AN ABSTRACT OF THE THESIS OF

Gregory P. Golike for the degree of Master of Science

in Chemical Engineering presented on April 6, 1981

Title: Development and Dynamic Analysis of an Experimental Heat Integrated Distillation Column

Redacted for privacy

Abstract approved: Ferhan Kayihan

Heat integrated distillation has been studied using an experimental column built for that purpose. Heat integration involves the use of intermediate reboilers and condensers to increase the thermal efficiency of distillation. The intermediate reboiler and condenser systems used in this work were designed so that the experimental column could be operated either as a conventional distillation column (without the intermediate heat exchangers) or as a heat integrated distillation column. Sufficient instrumentation was included in the experimental setup so that liquid temperatures on each tray in the column could be monitored.

Pulse disturbances in four system parameters were used to generate dynamic responses for both conventional and heat integrated distillation using the methanol-water binary system. The disturbed parameters include the liquid feed flowrate, the bottom boilup rate, the external liquid reflux rate and the intermediate condensation rate. Based on comparisons of the dynamic responses for the two systems, it is concluded that heat integrated distillation in this case is more restrained in its response to disturbances. This restraint is attributed to a moderating effect provided by the

intermediate heat exchangers.

The experimental dynamic responses were compared with dynamic responses predicted by a theoretical model. This comparison indicates that the heat losses from the experimental column are substantial and must be reduced in further work on the experimental equipment.

# Development and Dynamic Analysis of an Experimental Heat Integrated Distillation Column

by

Gregory Philip Golike

A THESIS

submitted to

Oregon State University

in partial fulfillment of
the requirements for the
degree of

Master of Science

Completed April 1981

Commencement June 1981

APPROVED:

Redacted for privacy

Assistant Professor of Chemical Engineering
in charge of major

Redacted for privacy

Head of Department of Chemical Engineering

Redacted for privacy

Dean of Graduate School

Date thesis is presented April 6, 1981

Typed by Donna Lee Norvell-Race for Gregory Philip Golike

## ACKNOWLEDGMENT

It is a pleasure to mention those who have had a part in helping me to this point. First, special thanks to my advisor and friend, Ferhan Kayihan, for guidance and great expectations. Thanks are due also to Professor Bob Mrazek for all the practical advice necessary to actually build the equipment. The interest of Dr. Charles Wicks and the funds provided by the Chemical Engineering Department were crucial; without them there would be no equipment. Thank you!

Many friends provided extra help. Among these are Charlie Krogh and Ken Wicks who helped much in the construction and instrumentation. Sada Patil was always willing to donate an extra pair of hands when two were not enough.

The financial support provided by Weyerhaeuser Foundation and the Chemical Engineering Department is gratefully acknowledged. The fellowship and teaching assistantships kept bread on the table and me in school.

Finally, the encouragement and support of my family were unbeatable. And to Mary what can I say except thank you again and again!

TABLE OF CONTENTS

## LIST OF FIGURES

LIST OF TABLES

# NOTATION

| Symbol | | Definitions and Units |
|---|---|---|
| B | = | bottoms product flowrate (gmole/min) |
| D | = | distillate flowrate (gmole/min) |
| E | = | stage efficiency in terms of vapor concentrations (defined by equation 13) |
| F | = | liquid feed flowrate (gmole/min) |
| F' | = | vapor feed flowrate (gmole/min) |
| h | = | stage vapor holdup above tray (gmole) |
| H | = | stage liquid holdup on tray (gmole) |
| $L_i$ | = | liquid flowrate from ith tray (gmole/min) |
| L' | = | liquid flowrate to the intermediate reboiler (gmole/min) |
| $L'_R$ | = | liquid recycle flow from the intermediate reboiler (gmole/min) |
| L" | = | liquid flowrate through the intermediate "condenser" (gmole/min) |
| LBB | = | external liquid reflux flowrate (gmole/min) |
| $LW_i$ | = | liquid withdrawal rate from ith tray (gmole/min) |
| P | = | pressure (Pa) |
| q | = | the ratio of the heat required to convert 1 mole of feed to a saturated vapor to the molal latent heat |
| $Q_c$ | = | main condenser heat load (kcal/hr) |
| $Q_{ic}$ | = | intermediate condenser heat load (kcal/hr) |
| $Q_{ir}$ | = | intermediate reboiler heat load (kcal/hr) |
| $Q_R$ | = | main reboiler heat load (kcal/hr) |

NOTATION, continued

Symbol

| | | |
|---|---|---|
| $R$ | = | ideal gas constant (kcal/gmole - °K) |
| $SV$ | = | boilup rate from the intermediate reboiler (gmole/min) |
| $t$ | = | time (min) |
| $T_c$ | = | condenser temperature (°K or °C) |
| $T_{ic}$ | = | intermediate condenser temperature (°K) |
| $T_{ici}$ | = | temperature of liquid at inlet to intermediate "condenser" (°C) |
| $T_{ico}$ | = | temperature of liquid at outlet of intermediate "condenser" (°C) |
| $T_{ir}$ | = | intermediate reboiler temperature (°K or °C) |
| $T_o$ | = | ambient temperature (°K) |
| $T_R$ | = | reboiler temperature (°K or °C) |
| $T_j$ | = | temperature of liquid on the tray where j indicates the stage number (°C) |
| $V_i$ | = | vapor flow from vapor space above tray i (gmole/min) |
| $V'_i$ | = | vapor flow from liquid to the vapor space above tray i (gmole/min) |
| $VPP$ | = | boilup rate from the bottom reboiler (gmole/min) |
| $VW_i$ | = | vapor withdrawal from tray i (gmole/min) |
| $W$ | = | net work consumption (kcal/hr) |
| $x_B$ | = | bottoms product concentration (mole fraction) |
| $x_D$ | = | distillate product concentration (mole fraction) |
| $x_i$ | = | concentration of liquid on ith tray (mole fraction) |
| $x_{ic}$ | = | liquid concentration at stage where the intermediate condenser is located (mole fraction) |

NOTATION, continued

<u>Symbol</u>

| | | |
|---|---|---|
| $x_{ir}$ | = | liquid concentration at stage where the intermediate reboiler is located (mole fraction) |
| $y_i$ | = | concentration of vapor above the *i*th tray (mole fraction) |
| $y^*$ | = | equilibrium vapor concentration (mole fraction) |
| $z$ | = | feed concentration (mole fraction) |
| $\beta$ | = | actual reflux ratio divided by minimum reflux ratio |

# Development and Dynamic Analysis of an Experimental Heat Integrated Distillation Column

## I. INTRODUCTION

### Significance

Distillation is by far the most common unit operation used for separation in the chemical process industry. As such, it is used to upgrade feed stocks, to separate reaction intermediates, and to purify a wide range of products. In a recent study commissioned by the U.S. Energy Research and Development Administration (now the Department of Energy) and reported by Mix et al. (1978), it is conservatively estimated that the energy consumed by distillation represents nearly three percent of the entire national energy consumption. An earlier study (Prengle et al., 1974) pointed out that a 10 percent energy savings for distillation, easily a reasonable goal, would be equivalent to saving 100,000 bbl/day of oil. Given this significance for improvement in distillation operation, the study of more thermally efficient distillation columns becomes more and more important.

Over a quarter of a century ago D. C. Freshwater (1951) proposed a classification of the various methods for increasing the thermal efficiency of distillation. He divided the methods into three broad areas: multiple effect methods, heat pump or vapor recompression methods, and indirect methods. Multiple effect methods basically involve use of the distillate vapor generated by one column to supply the heat required in the reboiler of another column. Such methods involve more than one distillation column for their application. Robinson and Gilliland (1950) present and discuss several of these multiple effect schemes. Recently, several design studies dealing with these methods have been performed (Tedder and Rudd, 1978; Umeda et al., 1979).

In contrast to multiple effect methods, vapor recompression has been used to improve the thermal efficiency of a single column. With these methods, energy is supplied as work to overcome the thermodynamic reversibilities of distillation. Again these methods are discussed by Robinson and Gilliland (1950) as well as in some more recent publications (Freshwater, 1961; Null, 1976). Indirect methods involve the integration of the process heating and cooling streams from the column with other parts of the plant. In these methods the heat transfer streams are used for further heat transfer or for power generation elsewhere in the plant.

The method of heat integration studied here can be best classified as a multiple effect method, although the method considered in this thesis involves only one distillation column. The basic strategy of multiple effect methods is to improve thermal efficiency by controlled degradation of energy. That strategy is the same for the method studied here.

Heat integration as described and studied in this work is realized by the use of additional intermediate reboilers and condensers. The concept can best be understood by comparison with conventional distillation as shown in Figure 1. A single bottom reboiler and an overhead condenser are used in conventional distillation to provide the necessary heating and cooling. Heat integrated distillation by contrast requires the use of additional reboilers and condensers. These additional heat exchangers are located between the bottom reboiler and the overhead condenser as shown in Figure 2. The effect of these intermediate heat exchangers is to cause a reduction in the irreversibilities of distillation and thus to improve the thermal efficiency. The improved thermal efficiency of heat integration is actually observed as a reduction in the degradation of the heat energy passing through the column. If the modified column is subsequently integrated into the surrounding plant by the indirect

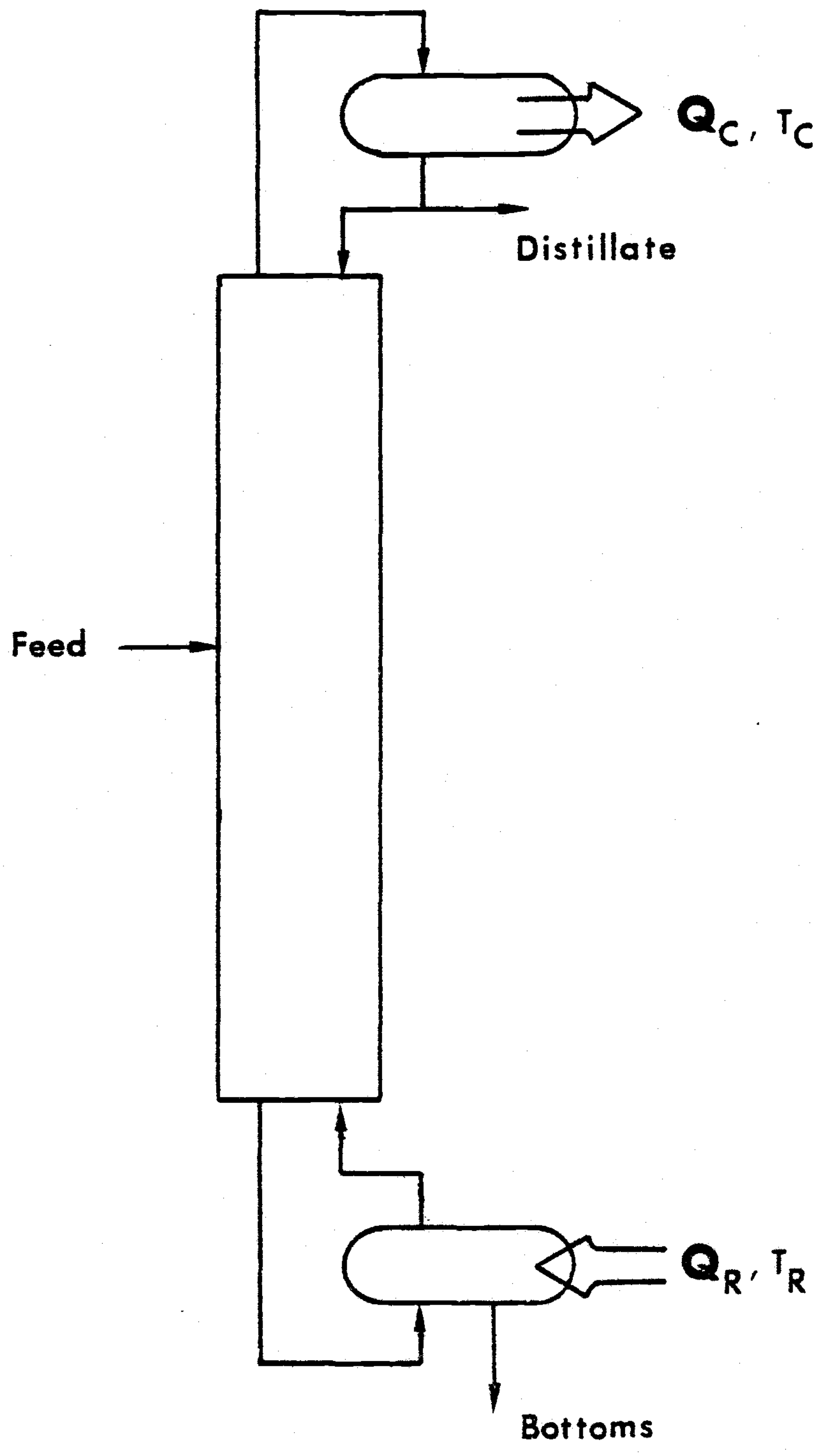



Figure 1. Conventional Distillation Column.

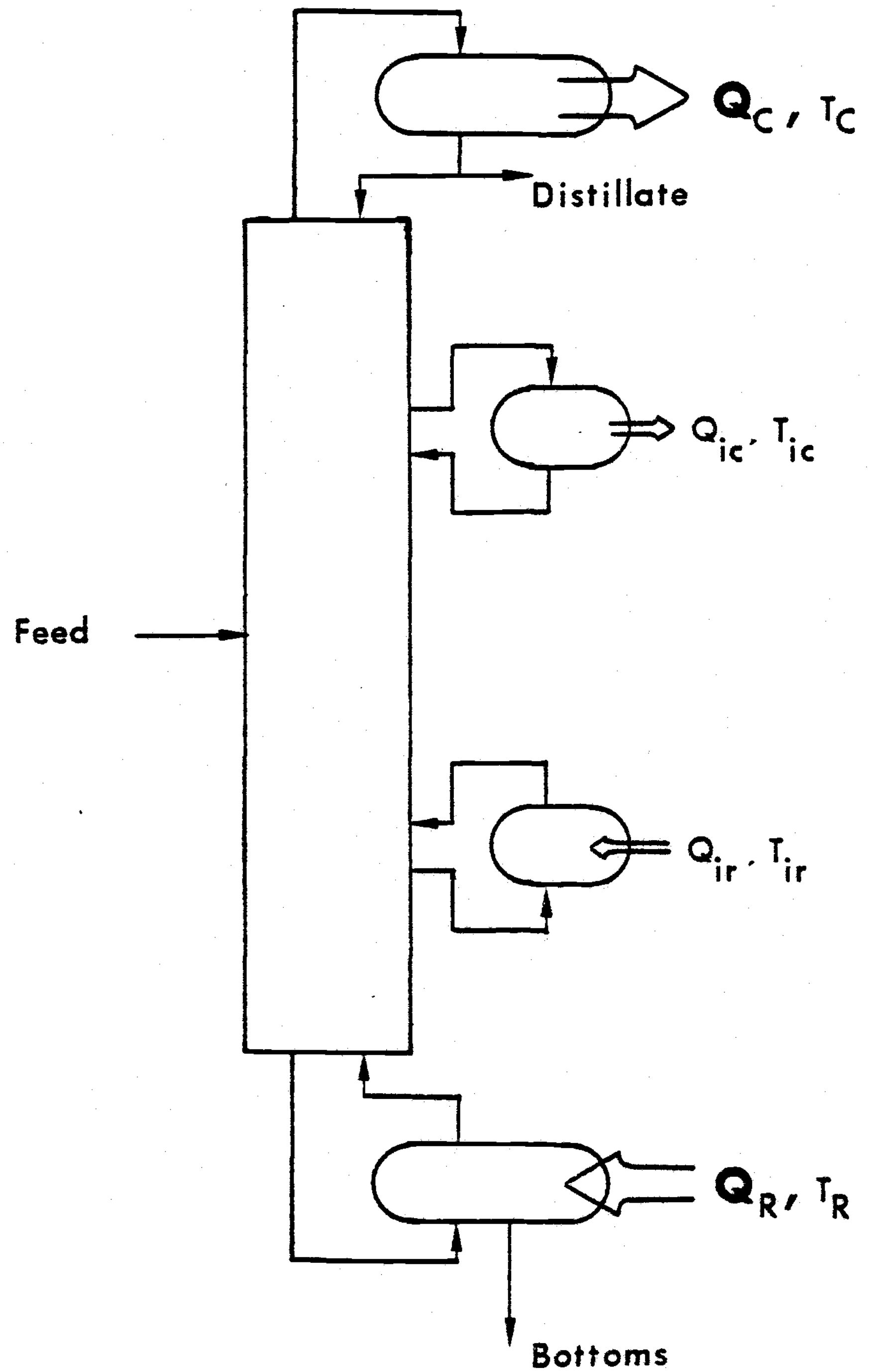



Figure 2. Heat Integrated Distillation Column.

methods previously described, then the increased thermal efficiency will be observed as a reduction in energy consumption.

