above was repeated for different values of y_{He}. All equilibrium curves are shown in Fig. 2.



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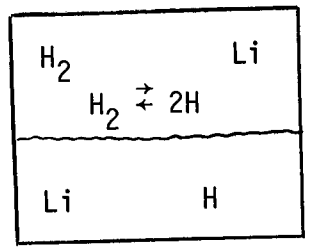
VAPOR PRESSURE VS TEMPERATURE
CURVE FOR LITHIUM (9)

DATE	DRAWN BY	FILE NO.	FIG.
12-18-68	RF	CEPS-X-66	10

6.2.4 Limitations in Using $x_T = K\sqrt{p_{T_2}}$ in Low Concentration Regions of x_T

Assume the following model for system:



where the reaction: $H_2 \rightleftharpoons 2H$ occurs in the gas phase. The equilibrium constant for the above reaction may be written in terms of fugacities as:

$$K = \frac{(f_H^V)^2}{f_{H_2}^V} \tag{2}$$

At equilibrium,

$$f_H^V = f_H^L \tag{3}$$

but,

$$f_H^V = \sqrt{K\, f_{H_2}^V} \quad \text{from Eq. (2)} \tag{4}$$

Assuming an ideal solution of ideal gases,

$$f_{H_2}^V = \pi y_{H_2} \tag{5}$$

From Eqs. (4) and (5),

$$f_H^V = \sqrt{K\pi y_{H_2}} \tag{6}$$

We can express

$$f_H^L = \gamma_H x_H H_H \text{ (modified Henry's Law)} \tag{7}$$

Thus, from Eqs. (3), (6), and (7)

$$\gamma_H x_H H_H = \sqrt{K\pi y_{H2}} \tag{8}$$

giving

$$x_H = K' \sqrt{\pi y_{H2}} = K' \sqrt{p_{H_2}} \tag{9}$$

where:

$$K' = \frac{\sqrt{K}}{\gamma_H H_H} \tag{10}$$

But,

$$K = \text{fcn}(T)$$

$$\gamma = \text{fcn}(T,P,x)$$

$$P^{vp} = \text{fcn}(T)$$

$$H = \text{fcn}(T)$$

Hence,

$$K' = \text{fcn}(T,P,x)$$

However, the dependence of γ on pressure is very weak. Since,

$$\left(\frac{\partial \ \ell n \ \gamma_i}{\partial P}\right)_{T,x_i} = \frac{\overline{V}_i - \overline{V}_i^o}{RT} \tag{11}$$

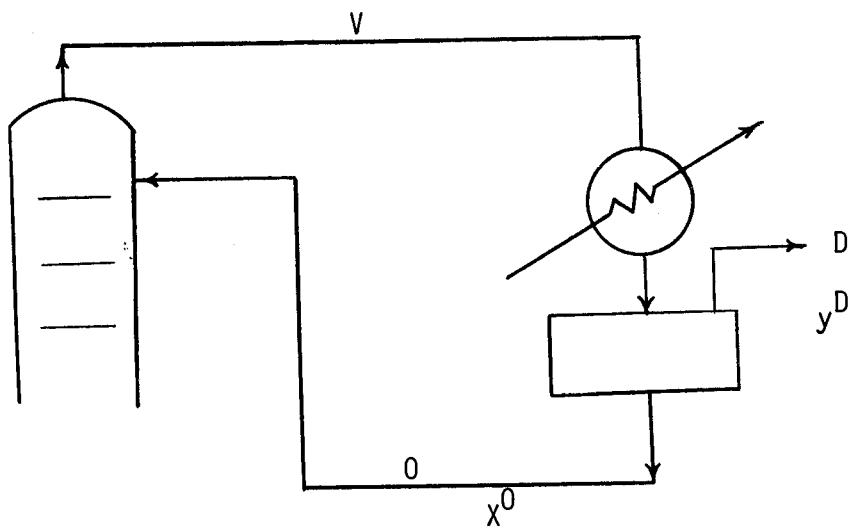
where $\gamma_i \to 1$ as $x_i \to 0$ (modified Henry's Law). Thus, $K' = f(T,x)$ and if the dependence on x is weak, the relationship

$$x_H = K \sqrt{p_{H_2}}$$

where $K = f(T)$, is reasonable. No reliable data are available to test the dependence of K on composition.

6.3 Calculation of Vapor Load and Minimum Reflux Ratio for Different Operating Conditions

For a chosen mole fraction tritium in the overhead condensate ($x^0_{T_2}$), the mole fraction tritium in the distillate product vapor ($y^D_{T_2}$) at 31 mm Hg can be determined from Fig. 2. Since there is a required rate of production of tritium, the molal flow rate (D) of the product vapor stream can be calculated by $(D)(y^D_{T_2})$ = required rate of production (moles/sec of T_2). Then, at different reflux ratios (R), the total vapor load (V) on the tower can be determined from a mole balance:



$$V = O + D$$

$$V = D(1 + R)$$

where: $R \equiv O/D$

Since the temperature at the top of the tower is approximately 800°C and the total pressure is 31 mm Hg, the volumetric flow rate can be determined.

<u>Sample Calculation</u>

For $x^0_{T_2} = 10^{-3}$, $y^D_{T_2}$ from the equilibrium plot is 10^{-2}.

production rate of tritium = 1 kg/day = 0.425×10^{-5} lb-moles/sec

So $(D)(y^D_{T_2}) = 0.425 \times 10^{-5}$ moles/sec

$$D = 0.425 \times 10^{-3} \text{ moles/sec}$$

At a reflux ratio of 5, vapor load V is given by,

$$V = D(1 + R)$$

$$= 0.425 \times 10^{-3} (1 + 5) = 2.55 \times 10^{-3} \text{ moles/sec}$$

Since one mole occupies 359 ft^3 at STP, at 800°C and 31 mm Hg pressure, one mole occupies $359(\frac{1073}{273})(\frac{760}{31}) = 34{,}700 \text{ ft}^3$.

$$V = (2.55 \times 10^{-3} \text{ moles/sec})(34{,}700 \text{ ft}^3/\text{mole}) = 88.5 \text{ ft}^3/\text{sec}$$

In a similar manner the vapor load was determined for reflux ratios of 54, 276, 1332, and 1663. The calculation was then repeated for $x^0_{T_2} = 2 \times 10^{-3}$, 5×10^{-3}, and 10^{-2}. The results are shown in Fig. 11.

These vapor loads are for the section of the column above the feed. If the feed is introduced as a liquid below its boiling point, the vapor load in the bottom section of the column will be greater.

The operating line for the enrichment section of the tower is given by:

$$y^n = \frac{R}{R + 1} x^{n+1} + \frac{1}{R + 1} y^D \qquad (12)$$

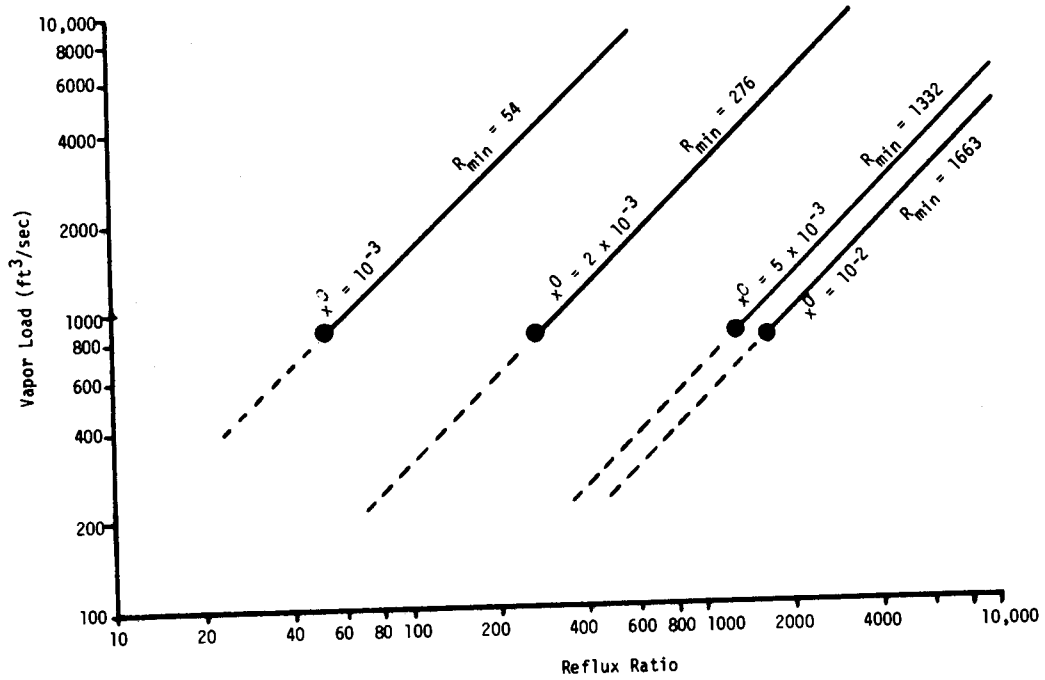
The minimum reflux ratios for operation with saturated liquid feed are found as follows. Since the operating lines for saturated liquid feed must intersect at x^F on the equilibrium curve, the value of y^F for the feed of $x^F = 1.6 \times 10^{-4}$ is easily determined. From Fig. 1 $y^F = 3.4 \times 10^{-4}$. Substituting these values in Eq. (12) for a required x^D, the value of R, the minimum reflux ratio, can be calculated.

$$R_{min} = \frac{y^D - y^F}{y^F - x^F}$$

For example at $y^D_{T_2} = 10^{-2}$,

$$R_{min} = \frac{10^{-2} - 3.4 \times 10^{-4}}{3.4 \times 10^{-4} - 1.6 \times 10^{-4}} = 53.7$$

This calculation was repeated for $y^D_{T_2}$ of 3.5×10^{-2}, 0.17, and 0.3. The values of the minimum reflux ratios are indicated on Fig. 11.



— Region where column is operable with saturated liquid feed.