## Purpose

As with all processing equipment, the decision for or against application of heat integrated distillation is an economic one. For heat integrated distillation, the decision will probably be based primarily on the trade-offs between the increased thermal efficiency and the increased capital expenditures required for this type of distillation column. The economic data necessary for this decision change with the specific application, with location, and with time; this data cannot really be studied generally on an experimental basis. The things which can be studied experimentally and which are likely more important with regard to actual application are the practical considerations. That is: How is a heat integrated distillation column operated and controlled? And how does it respond to system disturbances? Until now, no experimental studies have been performed to address these questions.

The purpose of this project, therefore, has been to study the operation and response of an experimental heat integrated distillation column. Particular emphasis was placed on comparison of the responses of both the heat integrated system and the conventional system to the same disturbances. A secondary purpose of this project has been to construct the experimental apparatus necessary for the study of the two distillation columns.

## Scope

In order to make the study manageable, the project was limited to four general parts which were completed in order. First, the experimental equipment was constructed and instrumented. Following the construction, the column was tested and adjusted. Then, the dynamic responses of the two distillation systems to various disturbances were measured. And finally the experimentally measured dynamic responses were compared with the responses predicted by a theoretical model. These four areas are presented as follows: Chapters II and III contain the necessary background on heat integrated distillation, Chapter IV presents the details of the experimental equipment, Chapters V and VI contain the results of both the experimental and simulated results along with appropriate observations and summaries, and Chapter VII draws up the strings with the conclusions.

It should also be noted before concluding this section that this work was performed in conjunction with a theoretical study on the response and control of heat integrated distillation (Patil, 1981). The study of heat integration was approached in two ways. This work is based on experimental results while the other is based on computer simulations.

# II. LITERATURE SURVEY

## Background

Although heat integration using intermediate reboilers and condensers has been known for some time to be a method of improving distillation efficiency, the method has only recently received more attention. Most likely the first analysis of distillation using intermediate heat exchangers is due to Benedict (1947). He showed that the work required for binary distillation is significantly reduced by the addition of an intermediate auxiliary condenser when a feed mixture of low composition is separated into relatively pure products.

More comprehensive summaries of heat integration in distillation are given both by Robinson and Gilliland (1950) and by King (1971). In these books, heat integration using intermediate heat exchangers is presented as one of the options available for increasing the thermal efficiency of distillation. Robinson and Gilliland point out, among other things, the effect that the characteristics of the mixture to be separated might have on the application of heat integration. They mention, for example, a proposed distillation of an ethanol-water mixture to produce a distillate with composition close to the azeotropic composition. In this case the minimum reflux is determined by a tangent pinch. If a feed relatively concentrated in ethanol were used, a substantial portion of the heat duty required could be supplied by an intermediate reboiler at a temperature only slightly above that of the overhead condenser.

King indicates the trade-offs between increased efficiency and greater capital costs involved with the use of intermediate heat exchangers in distillation. He states that the increased thermal efficiency of heat integrated distillation is not usually considered to offset the increased capital costs (associated with the extra heat exchangers and the extra stages required for this kind of distillation), except in the case of low temperature distillation. Pratt (1967) points out the possible capital savings associated with heat integration. The use of intermediate reboilers and condensers will result in reduced liquid and vapor flows towards the product ends of the column. These reductions may allow reductions in column diameters at the product ends of the heat integrated column. Based on this concept, Timers (1969), in a major analysis of the use of intermediate heat exchangers in distillation, has suggested a design criterion for heat integration based on minimized column volume.

More recently, Kayihan (1977) studied the optimum distribution and heat loads of the intermediate reboilers and condensers in heat integrated distillation. He found the solution to the optimization problem for the case of nearly complete separation of an ideal binary mixture. The results indicate that intermediate condensers have the greatest effect on column thermal efficiency for low feed concentrations while intermediate reboilers have the greatest effect when the feed concentration is high. Further, it was found that the first additional set of heat exchangers make the dominant contribution to increased thermal efficiency; subsequent heat exchangers make smaller and smaller contributions. The actual results of this study are considered more completely in Chapter III.

A distillation scheme making use of intermediate vaporization and condensation to improve the thermal efficiency of distillation has been presented by Mah et al. (1977). The scheme is similar to the method studied here in that intermediate heat exchange is used to increase thermal efficiency. However, in the scheme studied by Mah et al., the distillation of a single binary mixture is carried out in two columns operated at different pressures. One column is used as the stripping section, while the other is used as the rectifying section. The operating pressures of the two columns are chosen to allow heat transfer from the rectifying section to the stripping section. It is reported (based on computer simulations) that this distillation scheme has a potential for 50 to 75 percent utility reductions.

Fitzmorris and Mah (1980) indicate in a further study on the same distillation scheme that intermediate vaporization and condensation can substantially reduce the irreversibilities of distillation associated with the temperature and concentration differences inside the column. The irreversibilities arising from material and energy exchanges outside the column, however, are not affected. These external irreversibilities can only be reduced by better integration of the distillation column into the external plant surroundings.

## Applications and Qualifications

The references discussed up to this point have been entirely theoretical studies. That is not to say that actual application of intermediate heat exchangers to operating distillation columns has not occurred. In fact, they have been applied in specific cases. Intermediate condensers (or circulating refluxes as they are also called) have been used for some time on both the atmospheric and vacuum distillation units in oil refineries (Bannon and Marple, 1978). These circulating refluxes have been used both to recover useful heat and to balance column vapor loads, thus allowing reductions in column diameters. Duckham and Fleming (1976) mention two cases of low temperature distillation where intermediate heat exchange was used. They report that an intermediate condenser was used along with a heat pump to reduce the energy requirements of a helium-nitrogen-methane fractionator. In the other case, a column using an intermediate reboiler to supply 40 percent of the total heat duty was designed for a large-scale ethylene plant.

The cases cited here as examples for application of heat integration are all ones involving separations with high energy requirements. In these cases the incentive for improvement is great. In other less demanding separations it may be more difficult to justify application of heat integration with intermediate heat exchange. As Stephanson and Anderson (1980) point out, it is necessary to find use for less degraded heat withdrawn from the heat integrated column before the increased thermal efficiency afforded by the intermediate heat exchangers can be observed as a reduction in energy consumption. It should be noted as does Abrams (1978), that a distillation column with intermediate heat exchangers provides greater opportunities for integration with the total plant than does a conventional distillation column.

This survey of literature indicates that no experimental work has been done on heat integrated distillation using common systems. In addition, no consideration has been given to the changes intermediate reboilers and condensers might impose on the response of the distillation column to disturbances. It is in this area of dynamic response where one would expect to find additional criteria for choosing between conventional and heat integrated distillation. We will proceed in that direction.

# III. THEORY

Before presenting the equipment design and the results obtained, additional background material must be presented. It is probably best to begin first with a short review of conventional distillation. The information presented in this review is necessarily condensed; more complete summaries of the subject may be found in standard texts such as the one by Treybal (1968). In addition to the review of distillation, the concept of heat integration is presented and discussed along with the optimization results pertaining to the intermediate heat exchanger location and heat duty. Finally, to conclude the section, the mathematical model for distillation dynamics is developed.

Only the theory necessary to understand the work presented later is covered in this chapter. For this reason the following discussion is limited to binary distillation.

## Conventional Distillation

Consider the distillation column previously given in Figure 1. The overall action of the column is to separate the feed into its two components. The liquid feed mixture is separated into a distillate product concentrated in the least volatile component. Basically, the separation is accomplished by exploiting the boiling point differences of the two components. The operation of the distillation column performing the separation can be conceived as a number of boilers and condensers in which the feed liquid is partially vaporized, the resulting vapor being more concentrated in the volatile component is condensed separately, and the process is repeated until the specified products are obtained.

In actual practice only the bottom reboiler and overhead condenser are used to provide the heating and cooling capacity, and the column is equipped with vapor-liquid contacting devices to promote mass transfer between the phases. The internals of a bubble-cap tray distillation column are shown in Figure 3. It is clear from this figure that the liquid following down through the column is contacted on every tray by the rising vapor. It is in this manner that the vapor is concentrated in the volatile component, while the liquid is concentrated in the heavy component.

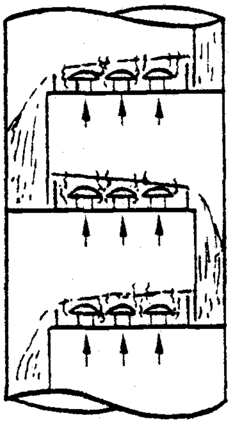


Figure 3. Bubble Cap Tray Column

The operation of the conventional column can be illustrated further with the McCabe-Thiele diagram shown in Figure 4. The solid straight lines on the graph are the column operating lines. They indicate the vapor-to-liquid flow ratios at any point in the column. The two lines--one for the stripping section and the other for the rectifying section--intersect at the point dictated by the reflux ratio and by the concentration and thermal state of the feed. In using straight lines to represent the operating lines, the liquid and vapor flows in the column are assumed to be constant between the feed point and the column ends. If, due to heat losses or changes in boilup, this assumption is not valid, then the lines will be curved or discontinuous to represent the liquid and vapor flows.

The differences between the operating lines and the equilibrium

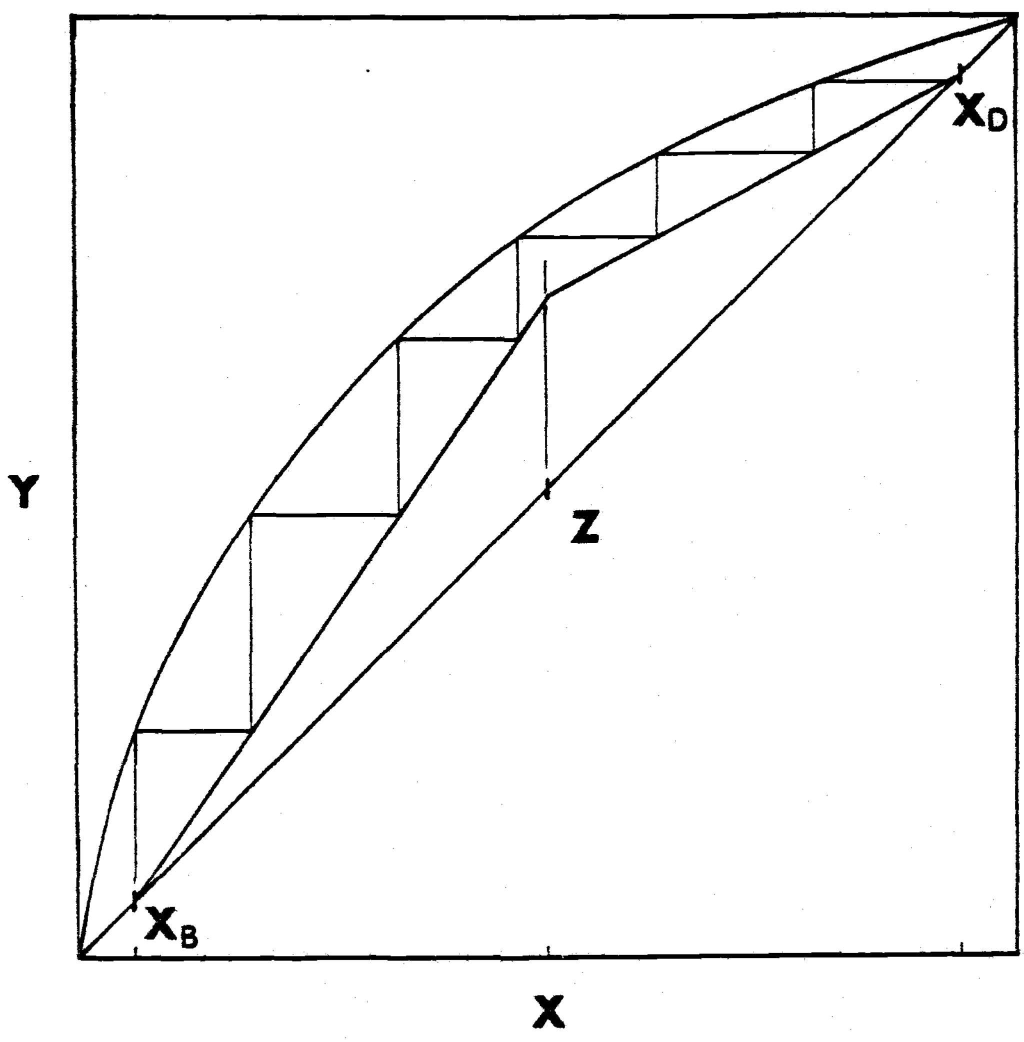



Figure 4. McCabe-Thiele Diagram for Conventional Distillation.

line indicates the driving forces for heat and mass transfer existing within the column. These differences, while insuring that the separation can be accomplished in a finite number of stages, are the source of the internal irreversibilities. These are the irreversibilities we desire to reduce with heat integration.

The equilibrium stages necessary to carry out the separation are indicated on the diagram in Figure 4. In an actual column, the contacting devices are not capable of operating as equilibrium stages, thus the points representing stage liquid and vapor compositions will not fall on the equilibrium curve as shown in Figure 4. Depending on the stage efficiency, the points will fall somewhere between the equilibrium curve and the operating lines.

The separation of a liquid mixture into its components requires a certain amount of work. For an ideal binary mixture, the minimum work required is given by

$$W_{min} = - FRT_o \sum_{i=1}^{2} z_i \ln z_i \quad . \tag{1}$$

This minimum, however, is only obtainable for an infinite column with a reboiler operating on each stage below the feed and with a condenser operating on each stage above the feed (Pratt, 1967). While this column is obviously impractical, it does indicate the direction one should head in search of increased thermal efficiency. The conventional distillation column has a net work consumption much greater than the minimum work requirement. The actual work (neglecting irreversibilities outside the column) is given by

$$W = Q_o T_o \frac{T_R - T_C}{T_R T_C} \tag{2}$$

where $Q_o$ for an adiabatic column processing saturated feed is equal to $Q_C$ and $Q_R$ (since they are the same in this case).

## Heat Integrated Distillation

The practical alternative to the infinite column with maximum thermal economy is a heat integrated column with a finite number of intermediate heat exchangers. The modified column already presented in Figure 2 is just such an alternative. Consider the operating lines for this modified column on the McCabe-Thiele diagram as shown in Figure 5. The solid lines are the operating lines for the heat integrated column, while the dashed lines are those for the conventional column having the same overall heat duty. It is clear that the heat and mass transfer driving forces are reduced for the heat integrated column (as compared to the conventional column). The price paid for the reduction in irreversibilities is an increase in the number of stages required for the same separation. This is indicated by the difference in the number of equilibrium stages required for the same separation (see Figures 4 and 5).

The gain in thermal efficiency can be seen also in the work requirement for heat integrated distillation. The work requirement for the modified column as shown in Figure 2 is given by

$$W = Q_R\frac{T_R-T_o}{T_R} + Q_{ir}\frac{T_{ir}-T_o}{T_{ir}} - Q_{ic}\frac{T_{ic}-T_o}{T_{ic}} - Q_c\frac{T_c-T_o}{T_c} \tag{3}$$

Since the temperature of the intermediate reboiler is lower than that of the bottom reboiler and since the temperature of the intermediate condenser is higher than that of the overhead condenser, the net work required for the heat integrated column is less than that required for the conventional column. Whether this reduction is actually

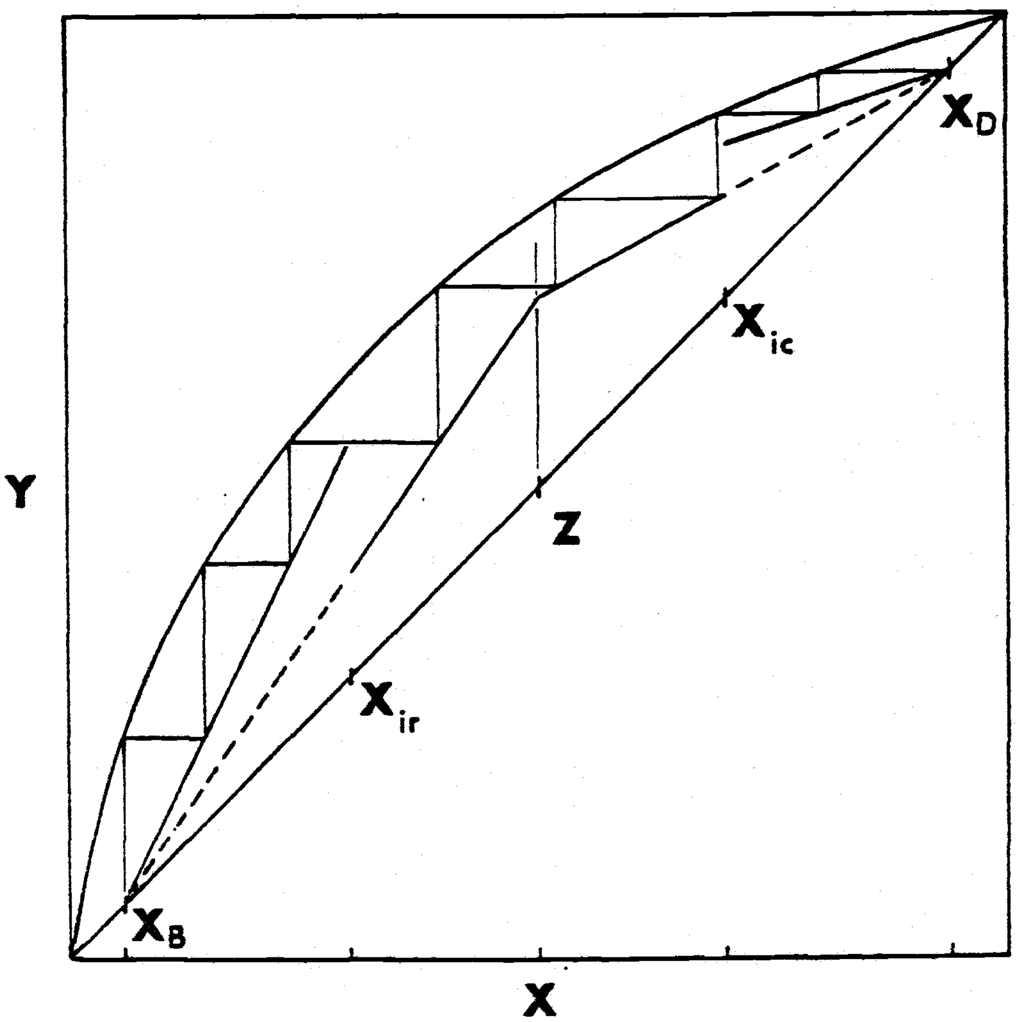



Figure 5. McCabe-Thiele Diagram for Heat Integrated Distillation (one set of intermediate heat exchangers).

realized depends primarily on the integration of the column into the plant as a whole.

The modified column, however, does provide more opportunity for integration with the surrounding plant. This advantage has been largely ignored in considering the application of heat integrated distillation. For example, vapor feeds to the column may be sufficiently warm to supply the intermediate reboiler heat duty; cold feeds may be heated nearly to saturation by heat exchange in the intermediate condenser; the heat available in the bottoms product may be useful for heat transfer in the intermediate reboiler; the cooling water flow to the overhead condenser may be further used in the intermediate condenser; and finally, the heat transfer fluid used in the bottoms reboiler may also be sufficient to provide the heat duty for the intermediate reboiler since it operates at a lower temperature. These examples serve to illustrate the variety of choices available with a heat integrated column.

## Optimization Results

In applying heat integration using intermediate reboilers and condensers, one would naturally want to know the best number of intermediate heat exchangers to use in a particular situation. In addition, the best location and heat loads for the intermediate heat exchangers should be known. Some optimization work is required to get this information. For the specific case of nearly complete separation of an ideal binary mixture, the optimization problem has been solved (Kayihan, 1977). The material presented here is a summary of that original work.

The results of the work by Kayihan (1977) can first be summarized by the information presented in Figure 6. In this figure, the thermodynamic efficiency for distillation with various numbers of

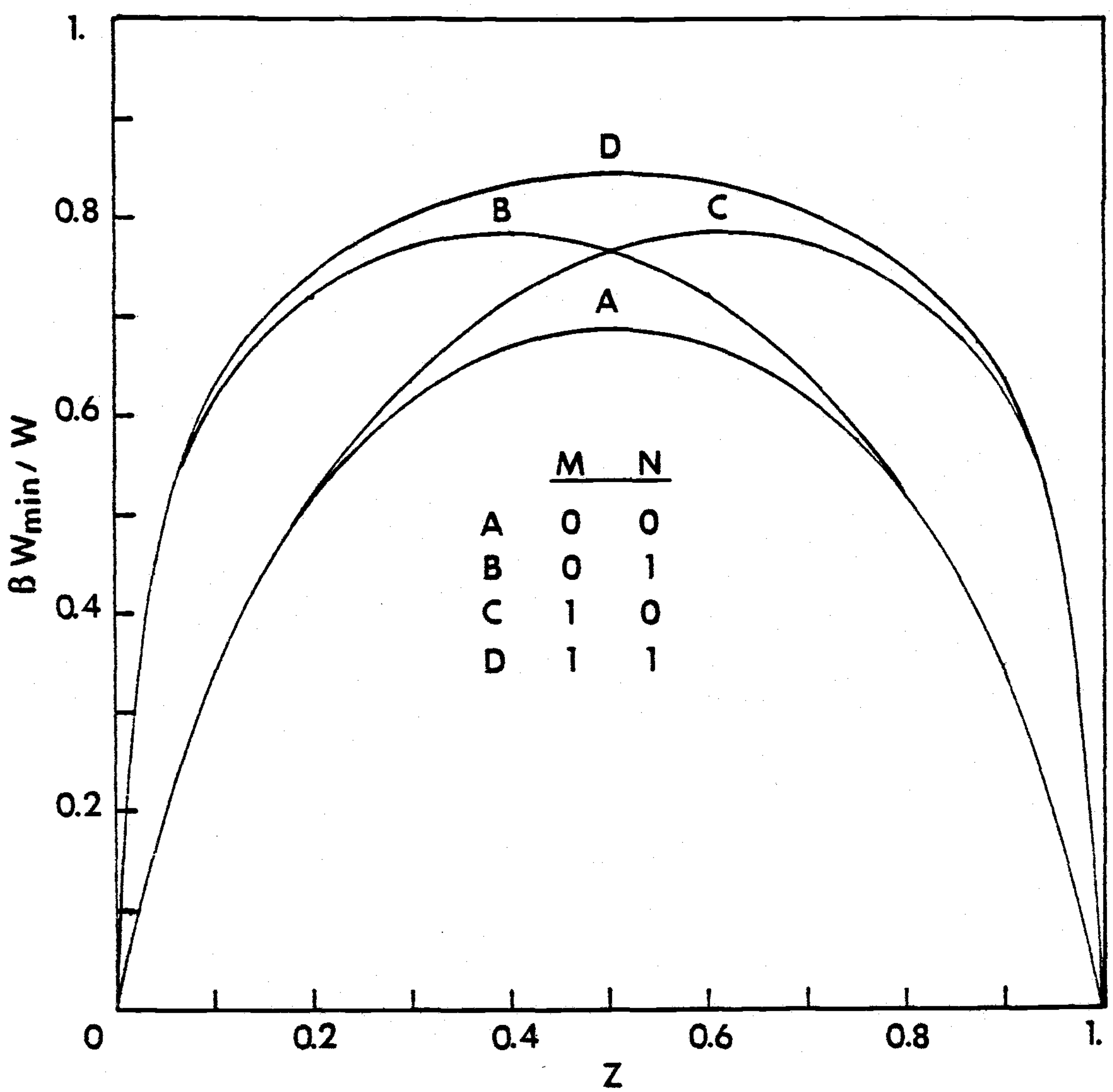



Figure 6. Maximum Thermodynamic Effect of Intermediate Reboilers and Condensers. From Kayihan (1977) M and N denote the number of intermediate reboilers and condensers respectively.

intermediate heat exchangers is plotted as a function of feed composition. It is clear from Figure 6 that the intermediate heat exchangers have a substantial effect on the thermal efficiency of distillation. Further, it can be seen that the intermediate condenser has the greatest effect for low feed concentration, while the intermediate reboiler has the greatest effect for high feed concentrations. It is clear, too, from the results presented by Kayihan that the first additional set of intermediate heat exchangers provides the biggest gain in thermal efficiency. Additional heat exchangers added to the system have decreasing effects on the improved thermal efficiency. These results should provide direction in setting up the experimental column.

The optimum heat loads and locations for the intermediate reboilers and condensers are also available for any number of added heat exchangers. The results for a single set of intermediate heat exchangers are presented here since they are most appropriate to this work. Assuming ideal binary mixtures, the relative heat loads are ultimately a function of feed composition only as given by

$$\frac{Q_{ic}}{Q_C + Q_{ic}} = 1 - \frac{z}{x_{ic}} \tag{4}$$

and

$$\frac{Q_{ir}}{Q_R + Q_{ir}} = 1 - \left(\frac{1 - z}{1 - x_{ir}}\right) \quad . \tag{5}$$

In Equations 4 and 5, $x_{ic}$ and $x_{ir}$ are the concentrations of liquid on the stages where the intermediate condenser and reboiler operate. These concentrations can be found for the optimum conditions from expressions which are dependent only on the feed concentration. The results are as follows:

$$x_{ic} = z^{\frac{1}{2}} \tag{6}$$

and

$$x_{ir} = 1 - (1-z)^{\frac{1}{2}} \quad . \tag{7}$$

With these equations then, the optimum steady state conditions can be found. It should be noted, though, that these results are applicable to ideal binary mixtures and are at best only approximations when applied to nonideal mixtures.

## Dynamic Model

The mathematical model necessary to predict the dynamic behavior of tray type distillation is developed in this section. The equations making up the model are first developed generally and then are simplified to fit the expected experimental situation. The presentation given here follows closely the derivation given by L. A. Gould (1969).

For a tray column, a general tray of index "i" may be represented as shown in Figure 7. The symbols used in Figure 7 are defined in the nomenclature.

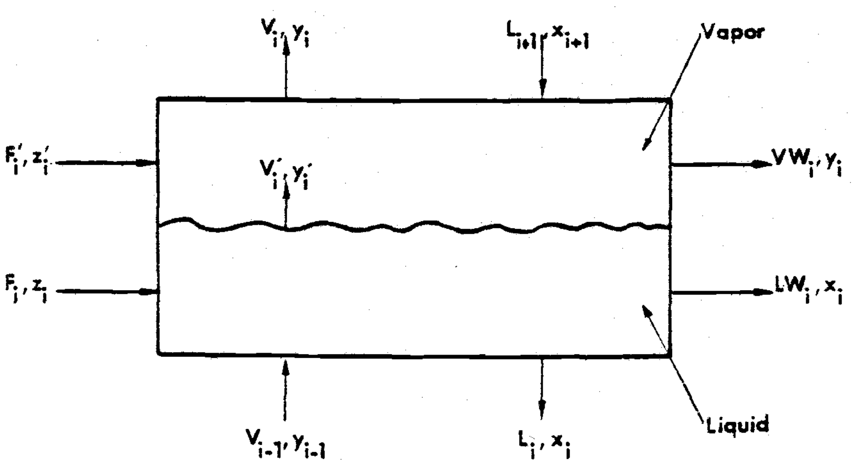



Figure 7. Generalized Tray

Taking a mass balance on the light or volatile component in the liquid phase gives

$$\frac{d(H_i x_i)}{dt} = V_{i-1}y_{i-1} - V_i'y_i' + L_{i+1}x_{i+1} - L_i x_i + F_i z_i - LW_i x_i . \quad (8)$$

A similar mass balance taken on the vapor phase gives

$$\frac{d(h_i y_i)}{dt} = V_i'y' - V_i y_i + F_i'z_i' - VW_i y_i \quad . \quad (9)$$

An overall mass balance on the liquid gives

$$\frac{dH_i}{dt} = L_{i+1} - L_i + F_i - LW_i + V_{i-1} - V_i' \quad . \quad (10)$$

The same balance taken over the vapor phase gives

$$\frac{dh_i}{dt} = V_i' - V_i + F_i' - VW_i \quad . \quad (11)$$

Now the relationship between $y_i$ and $x_i$ is related to the equilibrium relationship which is given by

$$y_i^* = f(x_i, P_i) \quad . \quad (12)$$

In Equation 12, $P_i$ is the pressure above the tray.

In general, the vapor leaving the liquid phase is not in equilibrium with the liquid, thus a tray efficiency must be used to relate $y_i^*$ to $y_i$. In terms of the vapor compositions, the efficiency is given as

$$E_i = \frac{y_i - y_{i-1}}{y_i^* - y_{i-1}} \quad . \tag{13}$$

To complete this set of equations, some additional relationships are necessary. First, the liquid flow, $L_i$, is generally a nonlinear function of the stage holdup and pressure drop. This can be expressed as

$$L_i = g(H_i, P_{i-1} - P_i) \quad . \tag{14}$$

The vapor holdup above the tray is some function of pressure or

$$h_i = h(P_i) \quad . \tag{15}$$

In addition, the pressure drop for vapor flowing through liquid is a nonlinear function of the vapor flow; thus

$$P_{i-1} - P_i = p(V_{i-1}) \quad . \tag{16}$$

Finally, if equilibrium boiling, constant heat of vaporization, and minimal vapor holdup in the liquid can be assumed, then

$$V_{i-1} = V_i' \quad . \tag{17}$$

Equations 8 through 17, as presented above, are sufficient to model the dynamic behavior of binary distillation. However, this general model is too complicated and can be greatly simplified for expected situations. The following assumptions are generally valid and serve to simplify the model.

1. The vapor phase holdup ($h_i$) can be neglected as small in comparison to the liquid phase holdup ($H_i$).
2. Usually no vapor products are withdrawn, thus $VW_i = 0$.
3. In most cases, the feed enters at or below the bubble point so that $F' = 0$.
4. The liquid phase holdups may be assumed constant since the liquid flow transients are generally much faster than the composition transients.
5. Since the pressure drop across the column is small in comparison to the absolute pressure, the system behavior is assumed to be independent of pressure.

Based on these assumptions, the model equations are modified considerably. Equation 8 (for the general tray) becomes

$$H \frac{dx_i}{dt} = V(y_{i-1} - y_i) + L(x_{i+1} - x_i) \tag{18}$$

where V is the vapor rate inside the column. The vapor rate is continuous throughout the column for conventional columns with saturated feed and no heat losses. L in Equation 18 is the liquid flow rate and is equal to $L_l$ or $L_u$, where these indicate the flows in the lower and upper sections of the column. Here again the liquid rate is continuous for the adiabatic conventional column.

For the bottom reboiler, the general equation becomes

$$H_o \frac{dx_B}{dt} = - Vy_o + L_1x_1 - Bx_B. \tag{19}$$

And the overall mass balance for the reboiler is

$$L_1 = B + V. \tag{20}$$

The holdup for the reboiler is given by $H_o$ since it is usually much larger than that of the tray. The feed tray equations become

$$H \frac{dx_f}{dt} = V(y_{f-1} - y_f) + L_u x_{f+1} - L_1 x_f + Fz \tag{21}$$

and

$$L_1 = F + L_u \ . \tag{22}$$

A total condenser (as used in the experimental setup) having no appreciable holdup can be covered simply with a mass balance or

$$D = V - L_u \ . \tag{23}$$

The general tray equation can be given for the top tray as follows:

$$H \frac{dx_t}{dt} = V(y_{t-1} - y_t) + L_u(y_t - x_t)$$

where $y_t = x_D$ .

The Equations 1 through 23 given above, together with the vapor-liquid equilibrium relationship and the tray efficiencies are sufficient to simulate the dynamic behavior of binary distillation provided the assumptions are valid. These equations constitute the basic structure for the computer program used to simulate the experimental results. Modifications are necessary, of course, to account for the complications of heat losses and intermediate heat exchangers. For these cases, the liquid and vapor flows will vary from tray to tray. In addition, certain applications of the intermediate heat exchangers may require some modifications to account for additional liquid withdrawals.

The model is now in hand so we close the books and head for the laboratory.

# IV. EXPERIMENTAL EQUIPMENT DESIGN

The results presented and discussed in this work were performed on distillation equipment specifically designed and constructed for that purpose. A portion of the equipment used existed prior to this project and had been used as a distillation experiment in the undergraduate Unit Operations Laboratory. This existing apparatus provided a basis for the new equipment. To begin with, the existing equipment is briefly described.

## Existing Equipment

The existing distillation equipment included three interchangeable columns, a reboiler with low pressure steam coils, a bottoms pot, an overhead condenser with a solenoid actuated reflux valve, distillate collection flasks, and a feed system which includes the feed pot, pump and tubing necessary to supply feed to the columns. All of this equipment is contained in and supported by a tubular framework. Pictures of the equipment may be found in Appendix A.

The existing distillation columns are all approximately 1.8 m tall, and are constructed of 100-mm-(4 in)-ID glass sections of varying heights. The sections are flanged together to form the columns. The columns are arranged around the bottoms pot and the overhead condenser in such a way that any one of the columns can be operated individually with the heat exchanger and feed systems. An individual column is connected to the bottoms pot below and the overhead condenser above by two 100-mm-(4 in)-ID glass elbows via two bellows flanges. The glass elbows are mounted on the bottoms pot and overhead condenser with special swivel flanges so that the elbows can be adjusted for connection to the desired column.

The glass reboiler used as the main heat exchanger for the system is a Corning HEB-9 heat exchanger with approximately 1.5 $m^2$ of surface area. The reboiler is mounted vertically in such a way so that it operates as a thermosyphon reboiler. Low pressure (approximately 10 psig) steam is supplied to the coils of the reboiler and is exhausted as condensate at atmospheric pressure. A pressure gauge is provided to indicate the supply steam pressure and another is provided to follow the pressure of the steam entering the reboiler. A valve is located on the steam line between these two gauges; the valve is used to adjust the steam pressure entering the reboiler. The temperature of the liquid being heated inside the reboiler can be followed using the thermowell provided.