-- Region of column operation with cooled feed

● Minimum reflux ratio for saturated liquid feed

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VAPOR LOAD AS A FUNCTION OF REFLUX RATIO FOR COLUMN ABOVE FEED

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6.4 Calculation of Tower Diameter for Different Tower Vapor Loads

A modified form of the Sanders-Brown equation was used to determine maximum allowable vapor velocities through the distillation tower. The relationship given by Lowenstein (13) is:

$$v_{max} = \lambda(D) \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} \tag{13}$$

where:

v_{max} = maximum allowable velocity, ft/sec

$\lambda(D)$ = empirical constant which is a function of tray spacing

ρ_L = liquid density, lb/ft^3

ρ_V = vapor density, lb/ft^3

Since column diameters were not expected to exceed 7 ft, a tray spacing of 20 in. was taken (8). From Fig. 1 of Ref. (13), $\lambda(D)$ was found to be 0.15. It was then possible to obtain v_{max}. It is desirable to have a vapor velocity equal to 65% of v_{max}.

The column diameter is given by

$$d(ft) = \sqrt{\frac{4V}{V\pi}} \tag{14}$$

where:

V = vapor load on the column, ft^3/sec

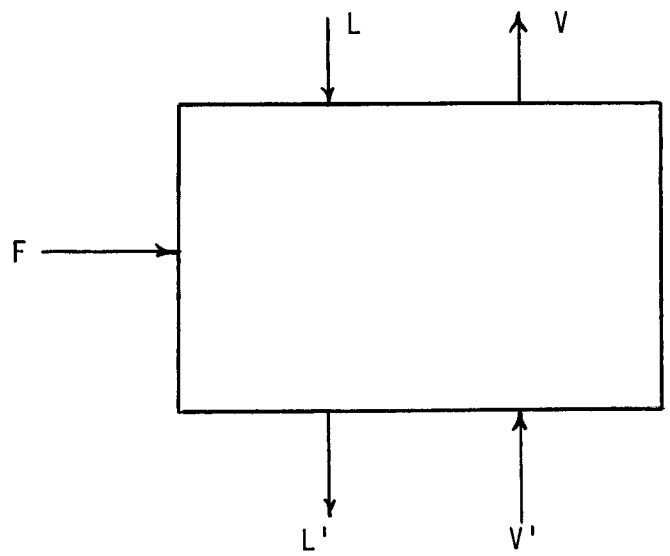
d = diameter of column

If the assumption of equal molal overflow is made and the feed is introduced as a liquid at its boiling point, the vapor load is constant throughout the tower. The helium gas is such a small fraction of the feed that its effect on the feed quality can be neglected.

Define,

$$q \equiv \frac{\text{heat to convert one mole of feed to a saturated vapor}}{\text{molar heat of vaporization}} \tag{15}$$

Consider the feed plate



where:

L, L' = liquid rates above and below feed plate, respectively

V, V' = vapor rates above and below feed plate , respectively

Thus,

$$L' = L + qF$$

$$V = V' + (1 - q)F$$

but for saturated liquid feed $q = 1$.

$$\therefore \quad L' = L + F$$

$$V' = V$$

The vapor load at the top of the tower should be used in calculating tower diameters when $q \leq 1$.

<u>Sample Calculations</u>

Assumptions:
(1) vapor is lithium
(2) temperature = 1800°F
(3) tray spacing = 20 in.
(4) feed is a liquid at its boiling point

$$\rho_L = 28 \text{ lb/ft}^3$$

One pound mole occupies:

$$359\left(\frac{760}{31}\right)\left(\frac{2260}{492}\right) \text{ ft}^3\text{/lb mole} = 40{,}500 \text{ ft}^3\text{/lb mole}$$

$$\therefore \qquad \rho_V = 7 \text{ lb/lb mole } (1/40{,}500 \text{ lb mole/ft}^3) = 1.73 \times 10^{-4} \text{ lb/ft}^3$$

From Eq. (13),

$$v_{max} = \lambda(D)\sqrt{\frac{\rho_L - \rho_V}{\rho_V}}$$

$$= 0.15\sqrt{\frac{28 - 1.73 \times 10^{-4}}{1.73 \times 10^{-4}}} = 61 \text{ ft/sec}$$

Taking 65% of this value to give desired flow rate of vapor,

$$v = 0.65\, v_{max} = 40 \text{ ft/sec}$$

Now from Eq. (14),

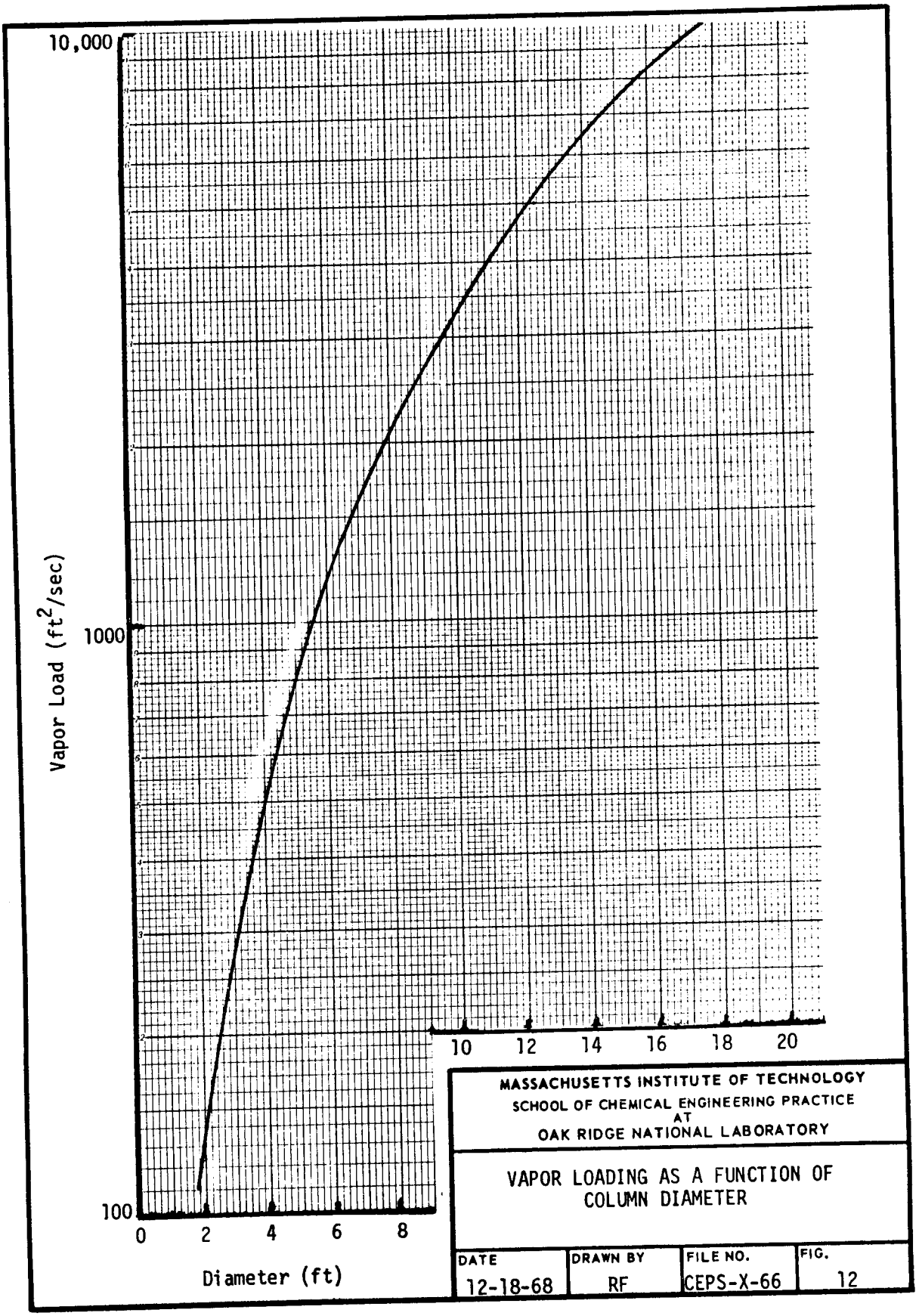
$$d = \sqrt{\frac{4V}{v\pi}} = \sqrt{\frac{4V}{40\pi}} = 0.177\, V^{\frac{1}{2}}$$

For V = 100, d = 1.77 ft. This calculation was repeated over a range of values of V and the results are shown in Fig. 12.

6.5 Distillation Tower Design

6.5.1 Tower Size

Take $x^0_{T_2} = 0.01$.


10,000
1000
100
Vapor Load (ft²/sec)
0
2
4
6
8
10
12
14
16
18
20
Diameter (ft)
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VAPOR LOADING AS A FUNCTION OF
COLUMN DIAMETER
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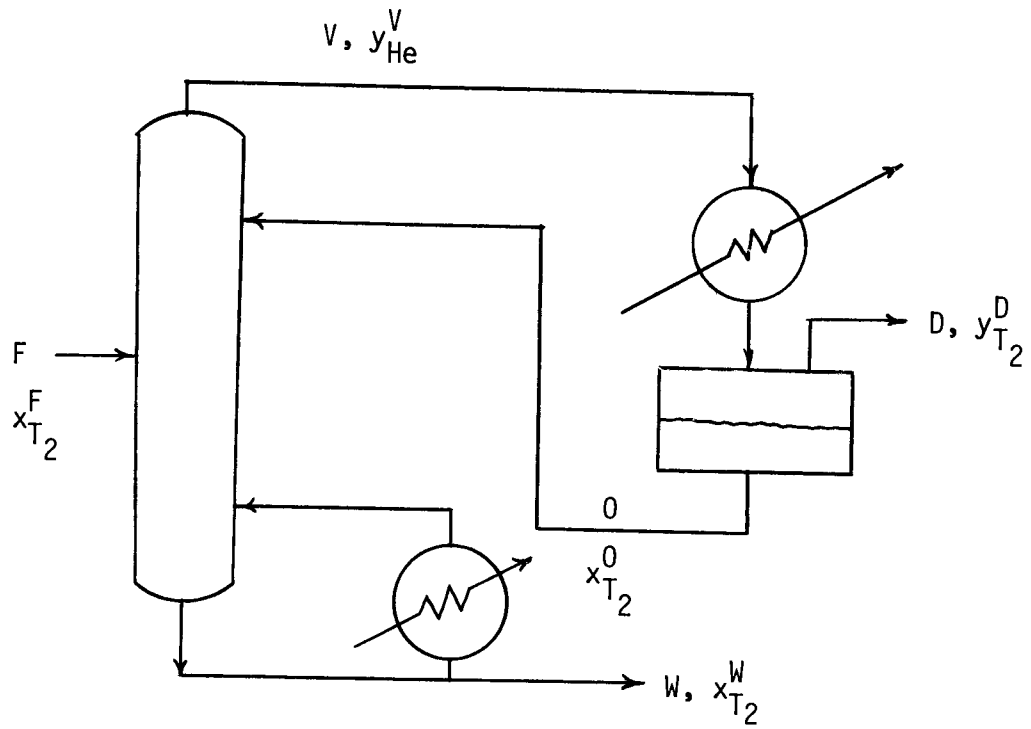
FIG.
12



To obtain $y^D_{T_2} = \frac{1}{2} y^D_{He}$ for gas in equilibrium with liquid of composition $x^0_{T_2} = 0.01$, it is found from Fig. 1 by trial and error that $y^D_{He} = 0.6$, $y^D_{T_2} = 3$. This composition is represented by point A on Fig. 1, but

$$(D)(y^D_{T_2}) = 0.425 \times 10^{-5} \text{ moles/sec } (\equiv 1 \text{ kg/day})$$

So, $$D = \frac{0.425}{0.3} \times 10^{-5} = 1.42 \times 10^{-5} \text{ moles/sec}$$

Choose $R = \frac{0}{D} = 3000$. (This allows feed at its boiling point to be used, see Fig. 8). So,

$$0 = 4.26 \times 10^{-2} \text{ moles/sec}$$

But,

$$V = 0 + D$$

$$V = 4.26 \times 10^{-6} \text{ moles/sec}$$

But,

$$(V)(y^V_{He}) = 0.85 \times 10^{-5} \text{ moles/sec } (\equiv 1.33 \text{ kg/day})$$

So,

$$y_{He}^{V} = \frac{0.85 \times 10^{-5}}{4.26 \times 10^{-2}} = 0.0002$$

This value of y_{He}^{V} applies throughout the rectifying section of the tower assuming constant molal overflow and feed introduced at its boiling point.)

The operating line for the rectifying section of the tower is given by:

$$y_{T_2} = \frac{R}{R+1} x_{T_2} + \frac{y_{T_2}^{D}}{R+1} \qquad (16)$$

This line is plotted on Fig. 1 for R = 3000 and $x_{T_2}^{0} = 0.01$. The usual McCabe Thiele construction (see Fig. 1) is started from point B using the $y_{He} = 0.0002$ line as the appropriate equilibrium curve. The total number of theoretical plates for this particular feed, reflux ratio, and separation was found to be seven.

No data are available for the expected plate efficiency for this type of liquid metal distillation. A value of 50% was assumed.

the number of actual plates is then $= \frac{100}{50}(7) = 14$

From Fig. 11 with R = 3000 and $x_{T_2}^{0} = 0.01$, the vapor load = 1450 ft^3/sec. From Fig. 12 for this vapor load a tower diameter of 6.7 ft is required.

The final approximate tower dimensions are as follows:

number of plates = 14

tower diameter = 7 ft

tray spacing = 20 in. (arbitrary)

tower height = 25 ft

feed introduced in 8th plate from the top

6.5.2 Condenser and Reboiler Requirements

For a vapor load of 1450 ft^3/sec the reboiler heat load is 9.6×10^6 Btu/hr (Fig. 14 in Sect. 6.7.2) and the condenser heat load is 3.95×10^5 Btu/hr (Fig. 18, Sect. 6.7.2). The areas for these heat exchangers are:

area reboiler = 1800 ft^2 (see Appendix 6.8)

area condenser = 900 ft^2 (see Appendix 6.7)

6.5.3 Mass Balance

$$(F)(x^F_{T_2}) = (D)(y^D_{T_2}) + (W)(x^W_{T_2}) \tag{17}$$

$$F = D + W \tag{18}$$

From Eqs. (17) and (18),

$$W = \frac{D(y^D_{T_2} - x^F_{T_2})}{x^F_{T_2} - x^W_{T_2}}$$

Plugging in appropriate values,

$$W = \frac{1.42 \times 10^{-5}(0.3 - 1.65 \times 10^{-4})}{1.15 \times 10^{-4} - 1.0 \times 10^{-4}} = 0.0656 \text{ moles/sec}$$

$$F = 0.0656 + 1.42 \times 10^{-5} = 0.0656 \quad \text{moles/sec}$$

6.5.4 Liquid Loading

For R = O/D = 3000

$$O = 4.26 \times 10^{-2} \text{ moles/sec}$$

$$L' = F + O = 0.1082 \text{ moles/sec}$$

$$= 0.754 \text{ lb/sec}$$

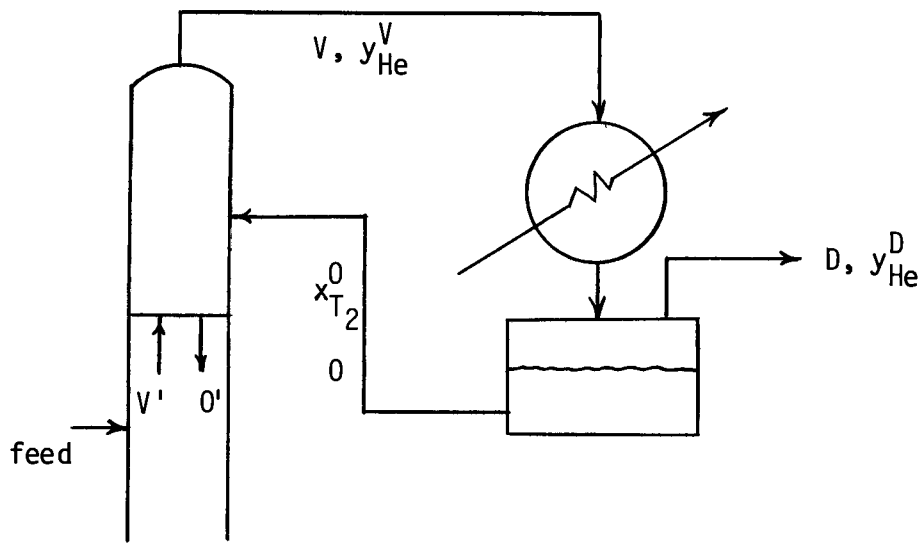
$$\rho_L = 28 \text{ lb/ft}^3$$

so $L' = 0.027 \text{ ft}^3\text{/sec}$

Since the diameter of the tower = 7 ft, $L'/d = 0.00385$ ft^3/sec-ft-diam. Ref. (12) gives a maximum value of $L'/d = 0.165$ ft^3/sec-ft-diam for single pass cross flow tray; consequently this liquid load is reasonable.

6.6 Determination of Helium Mole Fraction in the Enrichment Section of the Column and in the Condenser

A mass balance on the condenser shows



$$V = 0 + D = (R + 1)D$$

where:

$$R = \text{reflux Ratio} = 0/D$$

However, for a required helium production rate,

$$(D)(y^D_{He}) = \text{production}$$

In addition, since there is no helium in the liquid phase,

$$(y^V_{He})(V) = (y^D_{He})(D)$$

From this it can be seen that

$$y^D_{He} > y^V_{He} \text{ since } V > D$$

A material balance around the enrichment section of the column (above the feed plate) assuming equimolal overflows shows that:

$$V'y'_{He} = (V)(y^{V}_{He}) \text{ (material balance)}$$

$$V' = V \text{ (equimolal overflow)}$$

Therefore,

$$y'_{He} = y^{V}_{He}$$

The mole fraction of helium is a constant throughout the vapor in the enrichment section of the tower. For a particular value of $x^{0}_{T_2}$ there are values of y^{D}_{He} and $y^{D}_{T_2}$ which satisfy the requirement that $y^{D}_{He} = 2y^{D}_{T_2}$. For a given production rate of tritium the value of D can be obtained and the value of $(D)(y^{D}_{He})$ determined. Then for a given reflux ratio, the vapor load V in the tower and the mole fraction of helium in the tower vapor can be calculated.

6.7 Partial Condenser Size Estimation

6.7.1 Method

It is proposed to condense the vapor from the top of the distillation tower using potassium, at its bubble point, as a coolant. The operating conditions chosen are:

1. Lithium condensing on tube side at π = 31 mm Hg and T = 800°C.

2. Potassium on shell side at π = 400 mm Hg (boiling temperature = 700°C at 400 mm).

The reasons for these conditions are:

1. The condensed lithium contains some tritium, so to maintain a low inventory of tritium in the condenser, lithium condensation in the tubes rather than outside the tubes is desired.

2. Conditions on the tube side should be the same as exist on the top plate of the tower.

3. The shell side temperature and pressure for potassium are fixed.

After the operating conditions have been chosen, there are four criteria that must be satisfied:

1. Sufficient heat transfer area must be provided for the condensation of the lithium.

2. Vapor velocities on the tube side (condensing side) should be less than 300 ft/sec (14).

3. Vapor velocities on the shell side should be less than 100 ft/sec (14).

4. The condensate drainage rate from the tubes must equal the reflux flow rate (for flow continuity).

To determine which condition was limiting, area requirements for each were determined. It was found that criterion (2) specified the size of the condenser.