The reboiler is connected at the top via a reducing elbow to the side of the bottoms pot. At the bottom the reboiler is connected by 40-mm-(1.5 in)-ID glass pipe to the lower outlet of the bottoms pot. This arrangement of reboiler and bottoms pot allows the bottoms liquid to be well circulated when the reboiler is operating. The bottoms pot mentioned here is a Corning VSD 50 spherical vessel approximately 0.6 m in diameter with four necks to accept reboiler, drain, column and sample connections. To allow measurement of overall column pressure drop, a glass stand pipe is connected into the reboiler-bottoms pot line. Completing the reboiler system, 6-mm-OD steel tubing connects the bottoms pot to the feed pot and serves as the bottoms product line.

The overhead condenser and reflux valve are located above the columns on the floor above the reboiler system. The condenser itself is a Corning HE 4 coil type heat exchanger with approximately 0.5 $m^2$ of heat transfer area. Cold water is supplied to the internal coils of the condenser and is exhausted to a nearby drain. The system is arranged so that vapor condensed inside the heat exchanger falls down to a collector and can then either be taken off as product or be returned to the column as reflux. This feat is accomplished by the reflux valve. The valve is opened or closed by a solenoid, which in turn is activated by the reflux ratio controller. This controller has adjustable stops which can be moved separately to produce a desired reflux ratio. Once the reflux valve has moved liquid to the overhead product line, the liquid moves via 25-mm-(1 in)-ID glass tubing to two in-line spherical distillate collection vessels. These vessels can be drained into the feed pot. Included in the product line is a sample tap for the overhead product and a small heat exchanger for product cooling.

Both the bottoms product and distillate product are drained into the feed pot (a large spherical vessel exactly the same as the bottoms pot. Here in the feed pot the mixing and storage of the two product flows take place so that a continuous feed may be introduced into the column. The feed is pumped to the column by an Eastern D-11 centrifugal pump through 6-mm-OD tubing. The flowrate of liquid feed can be measured by a Brooks Sho-Rate rotameter which was calibrated for liquid flow from 0-2000 ml/min (the calibration curve is available in Appendix B). The feed material in the feed pot can be circulated, mixed or sampled by appropriate manipulation of valves on the pump discharge line and on the feed pot drain line. A schematic diagram of the entire setup is included in Appendix A.

The distillation equipment just described is adequate for the undergraduate laboratories performed on it. However, it was found to be insufficient for the proposed experiments in a number of areas. First, the existing columns lack sufficient vapor and liquid ports for use with the intermediate heat exchangers. Second, the columns are without temperature-measuring devices on each stage. Finally, the dynamic response experiments performed for this thesis are best when done using high efficiency trays designed so as to reduce the effect of liquid flow transients. Because of these deficiencies, a new bubble cap tray column, along with the required heat exchangers and instrumentation, was designed and constructed for the experiments reported in this thesis.

## New Equipment

The first priority in constructing the new equipment was the column itself along with the necessary bubble cap trays and instrumentation. For this reason, it is described first followed by descriptions of the intermediate heat exchanger design.

### Distillation Column Design

The column was designed to fit into the empty space in the existing support structure so that the existing heat exchangers, feed system, and refluxer could be used just as with the other three columns. This new column, like the others, is approximately 1.8 m tall and is constructed of 10 separate tubular glass sections, each of which is 100 mm (4 in) in inside diameter and 150 mm tall. The glass sections are connected together by flanges to form the column. Between each section, two gaskets and a bubble cap tray are fitted and are held in place by the flange bolts. There are ten of these bubble cap trays held between the sections; an additional tray, a gasket and 100-mm-(4 in)-ID glass spacer were added at the top to bring the column to the required total height. At the top and bottom of the column 90°, 100-mm (4 in)-ID glass elbows are attached via flanges to the column. The elbows complete the column so that it can then be connected by bellows-type flanges to the bottoms pot below and the overhead condenser above in the same manner as described for the existing columns. As with the columns already installed on the support structure, this distillation column was firmly fixed above and below to the framework. Pictures of sections of the column are included in Appendix A.

Bubble cap tray design. The bubble cap trays installed in the new distillation column were designed and constructed by the author specifically for the project described in this thesis. Their design was chosen to meet the criterion set up for the experiments. The design can be seen in Figure 8 where the top and cross-sectional views of an assembled tray are shown. As can be seen from Figure 8, the major components of the bubble cap tray include the tray itself, the risers, the bubble caps and the downcomers.

The arrangement of these components on the tray was influenced by a number of considerations. First, it was desired to construct a tray with the highest contacting efficiency given the small tray area, thus the three bubble cap design was chosen. The caps were placed on the tray in such a manner so that the greatest distance separates them. This was done to avoid "vapor blow through." To insure quick liquid drainage from the tray, thus reducing the effect of liquid flow transients, two liquid downcomers were provided for each tray. Finally, the liquid sample port on each tray is designed both for liquid sampling and for liquid withdrawal.

The tray itself as well as the bubble caps and risers are made of polypropylene. The downcomers are all made from 13-mm-OD Teflon tubing with the exception of the downcomers for the first stage; these are of 13-mm-OD glass tubing. Beginning now with the tray itself, each of these parts are described in turn.

The tray itself was constructed from 19-mm-polypropylene sheet, and thus is 19 mm thick in general. The tray is circular and is 127 mm in diameter with positions for three bubble caps and their associated risers along with the two downcomers and the liquid port. The outer 13 mm of the tray is cut down 3 mm to allow for gasket seating and centering of the glass section on the tray. The three positions for the bubble caps and risers are located as shown in Figure 8. The tray is drilled through and tapped with 25-mm-(1 in) National Taper Pipe Thread (NPT) in the three places to provide for riser support; a 3-mm counterbore 32 mm in diameter is also included to provide seating for the bubble cap. Two 13-mm-ID holes located as shown in Figure 8 are provided for the downcomers. A 2-mm-ID weep hole is also included; this hole allows for tray drainage when the system is shut down.

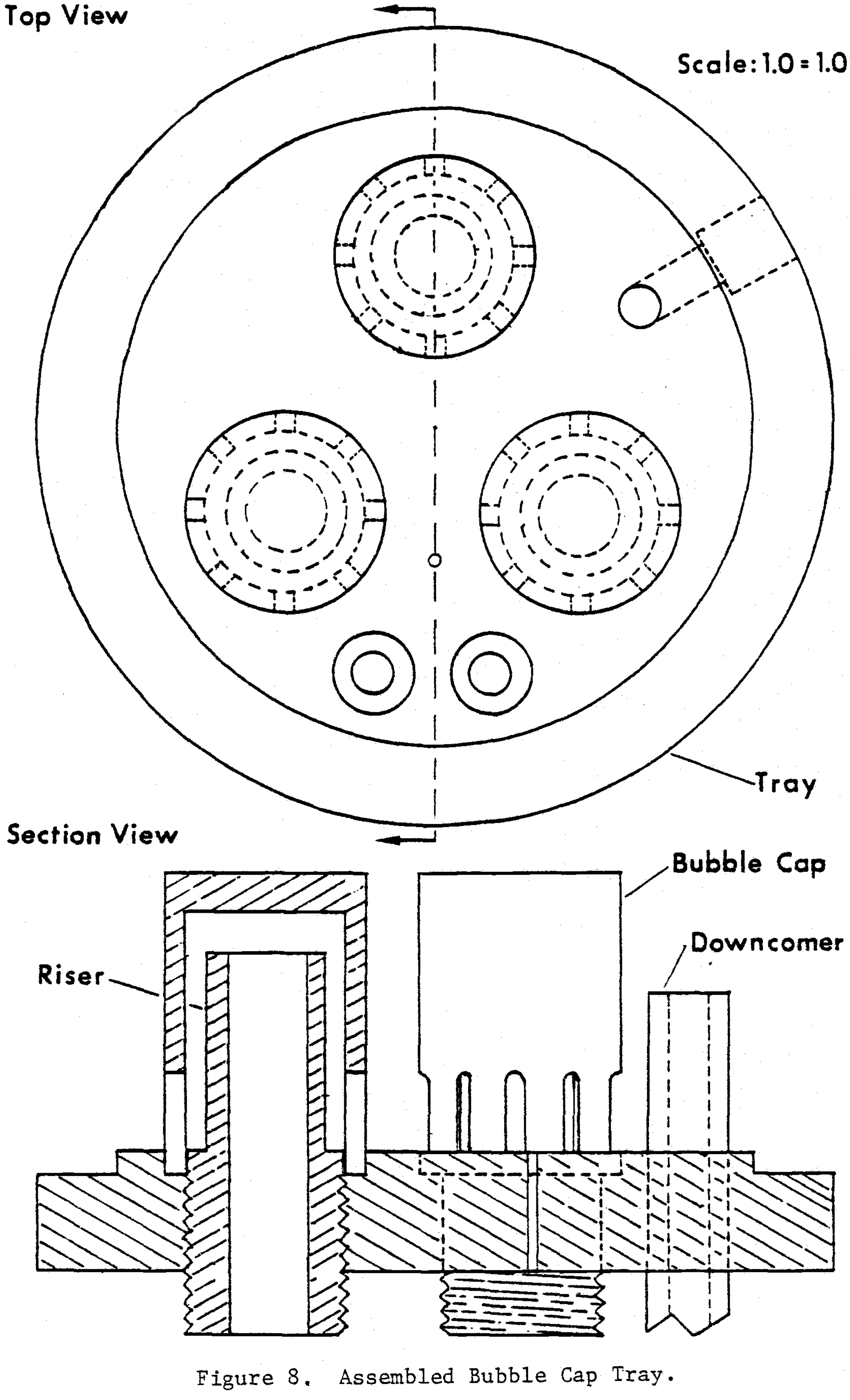



Figure 8. Assembled Bubble Cap Tray.

The liquid sample port completes the tray. The port is located as shown in Figure 8 and is formed by the intersection of a 6-mm-ID hole 13 mm deep in the tray face and a 6-mm-ID hole drilled from the side. The side hole is tapped 12 mm deep with ½-in-(13 mm) NPT.

The risers are made from 25-mm-diameter, tubular polypropylene stock and are 63 mm long. The lower 29 mm section of the riser is approximately 25 mm in diameter and is threaded with 1-in-(25 mm) NPT. The upper section of the riser is 19 mm in outside diameter. A 13-mm-ID hole extends completely through the riser lenghwise. To attach the risers to the tray, they were screwed into the trays from the bottom until the top of the upper section of the riser stood 32 mm above the face of the tray.

The bubble caps were machined from 32-mm-OD, tubular polypropylene stock. They are 48 mm long and 32 mm in outside diameter. A 24-mm-ID hole is centered and extends to a depth of 41 mm from the bottom of the cap. Finally, eight slots 3 mm wide and 13 mm tall are spaced equally around the bottom of the cap. These slots are located 3 mm up from the bottom of the cap. The caps are placed in position on the trays over the risers and are welded in place.

The downcomers are cut from 13-mm-OD and 6-mm-ID Teflon tubing to a total length of 194 mm. The tubes are cut on an angle at the lower end so that the liquid can flow easily from the tubes. The downcomer tubes are inserted through the holes provided for them on the tray so that the tops of the tubes extend 25 mm above the face of the tray. The Teflon tubing is rigid and relatively insensitive to heat so that the downcomers do not bend or fall from the holes during column operation.

The completed bubble cap tray shown in Figure 8 with the liquid sampling device shown in Figure 9 provide the necessary functions of liquid-vapor contacting and liquid withdrawal for the experimental distillation column. The sampling device shown in Figure 9, when screwed into position in the liquid sample port, makes possible

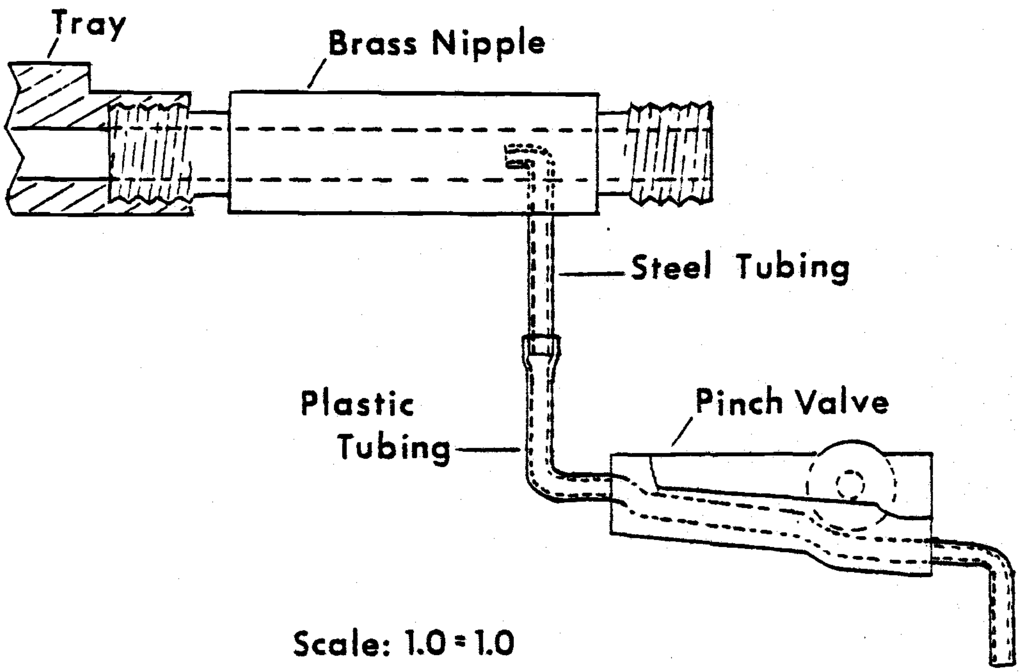



Figure 9. Liquid Sampling Device.

simultaneous liquid sampling and liquid withdrawal.

Stage design. The stage itself completes the column design. It includes the glass section, the liquid and vapor feed ports, and the thermocouple port. The stage provides the means for both measurement of and interaction with the liquid-vapor contacting taking place on the tray. An overall picture of the stage design presented here can be found in Figure 10. There, top and quarter-section views of the stage are presented.

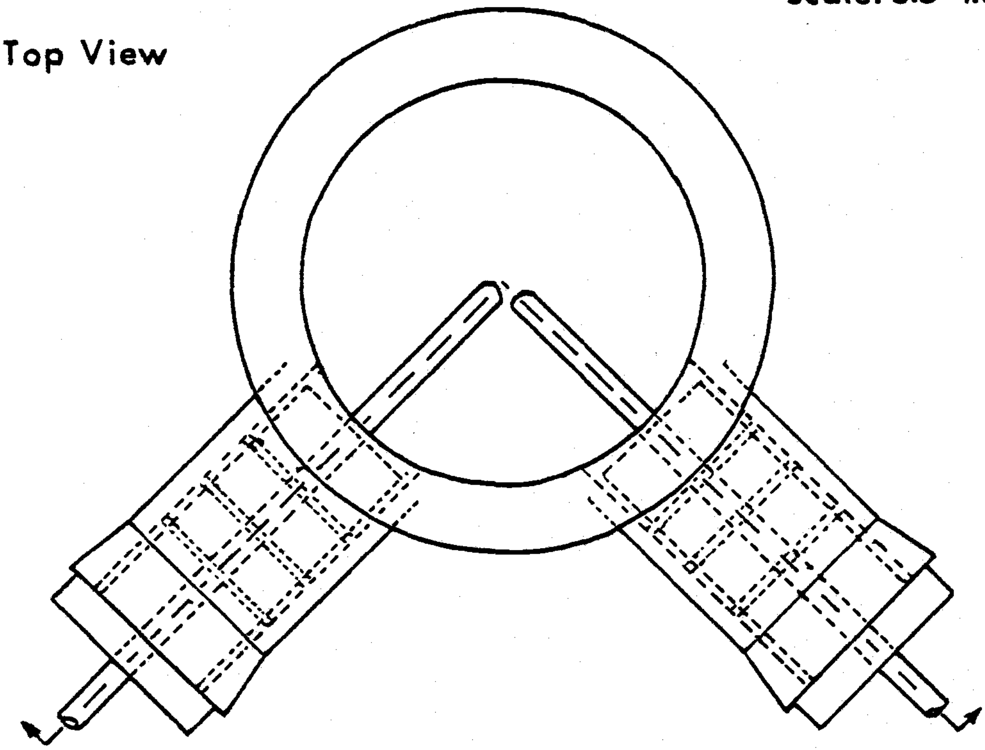
As can be seen from Figure 10, the glass sections are constructed with two 40-mm-(1.5 in)-ID glass ports arranged 90° apart around the middle of the section. It is these ports which give the column its flexibility. As applied in the operation of the column for this work, the right port (as shown in Figure 10) is devoted for use with liquid feed and thermocouple locations. The left port is used for vapor feed and vapor sampling.