6.7.2 Calculation of Heat Transfer Area Required

Necessary information:

1. Heat transfer coefficient for potassium boiling on the outside of tubes; h_o.

$$h_o = 3000 \text{ Btu/hr-ft}^2\text{-}^oF \quad (15)$$

2. Heat transfer coefficient for condensing lithium on the inside of tubes; h_i.

$$h_i = 10{,}000 \text{ Btu/hr-ft}^2\text{-}^oF \quad (16)$$

3. Heat transfer coefficient for niobium, h_{Nb}.

$$h_{Nb} = \frac{k_{Nb}}{x_{Nb}}$$

where:

k_{Nb} = thermal conductivity of niobium = 415 Btu/ft -hr-^oF (3)

x_{Nb} = thickness of niobium pipe walls

Assume x_{Nb} = 0.04 in. (15).

$$h_{Nb} = \frac{415}{0.04} = 10{,}360 \text{ Btu/hr-ft}^2\text{-}^oF$$

$$\frac{1}{U} = \frac{1}{h_i} + \frac{1}{h_o} + \frac{1}{n_{Nb}} = \frac{1}{10{,}000} + \frac{1}{3000} + \frac{1}{10{,}360}$$

$$U = 1875 \text{ Btu/hr-ft}^2\text{-}^oF$$

The heat of vaporization of lithium at $800^{o}C$ is 67.8×10^{3} Btu/lb mole (17).

Assuming all the vapor from the top of the tower is lithium (since $0 \simeq V$), the heat load for several vapor loads was determined (see Fig. 13). Similar consideration was used to construct Fig. 14 for the reboiler.

Since $U\Delta T$ is known ($\Delta T = 180^{o}F$), the heat transfer area can be determined from

$$Q = UA\Delta T$$

Figure 15 shows the area requirements for different heat loads.

6.7.3 Vapor Velocity Restriction

Assume maximum vapor velocity on tube side of 200 ft/sec (must be less than 300 ft/sec). Then for different vapor loads the cross sectional area for flow is calculated by

$$A = \frac{V}{\text{velocity}}$$

where:

V = vapor load (ft^3/sec)

A = cross sectional area (ft^2)

A similar calculation can be made on the potassium side, since from the heat load the amount of potassium vaporized is also known.

The condensate drainage rate for condensates in a pipe is given by

$$Q = \frac{\rho g w \delta^3}{3\mu} \quad (18)$$

where:

Q = drainage rate, ft^3/sec

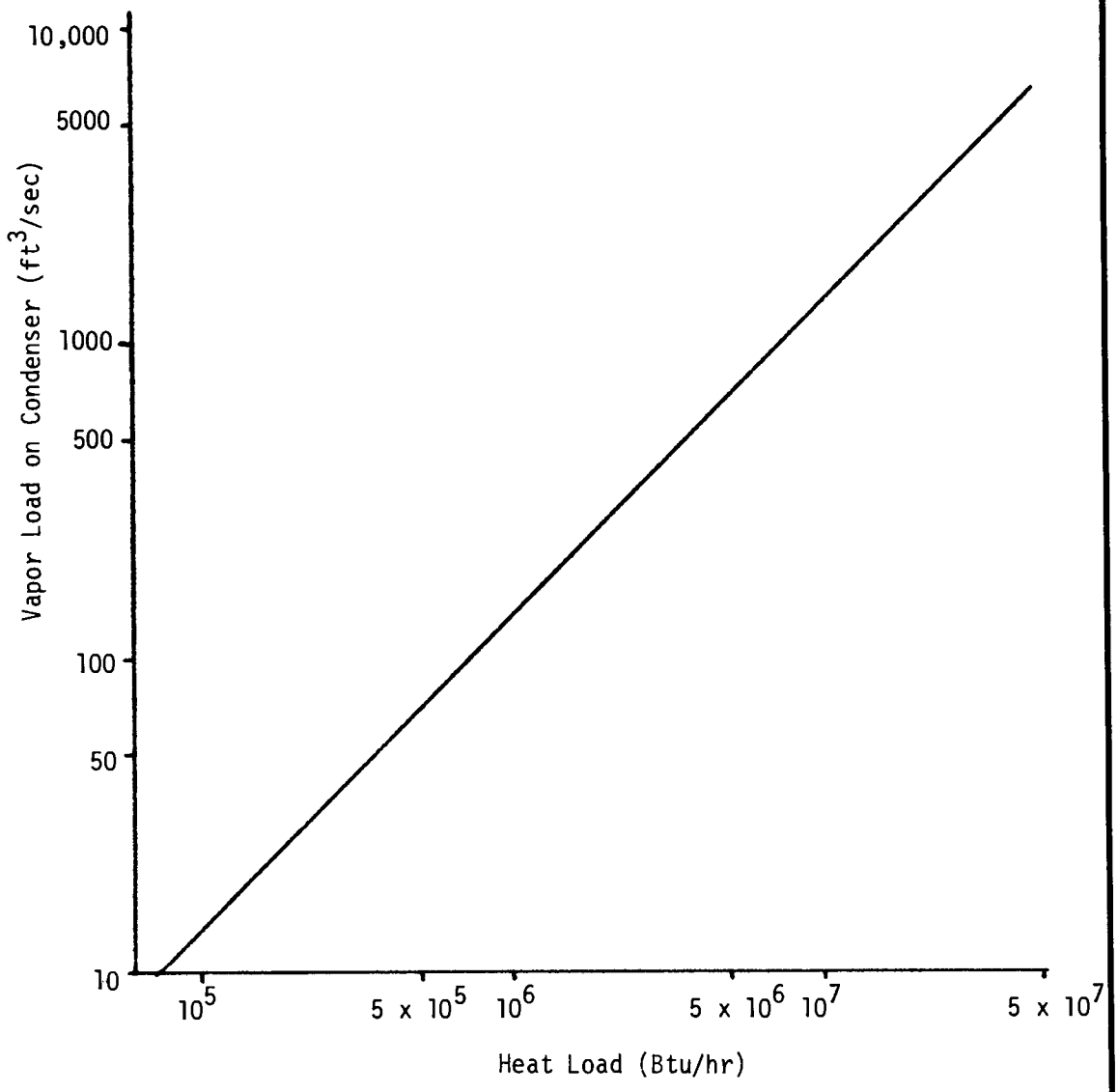
ρ = density of liquid, lb/ft^3

g = acceleration due to gravity = 32.2 ft/sec^2

w = width of pipe, ft

δ = film thickness, ft

μ = viscosity of liquid, lb/ft-hr



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HEAT LOAD AS A FUNCTION OF VAPOR LOAD IN THE CONDENSER

DATE	DRAWN BY	FILE NO.	FIG.
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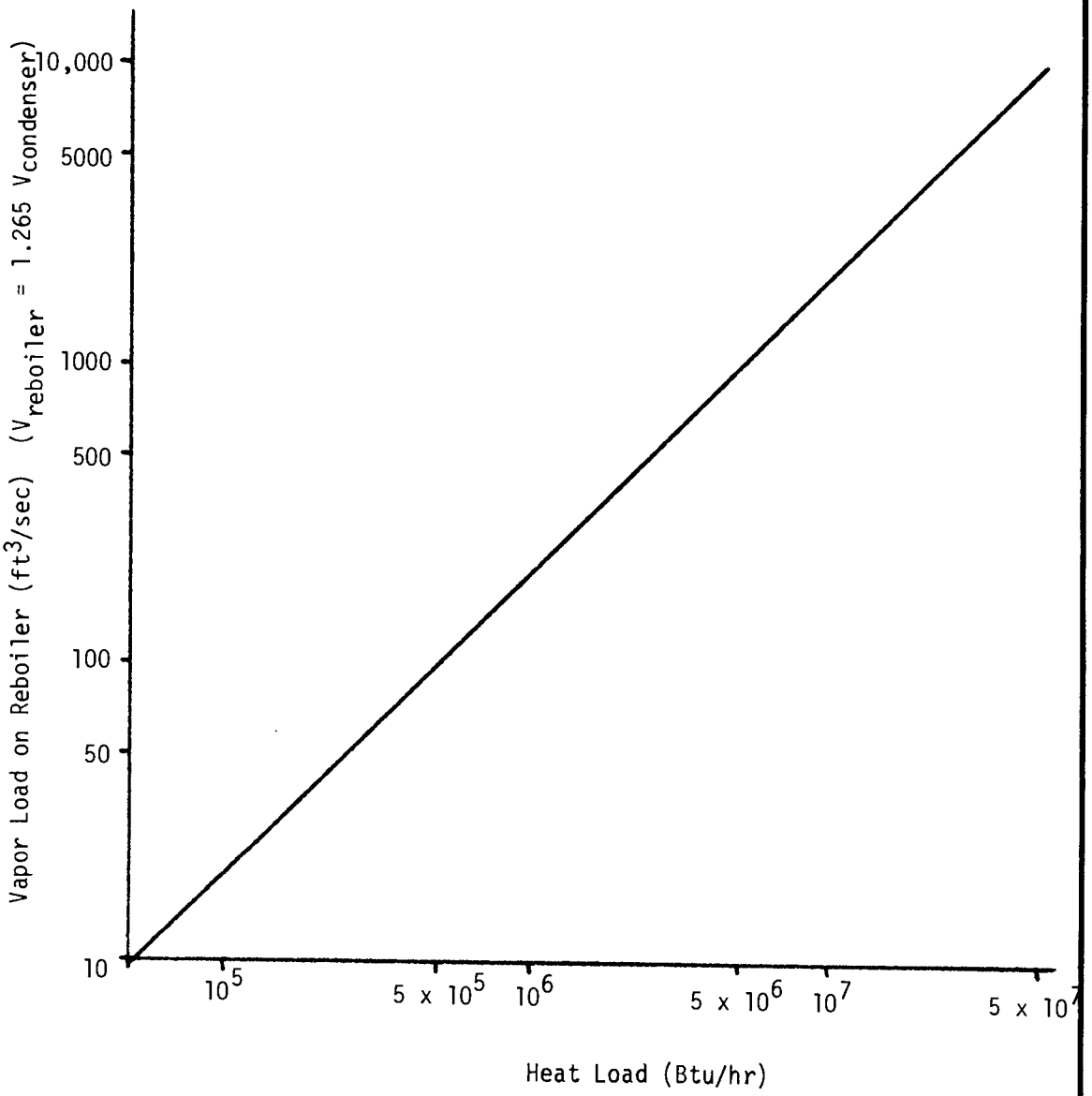

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VAPOR LOAD ON REBOILER VERSUS HEAT LOAD

DATE	DRAWN BY	FILE NO.	FIG.
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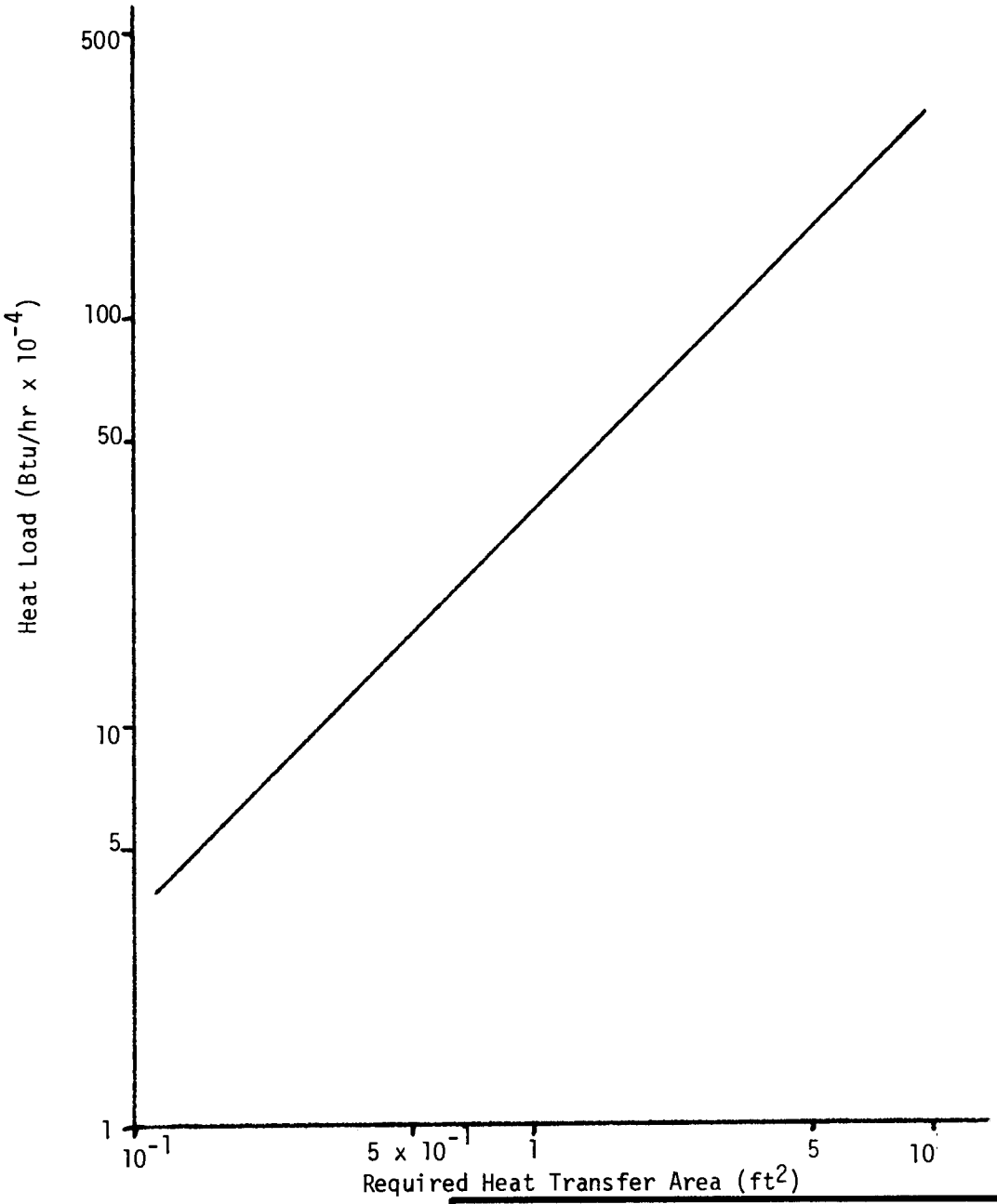

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HEAT LOAD VERSUS HEAT TRANSFER AREA FOR CONDENSER

DATE	DRAWN BY	FILE NO.	FIG.
12-18-68	RF	CEPS-X-66	15

The film thickness for condensing vapors is of the order of 10 mils (14).

Condenser area requirements to prevent a lithium velocity restriction and a drainage restriction were calculated at different vapor loads. A tube length of 5 ft was assumed. As shown in Table 3, the condition which defined the maximum number of tubes in the condenser at all vapor loads, except 1450 ft^3/sec, was the velocity of the product lithium vapors. At a vapor load of 1450 ft^3/sec a very large number of condenser tubes was required to prevent a drainage restriction. Because this number of tubes could be reduced to the same values as calculated to prevent a vapor velocity restriction by simply allowing a slightly thicker condensate film (see note on Table 2), the number of condenser tubes determined to be necessary to prevent a vapor velocity restriction was used in calculating the condenser area. It was felt that these film thicknesses were reasonably close to the 10 mil thickness indicated by Fraas (14). If the maximum attainable δ is only 10 mils, then the cost of a condenser for the distillation process (for high vapor rates) would become prohibitive.

The tritium inventory for a 10-mil-thick lithium film (mole fraction tritium = 10^{-2}) on a 5-ft pipe is 0.02 lb (= 0.01 kg). The value of the tritium inventory is $\$1 \times 10^4$.

6.7.4 Materials of Construction

At high temperatures, lithium has a marked corrosive effect on steel (14). The condenser would therefore have to be fabricated from niobium. To prevent permeation of tritium through the niobium tubes, it would be necessary to vapor deposit a thin film of tungsten on the tubes. Tritium permeation through tungsten is extremely low (19).

It is necessary to use potassium as a coolant in the condenser because water reacts with niobium (3). Thus the direct generation of steam is not possible.

6.8 Reboiler Heat Transfer System

Essentially pure lithium is present in the reboiler as a liquid at its bubble point (1100°C). Because of the high temperature necessary in the reboiler, only two types of heat source were possible, a combustion heat source or an electrical heat source. Several problems present themselves in boiling lithium:

1. A maximum heat flux exists above which film boiling will occur.

2. A minimum heat flux exists below which unstable boiling (bumping) occurs.

Table 3. Minimum Number of Tubes Required to Satisfy the Indicated Condenser Design Criteria (for Various Vapor Loads)
Tube Length = 5 ft.
(Values in parentheses are the areas required for heat transfer)

	Tube Diameter			Vapor Load
	1/2 in.	1 in.	2 in.	(ft^3/sec)
Number of tubes required for drainage restriction	4.7	2.3	1.1	50
	7.9	3.9	1.9	90
	21.9	11	5	250
	1.1×10^5	5.6×10^4	2.8×10^4	1450
Number of tubes required to prevent lithium velocity restriction	184	46	11.5	50
	331	82.5	20.6	90
	735	183	46	250
	5.5×10^3	1.4×10^3	3.46×10^2	1450
Heat transfer area (ft^2) available for maximum number of tubes (lithium velocity restriction)	120(1.17)	60	30	50
	216(2.36)	108	54	90
	480(5.8)	240	120	250
	*3600(31)	*1800	*900	1450
Minimum flow area required to prevent potassium velocity restriction is $A_k = 0.15\ ft^2$.				
Volume of condenser	2 ft^3	3 ft^3	6 ft^3	

*These areas are based on the number of condenser tubes required to prevent a lithium velocity restriction and not that required to prevent a drainage restriction. To make the number of tubes required to prevent a drainage restriction equal to that required to prevent vapor velocity restriction, the following liquid film thicknesses are required:

½ in. pipe, δ = 17 mils

1 in. pipe, δ = 34 mils

2 in. pipe, δ = 20 mils

3. If electric wire heaters are used, an arrangement must be devised to permit heating of the lithium without corroding the heater surface, and without short circuiting the current by flow through the lithium.

4. If electrode heaters are used (I^2R being developed by passing a current through the lithium), the reboiler vessel must be electrically insulated from the environment to prevent accidental grounding.

5. Excess superheating of the lithium must be avoided.

Based on present state of the art, it was decided that a natural gas or coal combustion heat source would be most easily implemented. In this arrangement the lithium would pass through tubes (constructed of niobium internally coated with tungsten) and the combustion would occur external to the tubes. (Existing gas or coal burning power plants operate with effective boiler tube wall tempeatures of 1350°C and higher.)

For lack of sufficient data on boiling lithium, it was assumed that the required vaporization rate could be achieved by using twice the number of tubes as used in the condenser. The properties of niobium in a high temperature oxidizing atmosphere are unknown and may present a substantial problem.