Both ports in all the sections making up the column are fitted with special polypropylene plugs. The plugs are 82 mm long overall and are 38 mm in diameter on the smaller end and 51 mm in diameter on the larger end. Three rubber "O" rings are fitted into three separate grooves spaced 13 mm apart beginning 19 mm from the smaller end of each plug. The use of these "O" rings on the plugs means that a plug will give an air-tight seal when fitted into the port and also can be removed and replaced repeatedly.

The plugs for both the liquid feed and vapor feed ports are drilled through in the center to give approximately a 6-mm-ID hole. A 15.2-cm length of 6-mm-OD stainless steel tubing is fitted through both holes and sealed. The tubes provide liquid feed and vapor sampling capabilities. Inside the stage, the tubes are bent downwards to provide for vapor pickup for the sampler and to allow liquid feed to the center of the plate. Outside the stage, the tubing ends are fitted with valves and caps. The liquid feed line is capped until required for use with the feed line or with the liquid return line

Scale: 0.5 = 1.0

Top View



Quarter Section View

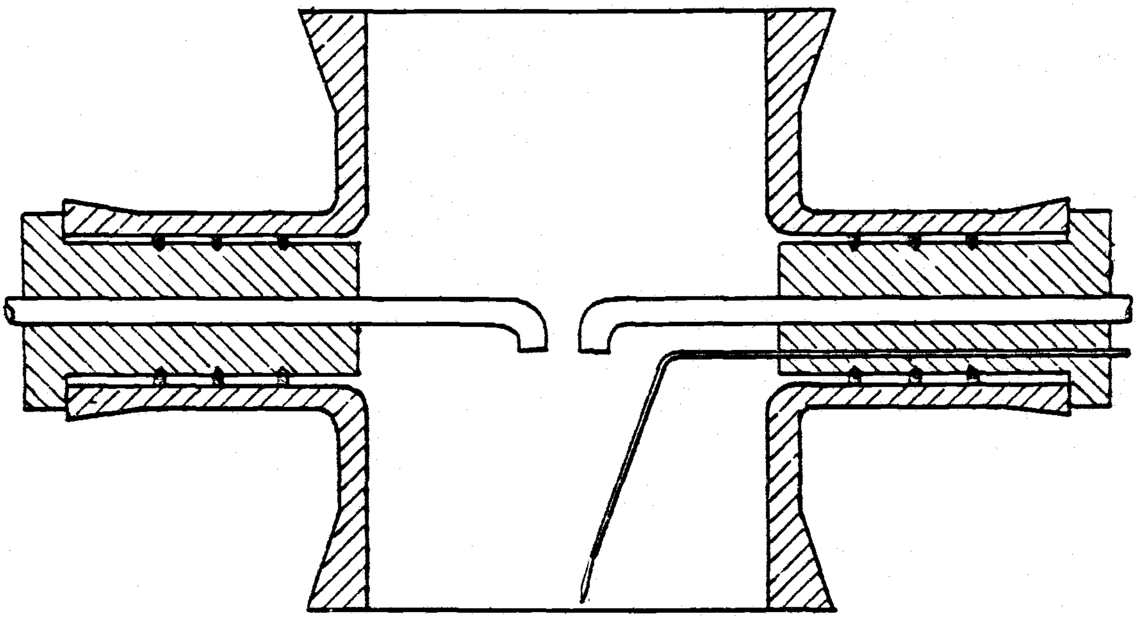


Figure 10. Individual Stage Design.

from one of the intermediate heat exchangers. The vapor sample line is fitted with a valve and is used with the vapor sampling device described later in this chapter.

The thermocouple port is located in the same plug as the liquid feed line. The plug is fitted with 3-mm-OD stainless steel tubing which extends through the plug parallel to the 6-mm-OD tubing. This 3-mm-OD tubing is positioned 10 mm below the 6-mm-OD tubing. Steel-clad, 1.6-mm-OD, K-type thermocouple wire is placed through the tubing and bent so that the thermocouple bead is positioned directly above the tray. Outside the section, the thermocouple-tubing connection is sealed with heat-shrink rubber tubing. The thermocouple wires extending from the steel clading are connected via Omega NMP Miniature Thermocouple Connectors to K-type thermocouple wire. This wire is routed to and connected to the measuring devices.

With all of the elements just described, the column is complete and prepared for connection to the intermediate heat exchangers.

## Heat Exchanger Design

Before constructing the intermediate heat exchangers necessary for this study, the decision had to be made about the number of intermediate exchangers to use. Based on the information presented in Chapter III, it is clear that the first set of intermediate heat exchangers are of the greatest benefit. For this reason, and because the fewer heat exchangers involved mean fewer problems in operation, it was decided to use only one intermediate reboiler and one intermediate condenser for this work.

Design and construction of these two intermediate heat exchangers were based primarily on three considerations. First, it is necessary to have the heat exchangers act to add or withdraw heat on a single stage. This means that material is taken from and returned to the column at the same stage. Second, the rate of heat exchange at the intermediate heat exchanger must be both measurable and controllable; this is to allow for correct distribution of heat duties.

Finally, the operation of the exchanger systems must be flexible enough so that the operational point of the system may be moved from stage to stage in the column. This flexibility is, in part, insured by the design of the stages.

The resulting heat exchanger systems are described in this section along with the additional bottoms reboiler.

Intermediate reboiler. The intermediate boilup necessary in the stripping section of the heat integrated column is provided by the reboiler system designed for this purpose. It is the complete system which will be described here and which is presented in Figure 11.

The basis of the system (see Figure 11 for details as necessary) is a Corning HEB-4 coil-type heat exchanger, which contains 0.15 $m^2$ of coil heat transfer area. As shown in Figure 11, low pressure steam is provided to the coils of the reboiler through a supply line which includes a valve and pressure gauge. The valve and gauge are useful for control and measurement purposes.

The glass reboiler is mounted on a structure shared with the intermediate condenser. Both heat exchangers are mounted by connection of the heat exchanger flange to a rectangular steel plate attached to the structure; the steel is cut to match the center and bolt holes of the flange and heat exchanger. A 22.9-cm-diameter, 19-mm-thick polypropylene disk also drilled to match the bolt holes of the flange and plate is centered and bolted using gaskets between the flanges and the plate.

The structure supporting the two heat exchangers is located below the level of the distillation column, even though it was the original intention to mount the intermediate reboiler next to and at the level of the stage where the boilup is desired. Mounting the reboiler at the level of the stage would mean that the intermediate reboiler could be operated as a thermosyphon reboiler. However, with a thermosyphon reboiler, the composition of liquid in the reboiler would not necessarily be the same as that on the stage; neither

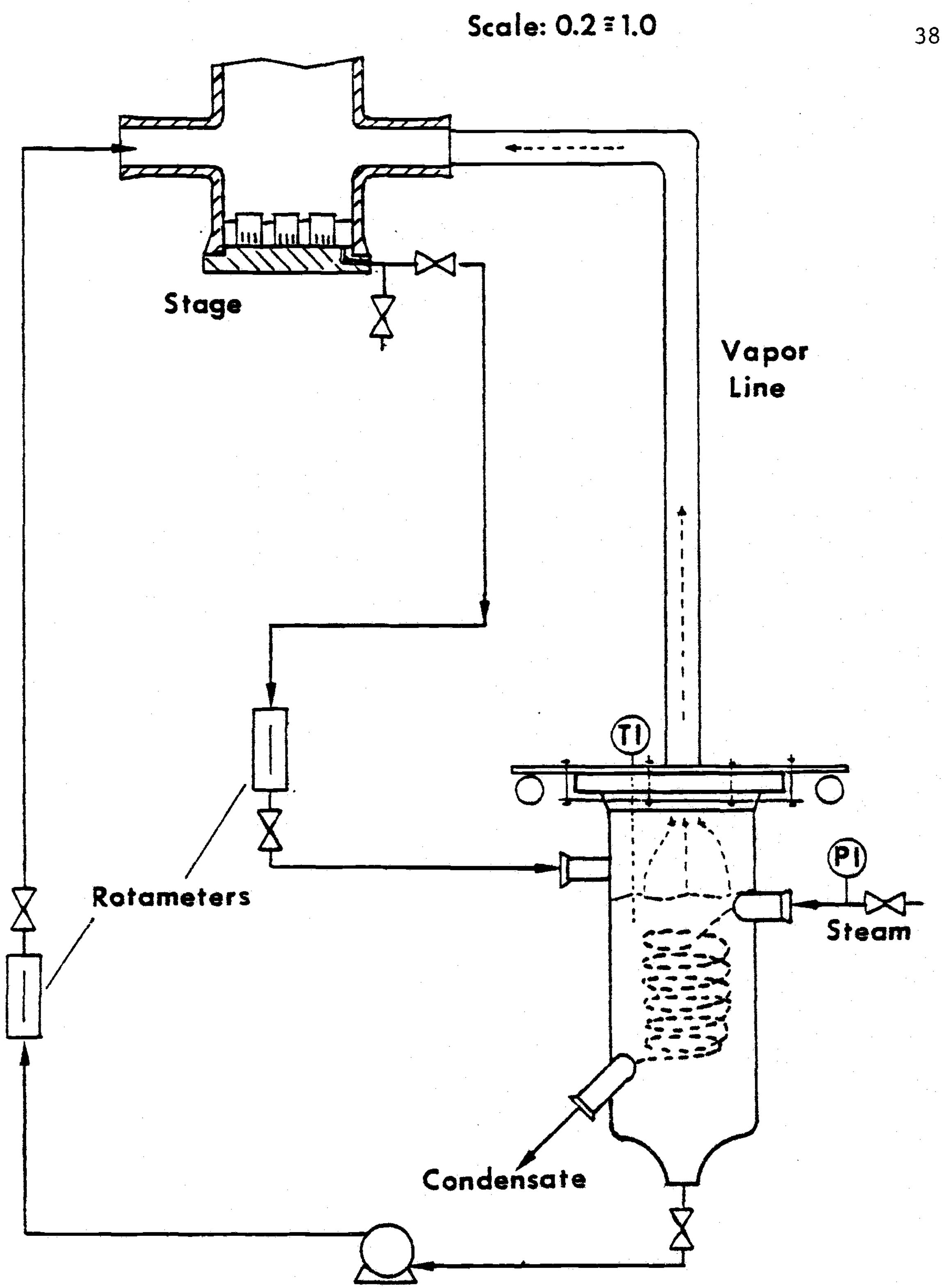



Figure 11. Intermediate Reboiler System Design.

would the vapor supplied by the reboiler be similar to that above the stage. For this reason and because of support difficulties, the intermediate reboiler is mounted below the column.

To insure that the reboiler operates with liquid very similar in concentration to the liquid on the tray, a substantial liquid recycle is necessary. In the system used here, a relatively large amount of liquid is drawn from the stage and fed to the reboiler where part is vaporized and returned to the stage as vapor while the rest is returned as liquid recycle. As the ratio of liquid flow received to liquid flow recycled back to the stage approaches unity (with vapor boilup making up the difference between the two), the reboiler liquid concentration becomes the same as the liquid on the tray. Of course, to achieve this with non-zero boilup rate requires high liquid flow rates, and practical considerations will determine the approach of the two concentrations.

Specifically, as shown in Figure 11, the liquid feed to the reboiler is drawn from the tray through the liquid sample port. The liquid flows by gravity through 9-mm-OD plastic tubing and a Fischer-Porter rotameter before entering the top of the reboiler. The Fischer-Porter rotameters used in the intermediate heat exchanger systems (there are three) are all the same; they are calibrated for liquid flow in the range of 0-1900 ml/min. The liquid flowrate is adjusted using the valve installed immediately following the rotometer on the 6-mm-OD line to the reboiler.

The recycle liquid drawn from the bottom of the reboiler is pumped through the recycle line rotameter and back to the stage via 6-mm-OD tubing. At the stage, the liquid is returned through the liquid feed port (see Figure 10). The pumps used for liquid return on both the intermediate reboiler and condenser systems are Micro Pump air-driven gear pumps capable of pumping liquid at flowrates up to 2000 ml/min. The high pressure air to drive the pumps is filtered and regulated using Speedaire Filter/Regulators. It is by adjustment of the regulator valve on the air line that the liquid recycle flowrate is most easily controlled.

Introduction of steam into the coils of the reboiler results in vapor production, and this vapor (the intermediate boilup) is directed through an insulated, 25-mm-ID Tygon tube back to the stage. The Tygon vapor line is connected on one end to the reboiler cap and on the other to a flange connected to the stage vapor sample port (see Figure 10). To attach the vapor line to a vapor sample port, the plug is first removed, then a flange and soft insert are fitted together over the glass port, and finally the flange attached to the vapor line is bolted to the port flange using a gasket.

A thermocouple has been inserted into the intermediate reboiler to allow measurement of the reboiler liquid temperature. The output of this thermocouple is monitored during operation to gain information about the liquid concentration. The thermocouple port is constructed in the same way as those already described for the stages. Steel-clad, K-type thermocouple wire is inserted through the 3-mm-OD steel tubing fitted into the reboiler's plastic cover. The thermocouple wire is extended so that the bead reaches well into the liquid. Outside the reboiler, the thermocouple-tubing connection is sealed as before with heat-shrink rubber tubing.

Intermediate "condenser". Intermediate condensation is required in the rectifying section of the heat integrated distillation column; this requirement is fulfilled for the experimental column by the intermediate "condenser" system described here. The quotes are used for good reason; this heat exchanger system, though it is designed to function as a condenser by withdrawing heat, is not actually a condenser. An inspection of this system design (as shown in the flow diagram presented in Figure 12) shows that the heat exchanger is designed to subcool and return the saturated liquid withdrawn from the column, rather than to condense vapor and return saturated liquid.

There are two basic reasons behind the choice for this type of

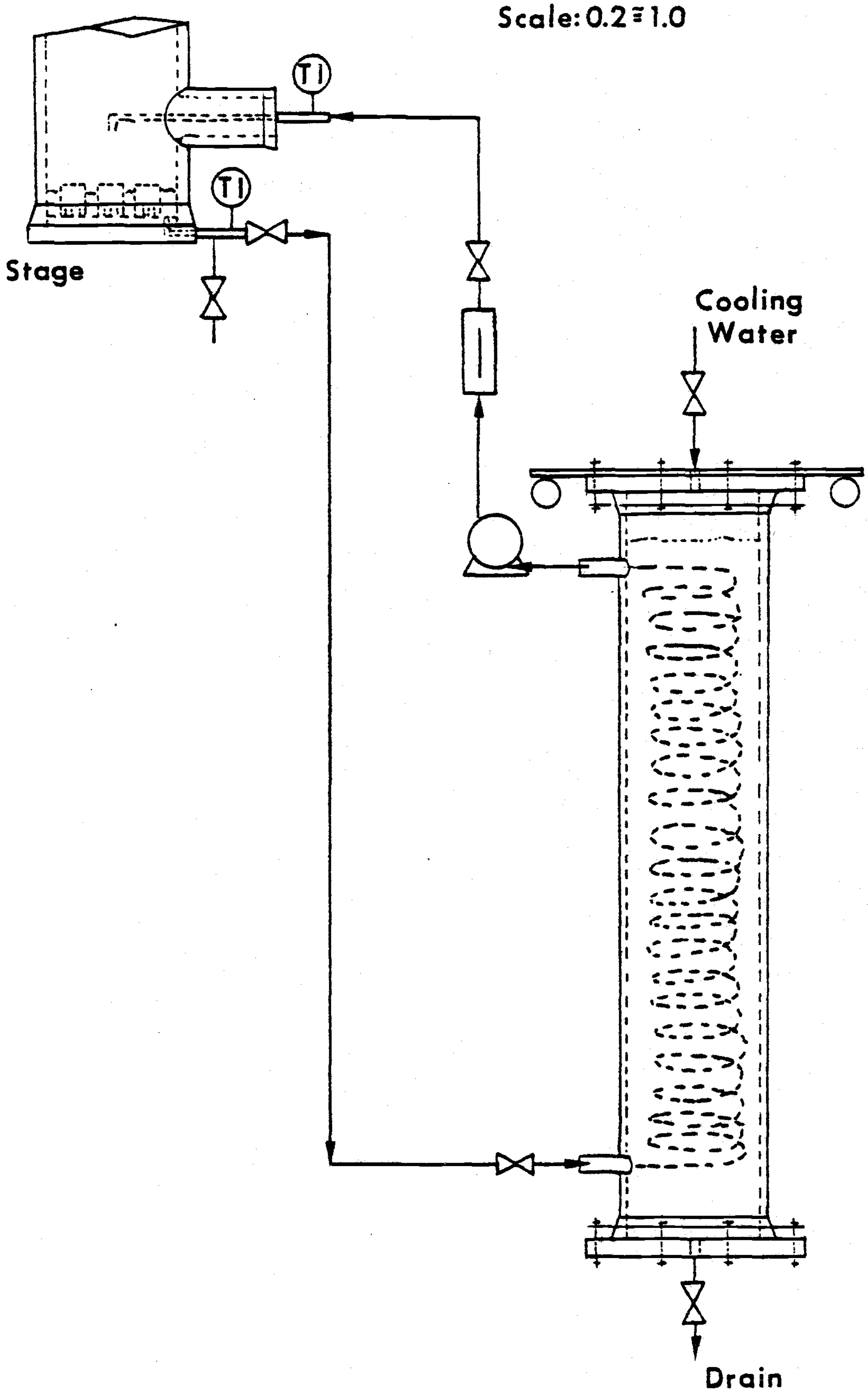



Figure 12. Intermediate "Condenser" System Design.

system. First, for these experiments, it is very important to know and to be able to control the heat removal rate. The condensation-type heat exchanger does not lend itself well to control of the heat removal rate. Secondly, it is important that the intermediate heat exchangers be designed to have minimal effect on the liquid flow transients of the column. The condensing-type heat exchanger would entail some kind of accumulator and this would certainly affect the liquid flow transients. In addition, the "condenser" design presented here is one which operates on a single stage. The subcooled liquid returned to the column from the system described here is best returned to the tray from which it came, while the condensed liquid from the condensing-type system is best returned to a higher tray where the liquid is more concentrated.

The basis for the intermediate condensing system is a Corning HE-4 coil-type heat exchanger. The 100-mm-(4 in)-ID, 61-cm-long jacket is open at both ends and contains coil with 0.5 $m^2$ of heat transfer area. This heat exchanger is mounted alongside the intermediate reboiler in the same support structure. It is mounted in the very same way as the reboiler. As with the reboiler, 22.9 cm-diameter, 19-mm-thick polypropylene disks are used as covers for the open ends of the "condenser." These disks are bolted to the exchanger with flanges as described previously, except that on the bottom no steel plate was used (since the "condenser" is hung from the structure and is not supported on the bottom).

Cold tap water provides the cooling medium for the "condenser." The water is introduced to the jacket side of the heat exchanger through the tap in the top plastic cover and is exhausted through the drain provided in the bottom plastic cover. Appropriate manipulation of the valve on the cold water inlet line will insure constant cooling water level in the jacket when the drain valve is completely open. A small vent hole in the top cover is provided to prevent air pressure buildup in the heat exchanger.

The liquid to be cooled in the condenser is withdrawn from the tray through the liquid sample line and is eventually introduced into the coil side of the heat exchanger. The liquid sampler has been modified for use with the intermediate "condenser" to include a thermowell. The thermocouple inserted in the thermowell allows the temperature of the liquid leaving the tray to be measured. After leaving the sample port the liquid flows through 6-mm-OD plastic and steel tubing to the lower coil entrance. Once through the "condenser," the cool liquid is pumped from the heat exchanger through a rotameter and finally back to the stage through the liquid feed port. Here, too, the liquid feed line is altered to include a thermowell. The thermocouple positioned in this well allows detection of the return liquid temperature. The pump and rotameter used with this "condenser" system are the same models as those described previously for the reboiler system.

The two temperature measurements on the circulating liquid along with the liquid flow measurement provided by the rotameter allow an accurate calculation of the heat removal rate. The liquid flowrate through the "condenser" coils can be varied by adjustment of the regulator valve on the air supply to the pump. These two items cover the necessary conditions for knowledge and control of the heat removal rate in the intermediate condenser.

A review of Figures 8 through 12 will indicate that the column, along with the intermediate heat exchanger systems, is designed to allow substantial flexibility in operation. The equipment is constructed to accommodate liquid feed, liquid withdrawal, vapor feed, vapor sampling and liquid sampling on any tray in the column. Thus, the intermediate heat exchange systems can be operated on nearly any stage in the column.

Additional bottoms reboiler. In addition to the intermediate reboiler and condenser just described, another reboiler was added to supplement the main bottoms reboiler. This reboiler was included to provide adequate boilup rates for a number of different binary systems. The additional reboiler is a Corning HEB-6 coil-type heat exchanger having approximately 0.5 $m^2$ of heat transfer area. This reboiler is connected on top to the bottoms pot directly opposite the existing reboiler via a 80-mm-(3 in)-ID bellows-type flange and an 80-mm-(3 in)-ID 90° elbow. The reboiler is tied in below to the 40-mm-(1.5 in)-ID glass line running between the bottoms pot and the stand pipe. A valve is included on the glass line below the reboiler so that liquid circulation can be stopped when the heat exchanger is not in use.

## Peripheral Devices

In addition to the equipment just described, some other more standard pieces of equipment were used in the course of the experiments. These include a control panel and the equipment mounted on it, a refractometer, a vapor sampler and an aspirator system. The control panel is constructed with positions for three Analog Devices model 2036 digital thermometers, a Honeywell model 112 multipoint temperature recorder, a Honeywell controller, a Honeywell three pen recorder and three Fischer-Porter rotameters. Each of these devices are mounted on the panel and are wired with electricity as appropriate.

The digital thermometers along with the multipoint recorder are the basic temperature-sensing instruments for the experiment. Both are designed for use with K-type (Chromel-Aluminel) thermocouples. Once wired with extension thermocouple wire and the Omega miniature thermocouple connectors for each input, these units can be connected with the thermocouple leads extending from the stages and heat exchangers of the column. The measuring devices can be connected to the inputs in one of two ways depending on the information desired. The digital meters provide immediate and accurate (± .1°C) readings for individual inputs. While the digital thermometers do have a scan mode, they only accept six inputs; thus the usefulness of the scan is limited. By contrast, the multipoint recorder gives relatively inaccurate (± .5°C) continuous temperature readings. Further, it can accept up to 24 inputs through which the recorder cycles periodically for sampling and recording. The two hook-up schemes are shown in Figure 13. The digital thermometers were generally used for taking data while the multipoint recorder was used only to follow trends and for start-ups.

The other devices on the panel include the rotameters which have been described already. They are mounted side by side on the panel to facilitate comparison, especially between the two used in the intermediate reboiler system. The control panel also serves to support the two air-driven gear pumps and their associated filter/regulators. The controller and three pen recorder mounted on the panel were not used in this work. They are a part of the equipment necessary for possible future work with this distillation setup (see Appendix F for more details).

Composition analyses of samples taken during the course of the experiments were performed using a Bausch and Lomb precision refractometer. This refractometer system includes a Sodium vapor lamp and a constant temperature bath. The refractometer was calibrated for the water-methanol system used in the experimental runs; the calibration curve is included in Appendix B.

Vapor samples can be collected through the vapor sample ports using a specially constructed sampling device. The sampling device consists of a length of 6-mm-OD copper tubing coiled inside a tin container 10 cm in diameter and 14 cm in length. With ice packed around the coil and with the tubing connected to the appropriate vapor sample line, the device can be used to collect a vapor sample as a liquid.

Finally, to avoid escape of methanol vapors from the column into

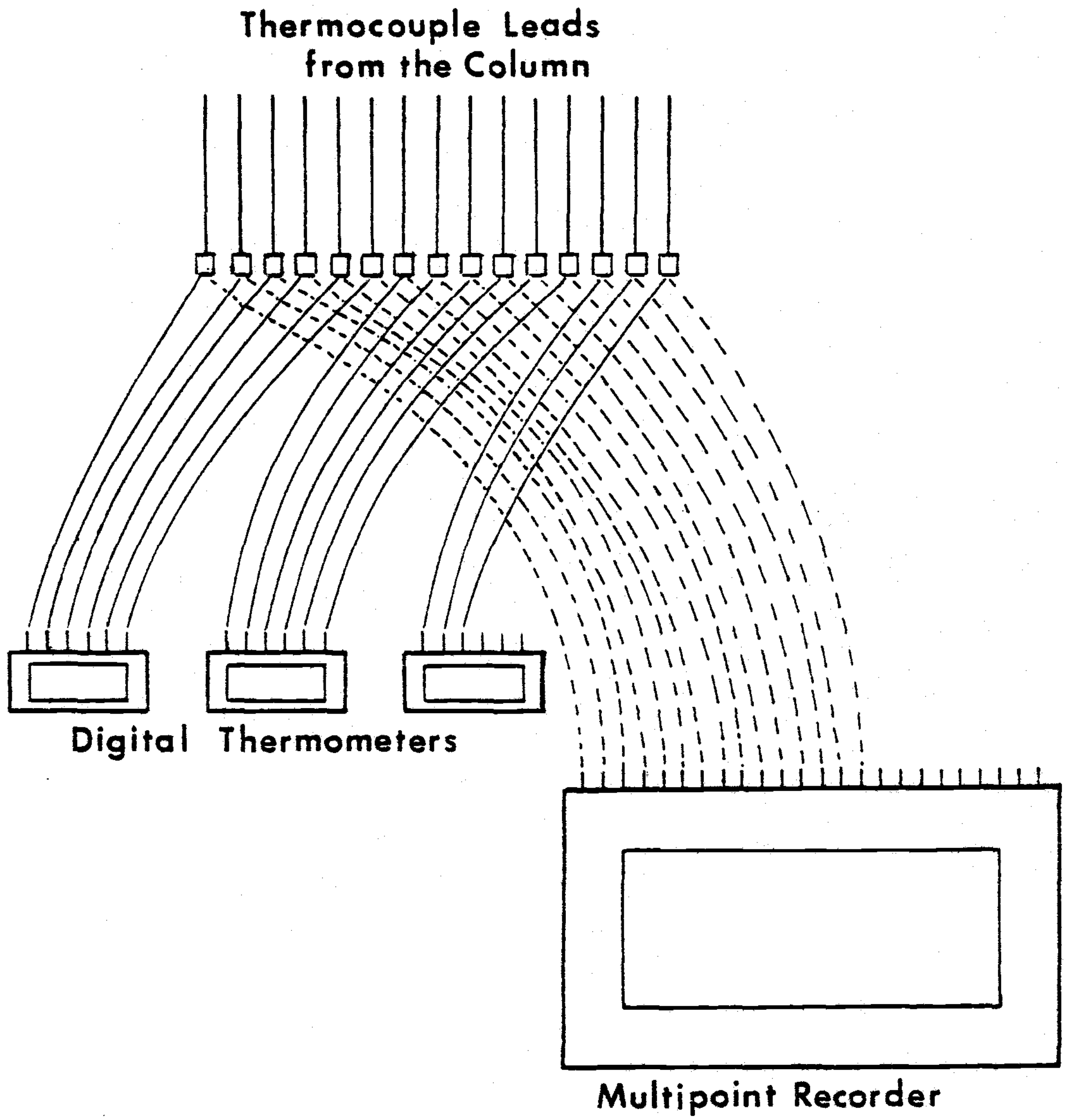



Figure 13. Schematic Diagram of the Temperature Measurement System.

the surrounding laboratory space, the column was capped and operated slightly below atmospheric pressure. An aspirator mounted and connected to the column provides the lower pressure. A water manometer is also connected to the top of the column so that the deviation from atmospheric pressure can be monitored.

At long last the equipment is complete and behold, the results come forth!

# V. EXPERIMENTAL RESULTS

The dynamic responses of both the modified (heat integrated) distillation and conventional distillation, as measured on the experimental column just described, are the major results presented in this section. However, before these responses could be measured, some preliminary information was required. These preliminary results are first presented followed by the dynamic response results.

## Preliminary Results

Two pieces of information are prerequisites for the analysis of the dynamic response data. These are, first, the overall heat transfer coefficients of the glass reboilers used to provide boilup and second, the relationship between the composition of the liquid on a tray and the temperature of that liquid as measured by the thermocouple. Both pieces of information were determined experimentally.

### Heat Transfer Data

The data necessary for calculation of an overall heat transfer coefficient for the reboilers were taken for two reasons. First, the overall heat transfer coefficient is crucial in the calculation of boilup rates for a wide variety of boiling temperatures and condensing steam temperatures. Second, disturbances placed on the system to generate the dynamic data will require calculation of the steam pressure change required to bring about a given change in the boilup rate. This calculation assumes a constant overall heat transfer coefficient over the range of steam pressures and boiling temperatures required in these experiments.

Working with a distilled water charge to the distillation system, the column was operated in total reflux, and the results presented in Figure 14 were collected. In the figure, the total heat transfer rate in the large bottom reboiler is plotted as a function of the temperature drop across the heat transfer coils. The heat transfer rate was determined from measurements of the steam condensate flowrate leaving the coils, while the temperature drop across the coils was determined by difference between the measured temperature of the water boiling in the reboiler and the saturation temperature of the steam supplied to the coils. As can be seen from Figure 14, the data represent a relatively wide span of temperature drops; these represent steam pressures ranging between 122 and 170 kPa (17.7 and 24.7 psia).

The application of linear regression to the results gives the straight line indicated in Figure 14. The slope of this line divided by the heat transfer area for the bottoms reboiler (1.5 $m^2$) is the average overall heat transfer coefficient for this reboiler. From the results this is found to be 190 kcal/$m^2$-hr-°C. By comparison, Corning (1978) reports a value of 350 kcal/$m^2$-hr-°C for all of the reboiler type heat exchangers (regardless of size) when the heat transfer medium is 446 kPa (64.7 psia) steam. They indicate proportionate reductions in the overall heat transfer coefficient for lower steam pressures. Thus the result given above is certainly reasonable and was used for calculation of the results to follow.

The other reboilers included in the distillation system, the additional bottoms reboiler and the intermediate reboiler, are both smaller than the main bottoms reboiler on which these experiments were carried out. However, since the three heat exchangers are the same in shape and design and differ only in size, they should have the same heat transfer characteristics. Thus the overall heat transfer coefficient obtained for the larger reboiler is applied to the smaller ones as well. With this assumption and the heat transfer coefficient presented above, one can calculate the boilup rates

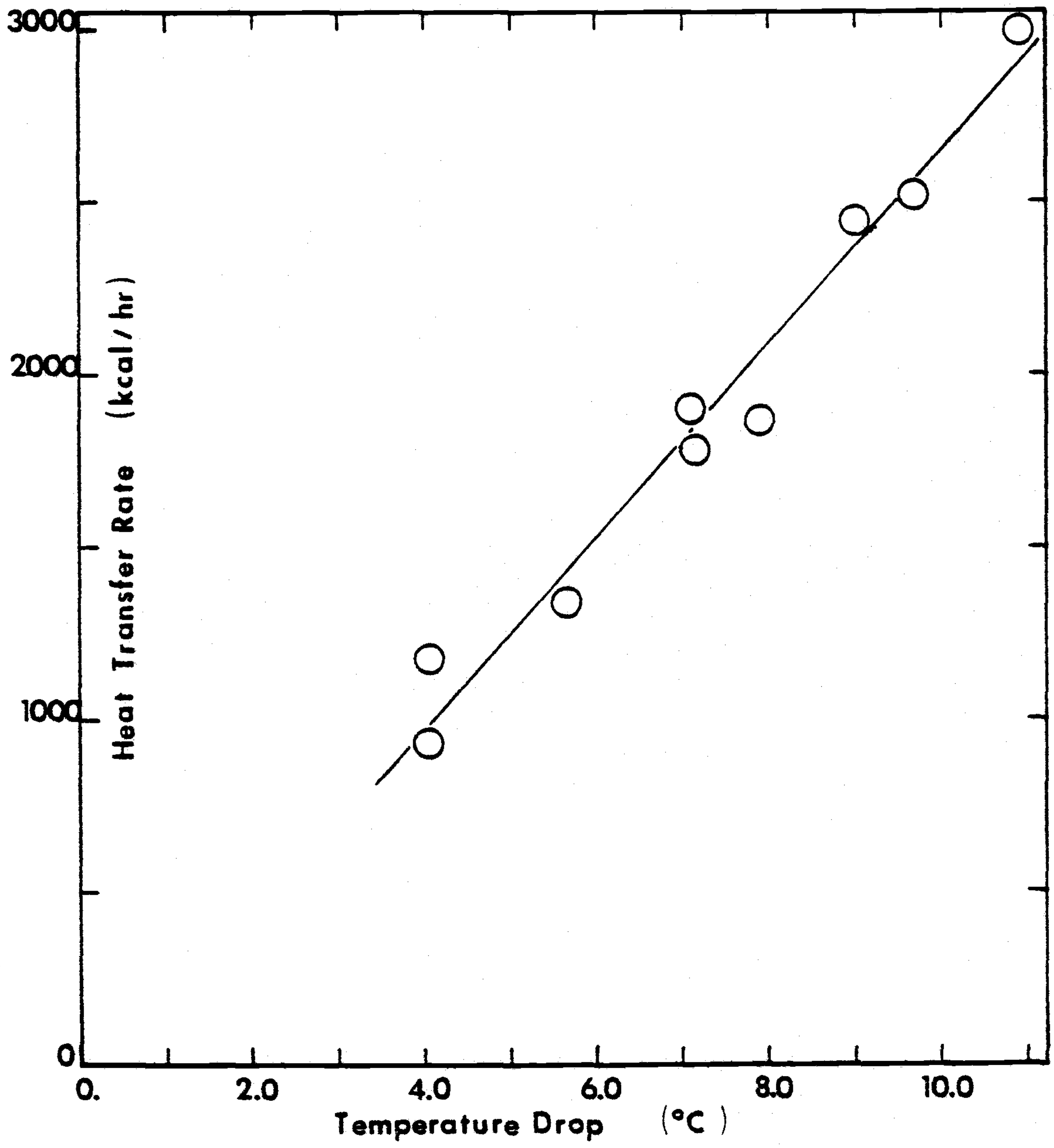



Figure 14. Heat Transfer Rate Results for the Corning HEB-9 Heat Exchanger.

for each of the reboilers over the range of steam pressures encountered in the dynamic experiments.