6.9 Heat Loads in the Distillation Column Reboiler

The heat load on the reboiler is given approximately by:

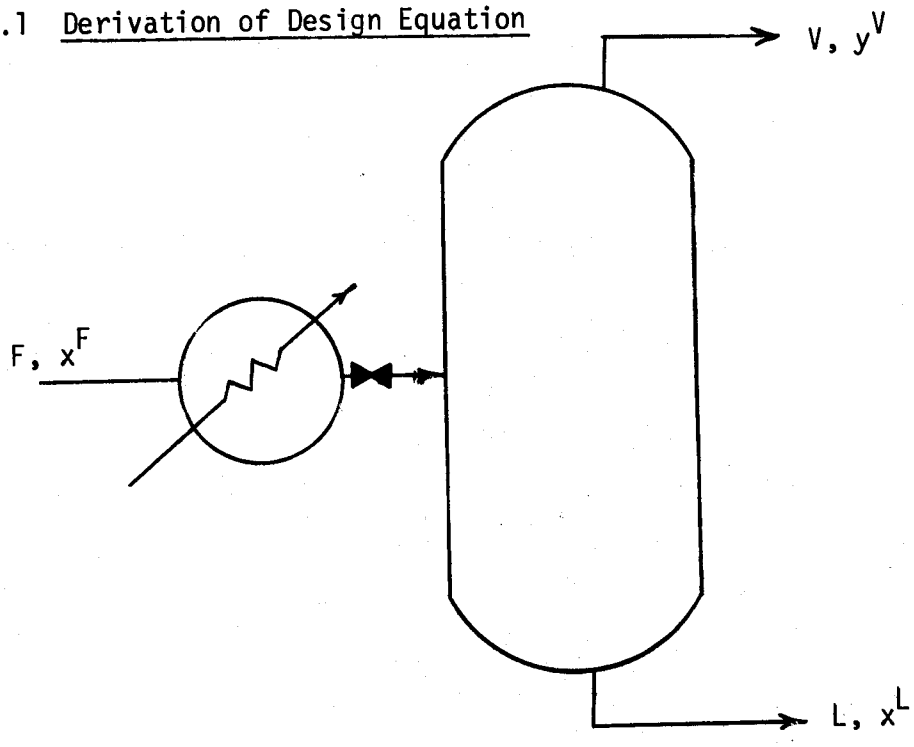
$$Q' \doteqdot V' \times \Delta H_{vap\ Li} \quad \text{(latent heat load >> sensible heat load)}$$

$\Delta H_{vap\ Li}$ at 1100°C (the approximate temperature in the reboiler) = 64.2×10^3 Btu/lb mole (11).

For various values of V', it is possible to obtain Q'.

6.10 Flash Vaporization

6.10.1 Derivation of Design Equation



From a material balance,

$$F = V + L \tag{19}$$

$$(F)(x^F) = (V)(y^V) + (L)(x^L) \tag{20}$$

Combining Eqs. (19) and (20) with the equilibrium relationship:

$$y_i = n_i x_i$$

gives a design equation

$$(y_i^V) = \frac{x_i^F(1 + \frac{L}{V})}{1 + \frac{L}{Vn_i}} \tag{21}$$

which is subject to the condition

$$\sum_i y_i^V = 1.0$$

The number of design variables it is possible to control = C + 3 where C is the number of components.

For the Li, T_2, He system there are six design variables. For this calculation it was decided to specify feed flow rate, pressure, and the composition, and the flash drum temperature and pressure. This eliminates five variables. The L/V ratio or mole fraction of helium can then be chosen; the feed temperature must be determined by a heat balance.

The equilibrium values of n_i for Li and T_2 were found by assuming that Raoult's Law held for the lithium, i.e.,

$$y_{Li} = n_{Li}\, x_{Li} \text{ where } n = \frac{p^{vp}}{\pi} \tag{22}$$

and that the relationship

$$x_T = k\sqrt{p_{T_2}} = k\sqrt{\pi y_{T_2}} \tag{23}$$

held for the tritium (see Sect. 6.2). From Eq. (23),

$$y_{T_2} = n_{T_2}\, x_{T_2} \text{ where } n_{T_2} = \frac{4x_{T_2}^L}{k^2\pi} \tag{24}$$

6.10.2 Flash System Design

Choose: flash temperature = 1800°F

flash pressure = 31 mm Hg

L/V = 10

From Eq. (22),

$$n_{Li} = \frac{30}{31} = 0.9675$$

From Ref. (5),

$$k^2 \text{ at } 1800^\circ F = 10^{-5}$$

Assume,

$$x_{T_2} = 1.6 \times 10^{-4}$$

From Eq. (24),

$$n_{T_2} = \frac{(4)(1.6 \times 10^{-4})}{10^5 \times 21} = 2.064$$

With L/V = 10 and using Eq. (21),

$$y_{Li} = \frac{0.99984(1 + 10)}{1 + 10/0.9675} = 0.97021$$

and

$$y_{T_2} = \frac{1.65 \times 10^{-4}\ (1 + 10)}{1 + 10/2.064} = 0.000311$$

Since $\Sigma y = 1.0$,

$$y_{He} = 1 - 0.97021 - 0.000311 = 0.0295$$

It can be seen that as the L/V ratio increases, the y_{He} increases (when L/V = 100, y_{He} = 0.0318). To obtain the value of y_{He} to give the production rate of helium equal to its generation rate in the reactor blanket, would require a very low value of L/V ($\simeq$ 0.001).

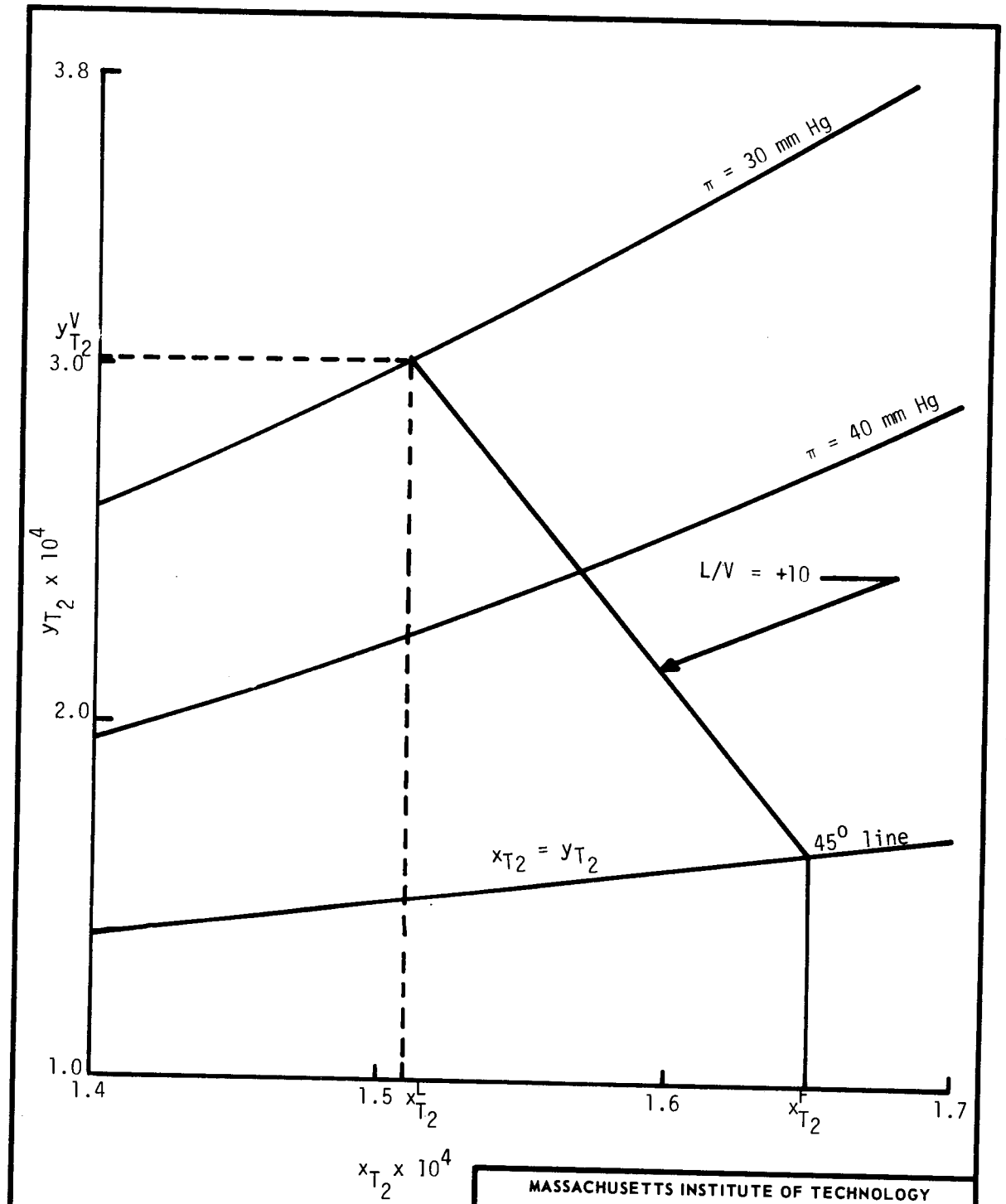
Low values of L/V are undesirable from an operating standpoint since large heat loads would be required to vaporize the feed {the latent heat of vaporization of lithium at 1000°C = 64.2 x 10^3 Btu/lb mole (17)}. The second advantage of operating at a high L/V ratio is that the concentration of T_2 in the gas increases as L/V increases. This is indicated in Fig. 16. The mass balance for L/V = 10. From earlier calculations,

$$y_{T_2} = 3.11 \times 10^{-4}$$

$$(V)(y_{T_2}) = 0.425 \times 10^{-5} \text{ moles/sec}$$

$$V = \frac{0.425 \times 10^{-5}}{0.311 \times 10^{-3}} = 1.368 \times 10^{-2} \text{ moles/sec}$$

Since L/V = 10,



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Equilibrium Diagram Li-T
(Conditions for Flash Separator)

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$$L = 0.1368 \text{ moles/sec}$$

$$F = L + V = 0.1505 \text{ moles/sec}$$

A mass balance for T_2 yields,

$$(F)(x^F_{T_2}) = (V)(y^V_{T_2}) + (L)(x^L_{T_2})$$

Substituting,

$$0.1505(1.65 \times 10^{-4}) = 0.425 \times 10^{-5} + 0.1368\ x^L_{T_2}$$

So,

$$x^L_{T_2} = 1.5 \times 10^{-4}$$

6.10.3 Calculation of Necessary Helium Makeup

The required makeup helium for this system is given by,

$$(V)(y^V_{He}) - \text{production rate in the lithium blanket} = 1.368 \times 10^{-2} \times 0.0295 - 0.85 \times 10^{-5}$$

$$= 3.95 \times 10^{-4} \text{ moles/sec helium makeup}$$

Thus, an inventory of helium or a helium recycle system would be required for operation of this system.