## Saturated Liquid Data

The distillation column used in this work is fitted with thermocouples installed to measure the liquid temperature on each tray (except for the top tray). To translate the temperatures into liquid concentrations, one must make an assumption about the state of the liquid on the tray. As the title of this section indicates, the assumption made here is that the binary liquid mixture is saturated. This is the most realistic assumption and the one giving the most direct relationship between liquid temperature and liquid composition (for binary mixtures).

To determine the validity of this assumption for the experimental column and the methanol-water system, a number of experiments were performed. With the distillation column operating at total reflux and at a reflux ratio of one, liquid samples from each stage and the bottoms reboiler were analyzed and at the same time the tray liquid temperatures were noted. The results are indicated in the graph shown in Figure 15. Clearly, the experimental results closely match the saturated liquid temperature-composition curve reported by Chu (1950) and plotted as a solid line in Figure 15. The deviations of the experimental liquid temperatures from the saturated temperatures are within the limits of the deviations induced by measurement errors.

Based on these results the saturated liquid assumption is valid for the water-methanol system in the experimental column. Therefore, throughout the remainder of this work, the temperatures measured on the trays are assumed to be saturated liquid temperatures.

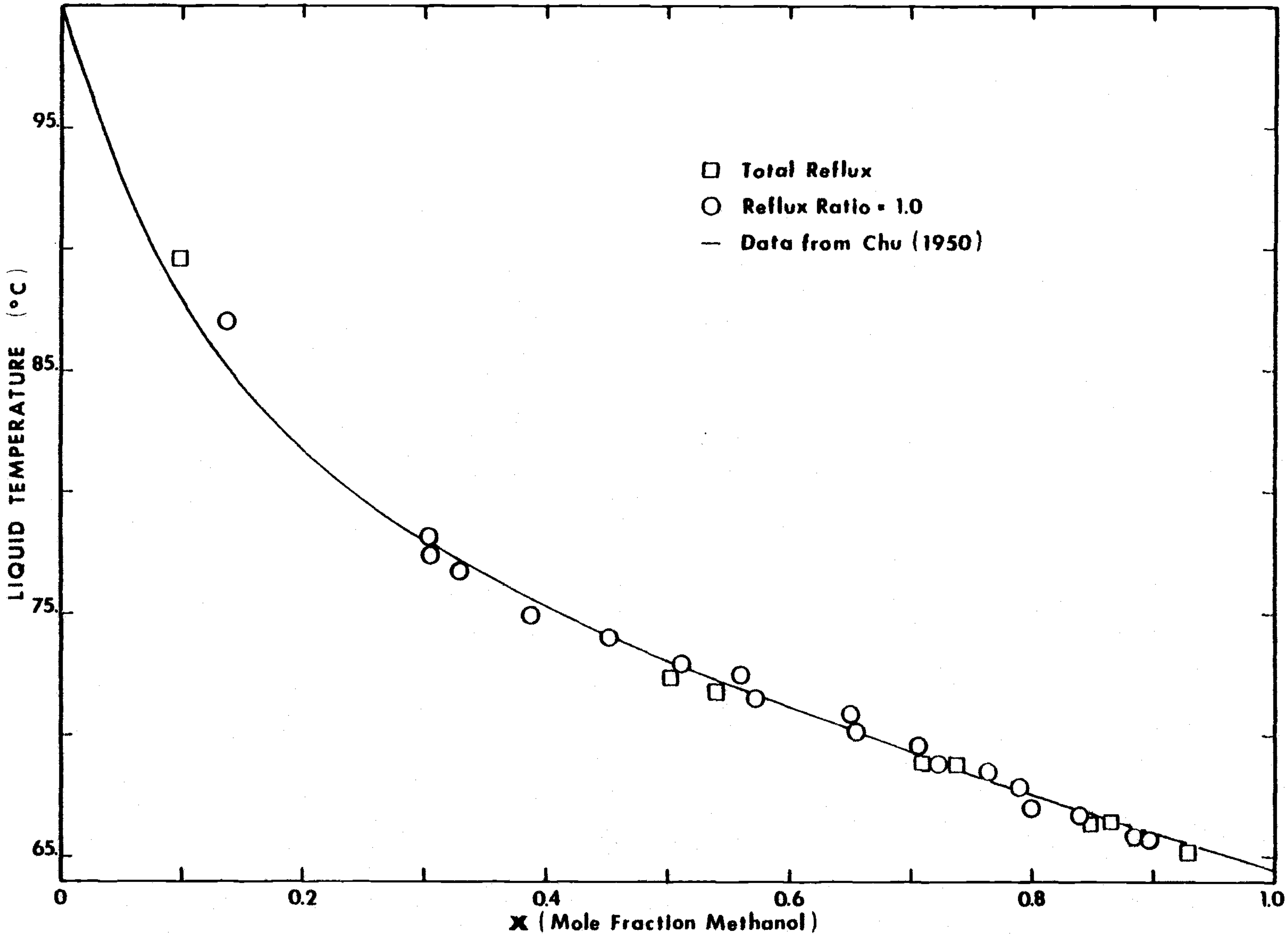



Figure 15. Tray Liquid Temperatures for the Experimental Column using the methanol-water system.

## Dynamic Response Results

The most important results presented in this thesis are the ones contained in this section. With these results it is demonstrated both that the experimental column can be operated as a heat integrated distillation column and that the column can be used to generate useful dynamic response data. In addition, the results presented here can be interpreted to give useful information about the operation of a modified distillation column as opposed to the conventional column. In this section then, the experimental setup and method are briefly described, followed by the results which are both presented and interpreted.

### Experimental Setup

The results were obtained using the experimental apparatus with the methanol-water system. The feed mixture for all the dynamic experiments was maintained at a concentration of approximately 60 mole percent methanol. For a feed of this composition, the optimum intermediate heat exchanger loads and locations can be determined using the results presented in Chapter III. For the 60 percent feed composition, the optimization results indicate that the intermediate "condenser" should be operated on the stage which has a liquid concentration of 77 mole percent methanol and that the "condenser" should provide 22 percent of the total heat removal duty. The intermediate reboiler should be used on the stage with liquid concentration of 37 mole percent methanol and should provide 37 percent of the total boilup duty. These results were calculated for an ideal binary mixture separated to nearly pure products in the experimental column. However, since no better information is available, these results were applied within the constraints of the experimental equipment.

The distillation equipment was connected as shown in Figure 16

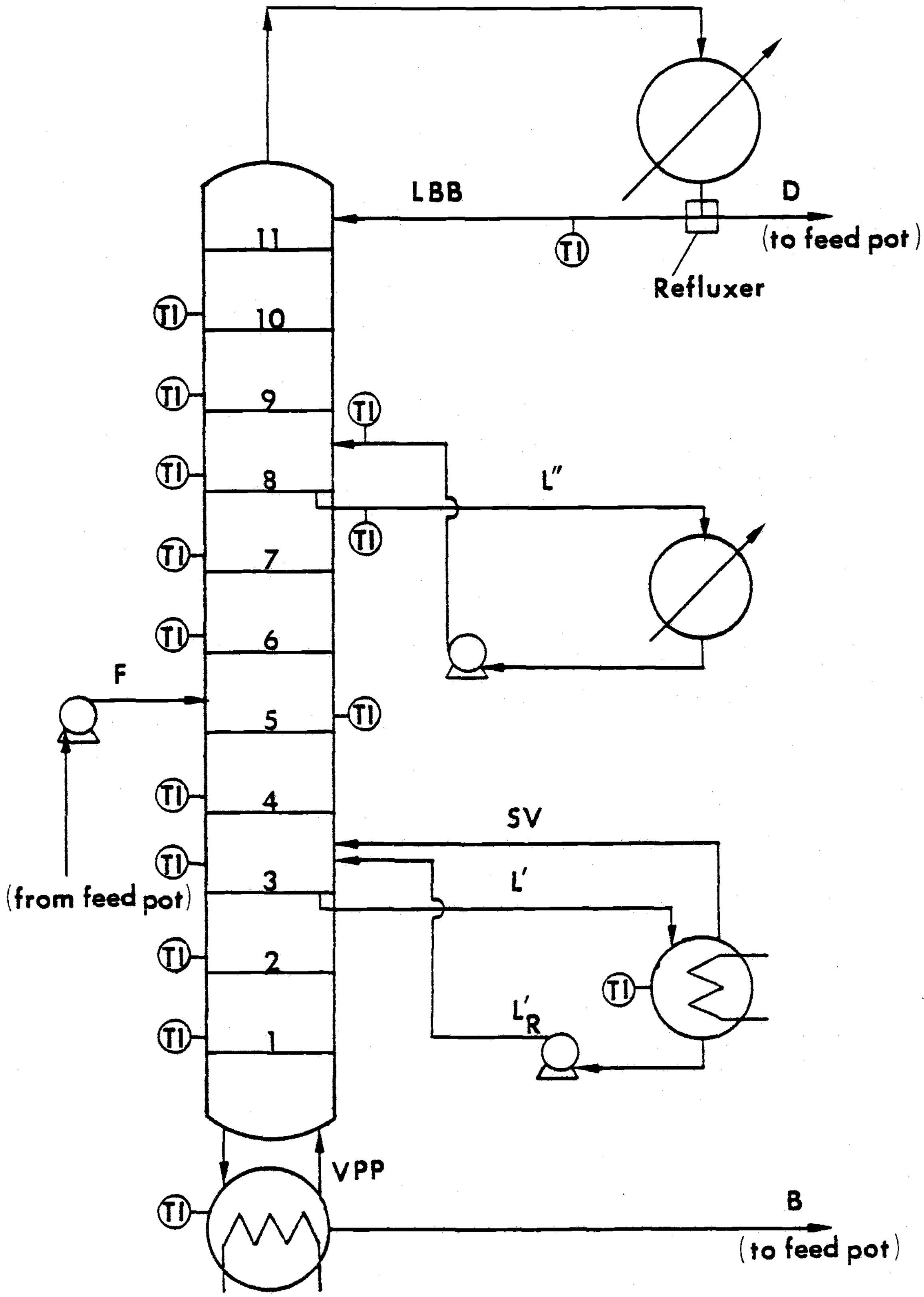



Figure 16. Experimental Setup for the Dynamic Response Experiments.

and charged with a mixture of distilled water and reagent grade methanol. As can be seen from Figure 16, liquid feed entered the column on the fifth stage, the intermediate reboiler provided additional boilup on the third stage, and the intermediate "condenser" operated on the eighth stage. These locations were chosen as the best compromise between optimum locations and the locations best suited for easier column operation. Based solely on the optimization results, the intermediate heat exchangers would have both been placed one to two stages lower. However, the operation of the intermediate heat exchangers in these positions tended to make it more difficult to maintain steady internal flows; for this reason the higher positions (stages three and eight) were chosen. The feed liquid to the column is subcooled, thus for greatest separation efficiency the feed should be introduced above the normal feed location (the stage where the liquid concentration matches the feed composition). Stage five then turned out to be the best feed stage both for the modified column (operated with the intermediate heat exchangers) and for the conventional column (operated without the extra heat exchangers).

It should be noted that the setup presented in Figure 16 is true only for the experiments performed on the heat integrated column. For the dynamic response experiments on the conventional column, the lines connecting the intermediate heat exchangers to the column were closed and the intermediate exchanger systems were shut down. Thus data were taken on the same column with and without the intermediate heat exchangers. The operating procedures for both the conventional column and the modified column are listed in Appendix C.

## Experimental Method

To generate a dynamic response from a distillation column, one or more of the parameters describing the steady-state operation of the column must be disturbed. It is important that the disturbance be initiated with the column operating at steady state so that the dynamic response measured can be attributed to the disturbance. Furthermore, for these studies the response from more than one disturbance would be very difficult to interpret not to mention the difficulty involved in actually imposing simultaneous disturbances. Thus, for the experiments reported here, the distillation columns were disturbed from steady state in a single parameter to generate the measured dynamic response.

There are a number of methods one could use to disturb a system; each of these have advantages and disadvantages. For these experiments a pulse disturbance was chosen as the best method. As shown in Figure 17, a 10-minute, 20 percent positive pulse change was used in all the experiments to generate the column dynamic response. The pulse was chosen so that the system would eventually return to the original steady-state conditions; the duration of ten minutes was

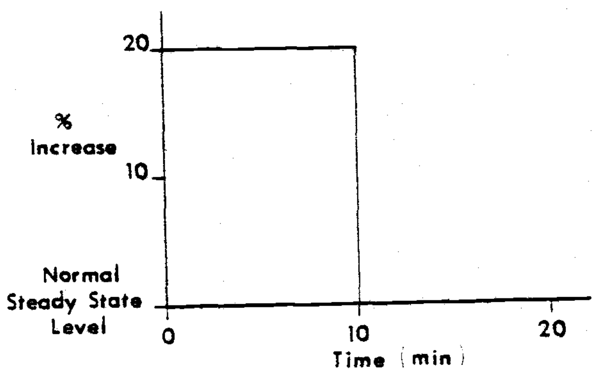



Figure 17. Method of Disturbance

selected as the longest time span which would minimize tendencies toward feed composition and reboiler holdup changes. The 20 percent change was selected so that the observed liquid tray temperatures deviated significantly from the steady-state levels.

Seven separate dynamic response experiments are reported here. These experiments involve disturbances in the following system parameters (please see Figure 16 for explanation of the symbols):

(1) the liquid feed flowrate (F); (2) the external liquid reflux flowrate (LBB); (3) the bottom reboiler boilup rate (VPP); and (4) the liquid flowrate through the intermediate "condenser" (L"). The first three parameters were disturbed in experiments on both the modified and the conventional columns, while the fourth one was disturbed only in an experiment using the modified column. In all the experiments, the pulse disturbance was applied with the column operating at steady state, and the resulting dynamic response was observed by following the liquid temperatures on each tray (excluding the eleventh tray), along with the temperature of the liquid boiling in the bottoms reboiler. Each of the temperatures was sampled at five-minute intervals.

## Experimental Response Results

The steady-state operating conditions for each of the seven experimental runs are tabulated in Table 1. The values presented there are sufficient to check the column operating mass balance and to determine the heat loss from the column given the thermal state of the feed. For each of the runs reported here, the feed is subcooled enough to give an average value of 1.1 for q. Using the information found in Table 1, along with q = 1.1 to calculate an internal column heat balance, one can show that the column experiences a total heat loss equal to a condensation rate of between 3.2 and 3.7 mole/min depending on the experiment. The magnitude of this heat loss is not at all surprising given the fact that the column is constructed of uninsulated glass sections.

The size of the column heat loss, however, does preclude direct application of the optimization results to the operation of the experimental column. The optimization results were derived, assuming both saturated feed and an adiabatic column. Neither of these assumptions are true for the experimental equipment, therefore the "optimum" heat load distribution was used only as an upperbound. As can be determined from the information found in Table 1, the

Table 1. Initial Steady State Operating Conditions for the Experimental Runs

| Column Set Up | Experimental Run (Disturbed Parameter) | Feed | | Distillate | | Bottoms | | Reboiler Boilup Rate | | Intermediate "Condensation" Rate | Reflux Ratio |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | z (mole %) | F (mole/min) | $x_D$ (mole %) | D (mole/min) | $x_B$ (mole %) | B (mole/min) | VPP (mole/min) | SV (mole/min) | (mole/min) | |
| CONVENTIONAL | F | 63.7 | 4.5 | 96.5 | 2.8 | 7.0 | 1.7 | 9.4 | | | 1.0 |
| | LBB | 59.7 | 5.5 | 97.3 | 3.2 | 7.4 | 2.3 | 10.5 | | | 1.0 |
| | VPP | 57.8 | 4.7 | 97.6 | 2.6 | 7.4 | 2.1 | 9.4 | | | 1.0 |
| MODIFIED | F | 65.1 | 4.8 | 97.2 | 3.1 | 7.0 | 1.7 | 9.4 | 1.9 | 1.03 | 1.0 |
| | LBB | 59.7 | 5.3 | 96.9 | 3.1 | 7.3 | 2.2 | 9.3 | 1.9 | 1.03 | 1.0 |
| | VPP | 60.6 | 5.4 | 97.9 | 3.2 | 8.0 | 2.2 | 9.6 | 1.8 | 1.06 | 1.0 |
| | L" | 60.6 | 5.4 | 97.3 | 3.2 | 7.6 | 2.2 | 9.4 | 1.8 | 1.05 | 1.0 |

Note:

For all the runs
- Number of stages 11
- Feed stage 5

For runs with the modified column
- Intermediate reboiler stage 3
- Intermediate "condenser" stage 8

intermediate heat exchangers were operated in these experiments to contribute approximately 15 percent of the total heat duty (neglecting the heat loss). For the intermediate reboiler, that level represents the highest duty it could deliver. The intermediate "condenser" was operated below the "optimum heat duty" to allow for the effect of the internal condensation due to heat loss.