6.10.4 Flash Separator Dimensions

For L/V = 10, the vapor loading in the separator = 570 ft/sec (see Fig. 5).

Assume that the maximum allowable velocity in the separator is the same as that for a distillation tower. Figure 12 gives the diameter of the separator as 4.5 ft.

The height of the flash separation vessel was taken as 20 ft.

6.10.5 Calculation of Condenser Requirements

For a L/V ratio = 10, Fig. 4 gives a condenser duty of 3.25×10^6 Btu/hr. Using the methods of Sect. 6.7, the required condenser area is 6000 ft^2. A heat balance will first be performed.

temperature basis: $T = 1800^oF$

$$F(T - 1800)\, C_{p_{Li}} = (\Delta H_{vap})D$$

$$T = 1800 + \frac{D}{F\, C_{p_{Li}}} \Delta H_{vap}$$

$D\Delta H_{vap}$ for L/V = 10 = 3.25×10^6 Btu/hr

$$F = 0.1505(3600) = 540 \text{ moles/hr}$$

$$C_{p_{Li}} = 7 \text{ Btu/lb mole-}^oF \text{ (16)}$$

$$T = 1800 + \frac{3.25 \times 10^6}{3.78 \times 10^3} = 2660^oF$$

The feed needs to be at a pressure of slightly over 1 atm and must be preheated to 2660^oF before being flashed.

6.10.6 Vapor Load in the Flash Separator

From Fig. 16 various values of $y^V_{T_2}$ were chosen and the corresponding L/V ratios calculated. The value of $(V)(y^V_{T_2})$ must equal the production rate of T_2 and thus V could be calculated. This value of V was then converted to a volumetric basis.

$$y^V_{T_2} = 3.4 \times 10^{-4}$$

$$L/V = \left(\frac{3.40 - 1.65}{1.60 - 1.65}\right) = 35$$

$$(V)(y^V_{T_2}) = 0.425 \times 10^{-5} \text{ moles/sec } T_2$$

hence $V = 1.25 \times 10^{-2}$ moles/sec

at the flash temperature and pressure of 1800^oF and 31 mm Hg respectively.

$$1 \text{ lb mole gas occupies } 359\left(\frac{2260}{492}\right)\left(\frac{760}{31}\right) = 40{,}500 \text{ ft}^3$$

so,

$$V = 40{,}500 \times 1.25 \times 10^{-2} \text{ ft}^3/\text{sec} = 506 \text{ ft}^3/\text{sec}$$

This procedure was repeated for other L/V values. The result is plotted in Fig. 5.

6.10.7 Heat Load in the Flash Separator

From the above calculations the moles of gas produced per second is known as a function of L/V. The latent heat of vaporization of lithium under these conditions is 64.2×10^3 Btu/lb mole. The product $(V)(\Delta H_{vap})$ is approximately equal to the heat load on the flash separator. This calculation was repeated for various L/V ratios. The result is plotted in Fig. 4.

6.11 Nonequilibrium in Condenser

6.11.1 Reaction Rate of $2Li + 1/2T_2 \rightleftarrows 2\ LiT$

Albert and Mahé (6) report an uptake of hydrogen by lithium of 1.5 $cm^3\ H_2/cm^2$ of surface/sec at 1 atm and 680°C. For the purposes of this report the rate of reaction was assumed to be given by the expression:

$$\text{rate} = k_1(p_{H_2})^{\frac{1}{2}}$$

and thus the rate of formation of LiT at the conditions in the condenser is given by

$$\text{rate} = 1.5\left[3.11 \times 10^{-4}\left(\frac{31}{760}\right)\right]^{\frac{1}{2}} \text{cm}^3/\text{cm}^2\text{-sec}$$

$$= 17.5 \times 10^{-5} \text{ ft}^3 H_2/\text{ft}^2\text{-sec}$$

$$\text{the rate of production of } T_2 = 0.425 \times 10^{-5} \text{ lb mole/sec}$$

$$= 17.4 \times 10^{-2} \text{ ft}^3/\text{sec}$$

Thus, for a condenser area of 1000 ft^2, the rate of reaction of tritium with lithium to form lithium tritide would be approximately equal to the rate of production of tritium from the flash separator. The condenser area

for the flash process is 349 ft^2 and therefore about 1/3 of the tritium entering the condenser would be expected to react. Concentration polarization (i.e., an increase in tritium concentration at the liquid lithium surface due to a balance between the rate of tritium reaction back diffusion, and transport to the liquid lithium surface by condensing lithium vapor) would be expected to cause a higher tritium concentration at the liquid surface, and thus increase the reaction rate over that calculated above. {A calculation should be made to determine whether the heat transfer resistance (to condensation) resulting from concentration polarization will be sufficient to lead to a condenser design which is defined by a heat transfer limitation.}

6.11.2 Discussion of the Condenser Design as Related to the Flash System

It is felt that the flash system is probably unsuitable for the following reasons:

1. A large amount of helium is required to achieve a reasonable feed temperature. This would increase the difficulty of the T_2-He separation step.

2. The amount of helium necessary will probably increase the condenser area required to condense the lithium and might enhance the relative rate of formation of LiT over the rate of condensation of lithium.

3. The more free tritium that reacts, the larger the top product stream needs to be for the required production of T_2. This has the adverse effect of increasing the condenser area.

4. If the condensate film thickness cannot be greater than 10 mils, the required surface area of the condenser will be much greater than that indicated in Table 3. This will substantially increase the condenser cost, and the amount of T_2 which reacts with the condensed lithium.

6.12 Cost of Equipment

All equipment is to be constructed of tungsten coated niobium. Since no data on costs for these materials are available, the following cost estimation procedure was adopted.

1. The installed cost of the equipment when fabricated in stainless steel was taken from Ref. (20).

2. The fabrication cost was obtained by dividing the installed cost by 1.43 (21).

3. The material cost of niobium is expected to be twenty times that of stainless steel in 1980 (15). The material cost could not be extracted from the fabrication cost, and it was assumed that the major capital cost item was the material cost.

4. No estimate can be made for the cost of vapor depositing tungsten on niobium. It is not certain if large pieces of equipment can be modified in this manner. As a very crude approximation the cost of tungsten coating would increase equipment cost by 100% (14).

5. To update costs to 1980 the ENR index was taken as 1500 (22).

The factors obtained in Steps 2 through 5 gave a multiplying factor to the cost of step 1 of 56. The costs for distillation columns, flash separators, and heat exchangers are given for various equipment sizes in Figs. 17, 18, and 19.

6.13 Cost Comparison Between Diffusion and Distillation Processes for a Lower Tritium Inventory

For a tritium inventory of 4 kg ($x_T = 1.6 \times 10^{-4}$), Fig. 1 indicates the need to use a slightly higher pressure and reflux ratio in the distillation column. Assuming the product required is the same (0.3 mole fraction of tritium in the vapor), the reflux ratio required is 15,000. The vapor load is therefore 6000 ft^3/sec and the required tower diameter is 13.5 ft. The tower cost is $\$16.5 \times 10^6$ which represents an additional $\$12 \times 10^6$ investment over the case for a tritium inventory of 8 kg. The costs for the reboiler and condenser also increase dramatically.

In contrast, to reduce the inventory to 4 kg by the diffusion process requires an additional capital investment of $\$3.5 \times 10^6$ giving a total cost for the process of $\$6.9 \times 10^6$.

6.14 Acknowledgement

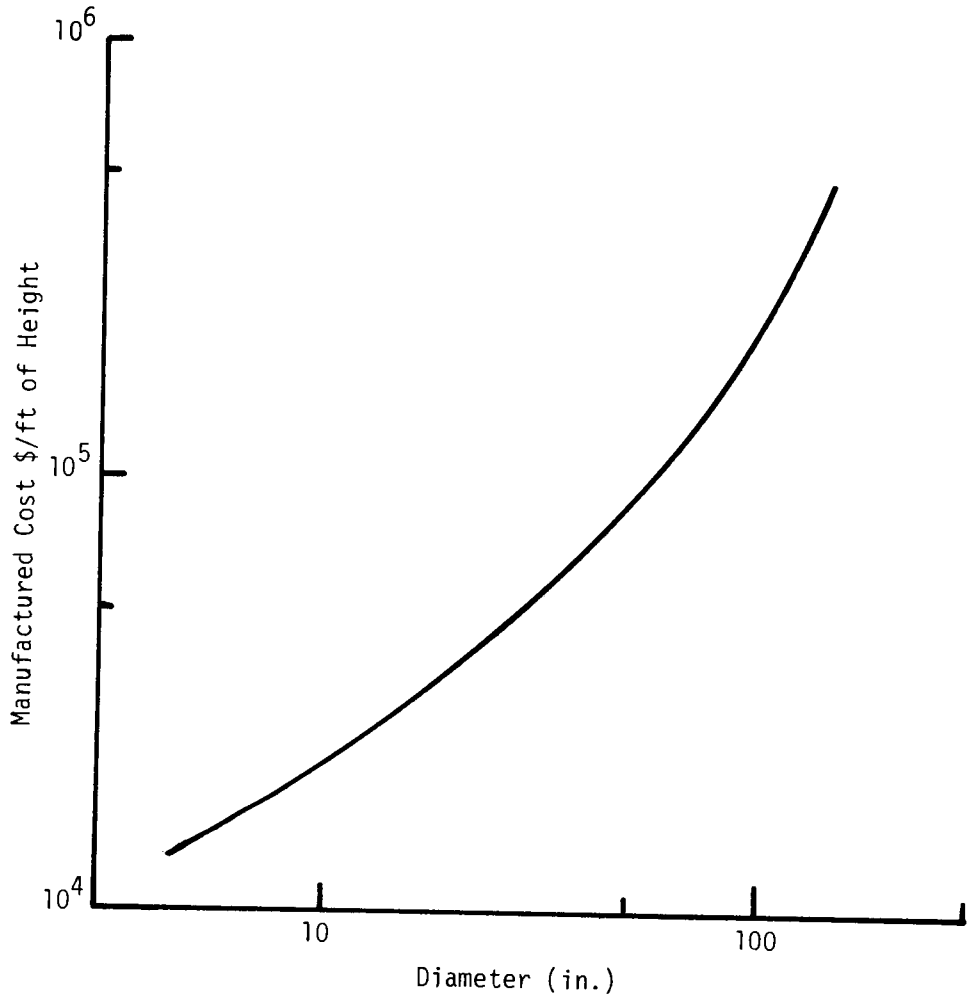
We are indebted to the help and guidance given us by our consultant, A.P. Fraas.

6.15 Nomenclature

A area, ft^2

d diameter, ft

D distillate rate of top product rate, lb mole/sec



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Estimated Cost of Distillation Columns

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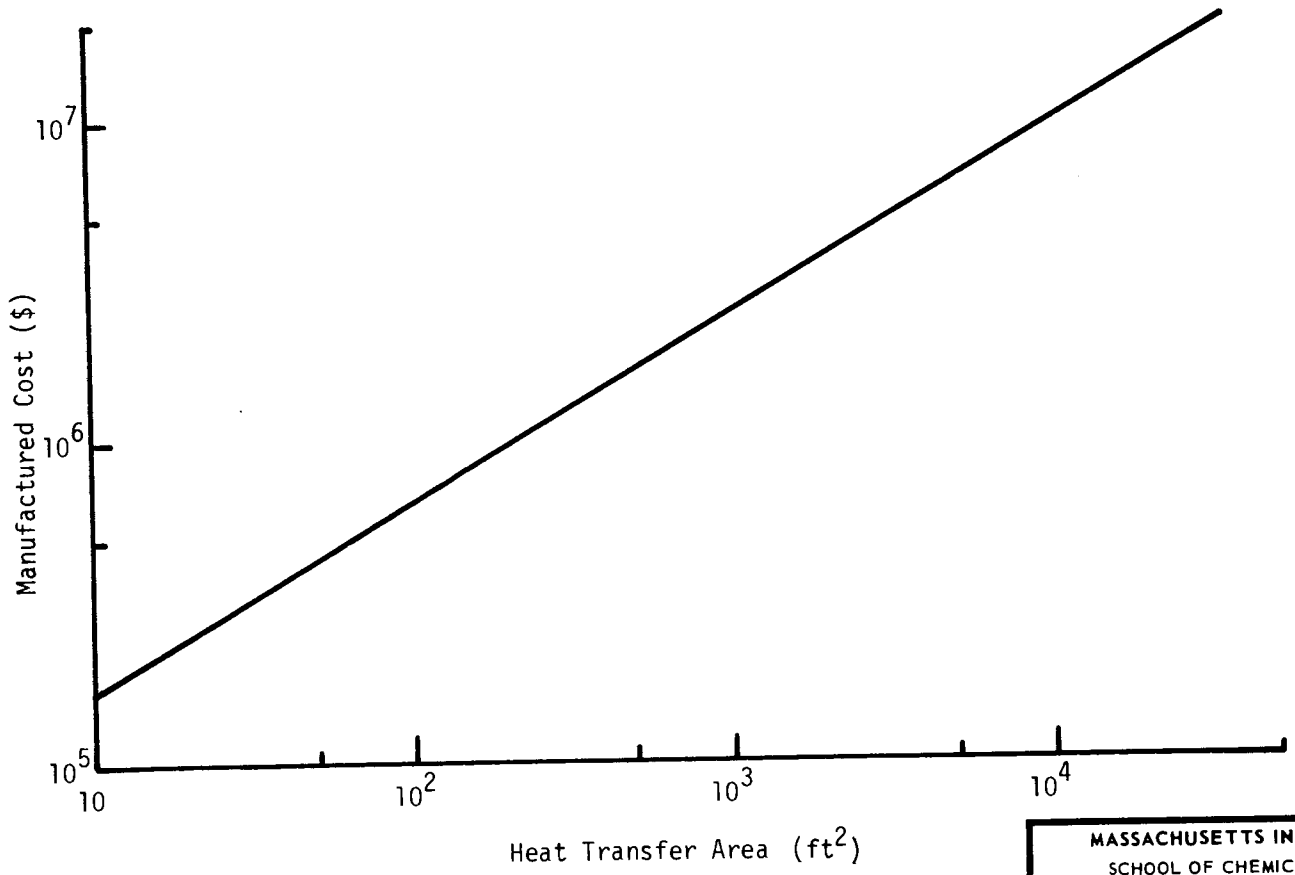

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Estimated Cost of Heat Exchangers

DATE	DRAWN BY	FILE NO.	FIG.
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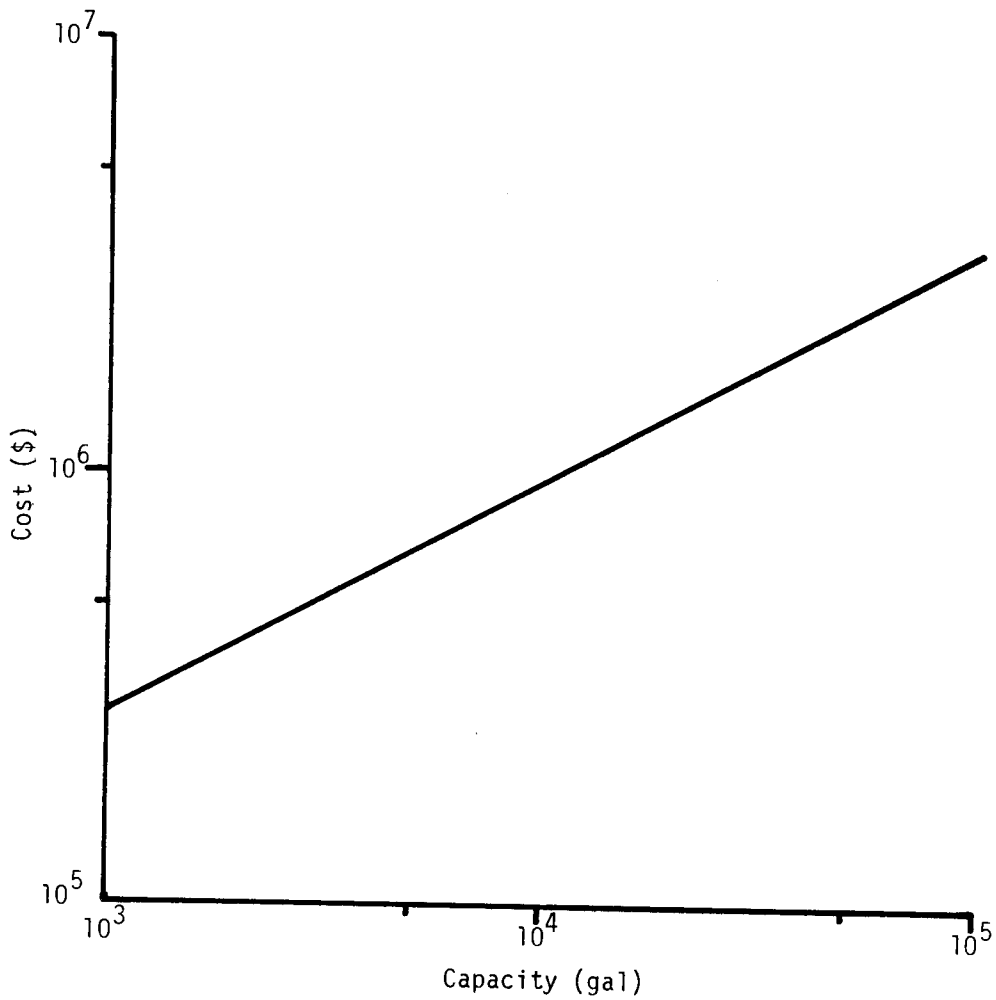

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Estimated Cost of Flash Separators

DATE	DRAWN BY	FILE NO.	FIG.
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F	feed rate to distillation column, lb mole/sec; vapor load, ft^3/sec
f	fugacity of component atm
g	acceleration due to gravity, ft/sec^2, cm/sec^2
h	heat transfer coefficient, Btu/hr-ft^2-^oF
ΔH_{vap}	latent heat of vaporization, Btu/lb mole
H	Henry's Law constant
k	thermal conductivity, Btu/hr-ft^2-^oF/in.
K	equilibrium constant, dimensionless
L	liquid flow rate in the rectifying section of distillation column, lb mole/sec or ft^3/sec
L'	liquid flow rate in the stripping section of distillation column, lb mole/sec or ft^3/sec
m	slope of vapor liquid equilibrium line, dy/dx
O	liquid rate to distillation column as reflux, lb mole/sec
p	partial pressure of component, mm Hg
p^{vp}	vapor pressure of component, mm Hg
q	quantity defined by Eq. (15)
Q	drainage rate, ft^3/sec
Q'	heat load on reboiler, Btu/sec
R	external reflux ratio, lb mole reflux/lb mole distillate
T	temperature, ^oF, ^oC, ^oR, ^oK
ΔT	temperature difference, ^oC, ^oF
U	overall heat transfer coefficient, Btu/hr-ft^2-^oF
v	velocity, ft/sec
V	vapor flow rate in the rectifying section of distillation column, lb mole/sec or ft^3/sec
V'	vapor flow rate in the stripping section of distillation column, lb mole/sec

$\overline{V}_i$ partial molar volume of ith species

$\overline{V}_i^o$ partial molar volume at infinite dilution

W residue rate, lb mole/sec

w width of pipe, ft

x concentration in the liquid, mole fraction

y concentration in the vapor, mole fraction

Greek Symbols

ρ density, lb/ft^3

μ viscosity of gas or liquid, lb/ft-hr

δ film thickness, ft

ψ number of degrees of freedom

λ Empirical constant in Eq. (13)

π total pressure, mm Hg

γ activity coefficient, dimensionless

Σ summation

Subscripts and Superscripts

D distillate

F feed

i inside of tube

L liquid

o outside of tube

V vapor

W residue

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