The dynamic response results for the seven experiments are presented in Figures 18-21. The results presented in these figures are the temperature responses of individual stages; they begin at time zero (with application of the disturbance) and continue until the tray liquid temperatures approach their initial values. The responses shown in Figures 18-21 are those of selected stages. Complete tabulations of data for all the experimental runs appear in Appendix D.

The dynamic response results for both types of columns (conventional and modified) are plotted side by side when the results are from the same disturbance. This was done to facilitate comparison of the two responses. It should be noted here that a comparison can be made only on the basis of the similar separations and the identical column design. No effort was made to make the overall heat input and removal rate the same for the conventional and modified column experiments, so a comparison cannot be made on that basis. Since the experiments were performed on the same column, similar separations and similar heat duties are mutually exclusive for the two column setups. Thus a choice has to be made and, in this case, the comparisons are made based on similar separations.

The conventional and modified column responses to the disturbance in feed flowrate are shown in Figure 18. There, the temperature responses of the first three stages above the bottom reboiler are shown since these stages are below the feed stage and, thus, experience the most disturbance from the subcooled feed liquid. The stages above the feed showed no deviation from the steady-state levels in both experiments.

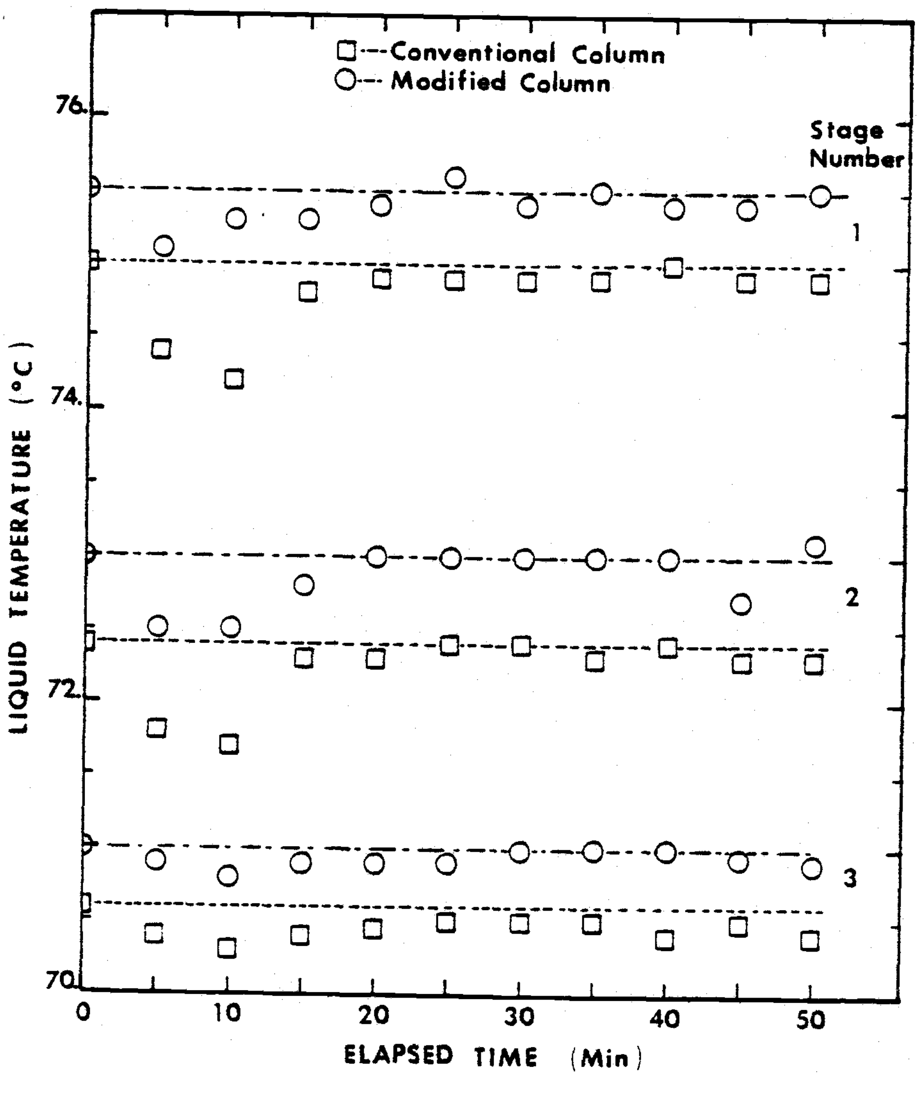



Figure 18. Stage Temperature Responses to a Pulse Disturbance in the Feed Flowrate (F).

It is easily observed by comparing the results presented in Figure 18 that the stage liquid temperatures deviate less with the modified column than with the conventional column. It is apparent that for this type of disturbance the modified column is more restrained in its response than is the conventional column. Although the modified column shows more restraint during the time of the disturbance, there seems to be no difference between the two column setups in the quickness of their return to steady state.

This restrained response of the modified column should be expected; it is due to the presence of the intermediate reboiler. By removing liquid and returning vapor to the column, the intermediate reboiler has the same effect as a surge tank. Thus the disturbance seen by the lower end of the column (in this case stages one through three) is moderated and, in turn, the tray temperature response is moderated for the modified column. If the feed were saturated or partially vaporized instead of being subcooled, the upper stages would also be disturbed and one would expect the intermediate condenser to moderate the deviation in the modified column.

The results for the experiments with a disturbance in the bottom boilup rate presented in Figure 19 help to confirm that the intermediate heat exchangers moderate the dynamic response of the modified column. As can be seen from the responses shown in Figure 19, the 20 percent increase in the boilup rate causes a change in the column operation and, as a result, the tray liquid temperatures begin to rise. Once the pulse is finished and the boilup rate is returned to the normal level, the temperature rise is reversed; the liquid temperatures drop dramatically. The temperature drop is due to the large amount of liquid flowing down the column. With the sudden drop in the boilup rate, excess liquid, which had been held up on the trays by higher pressure drop across each stage, is suddenly free to move down the column. The effect of all this is to drop the stage liquid temperatures below the steady-state levels, just as in the previous experiments.

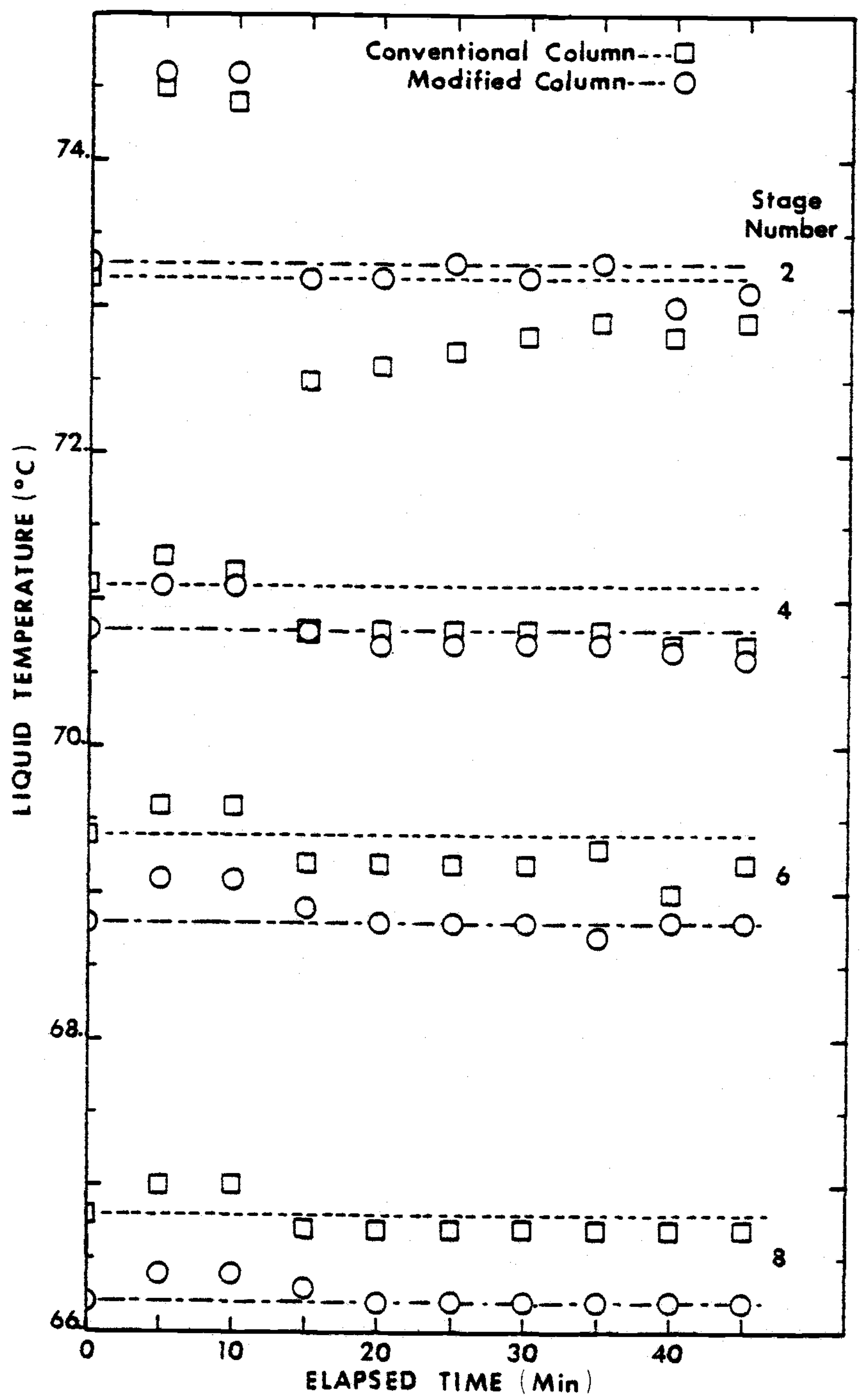



Figure 19. Stage Temperature Responses to a Pulse Disturbance in the Bottom Boilup Rate (VPP).

Consider now what effect the intermediate heat exchangers have on the column response. As can be seen from Figure 19, the initial positive deviations in the liquid temperatures are very similar for both the columns. In the lower stages, the intermediate reboiler provides no moderating effect during the time of the pulse and, indeed, should not be expected to since it is adding to the increased vapor rate. In the upper stages, the column heat loss serves to reduce the effect of the increased vapor flow and the intermediate "condenser" adds to that effect. With the end of the pulse and the increased liquid flow down through the column, the resultant negative temperature deviations are significantly different for the two columns. The intermediate reboiler in the modified column acts to moderate the liquid flow down the column just as it did in the previous experiments. The result is that for the modified column the lower stages experience significantly lower negative temperature deviations after the pulse. In the upper stages, the intermediate "condenser" in conjunction with the heat losses serves to reduce the influence of the increased vapor rate so that the negative temperature deviations after the pulse are reduced or eliminated for the stages in the modified column.

The dynamic responses resulting from the pulse disturbances in the external liquid reflux rate are presented in Figure 20. As seen in Figure 20, the effect of the increase in the liquid reflux, and eventually the increase in liquid flow through the column, is to induce a negative deviation in the tray liquid temperatures similar to that experienced in the first experiments (see Figure 18). Contrary to the previous results, the responses shown in Figure 20 do not clearly show the expected moderating influence of the intermediate heat exchangers. The stages below the feed show the exact same liquid temperature deviations for both the conventional and modified columns. Above the feed it is a different story, the stages in the modified column show smaller deviations from steady state than do the stages in the conventional column. These results are exactly

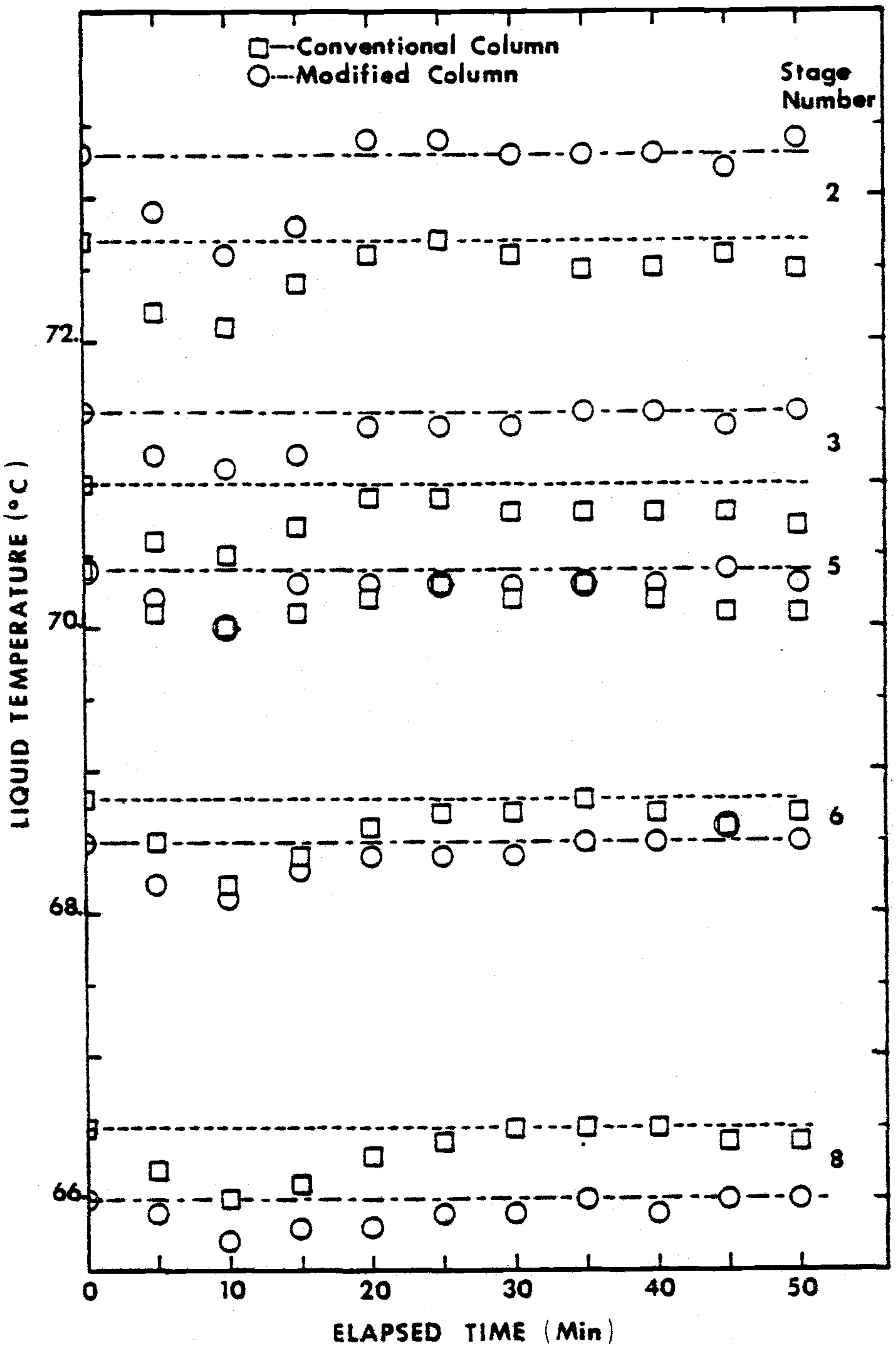



Figure 20. Stage Temperature Responses to a Pulse Disturbance in the Liquid Reflux Rate (LBB).

the opposite of what might be expected. The intermediate reboiler should have the greatest moderating effect for disturbances involving liquid flow, while the condenser should have greatest effect with vapor flow disturbances.

There are probably two reasons for the observed effect of the intermediate heat exchangers as shown in the responses of Figure 20. First, the nearer the heat exchanger is to the disturbance, the greater its effect on the resulting deviation. In this case, the intermediate reboiler is relatively far from the disturbance in the liquid reflux flowrate, thus it has a small effect on the resulting deviations. Second, the moderating effect of the intermediate "condenser" may be more due to the way it is designed than anything else. A reduction in the temperature of the liquid entering the "condenser" will result in a reduction in the heat removal rate and, thus, the condensation rate. A reduction in the condensation rate would definitely serve to moderate the increased liquid reflux to the column.

The partial response results for pulse disturbance in the liquid flowrate through the intermediate "condenser" are shown in Figure 21. Of course, this experiment was performed only on the modified column. The results given in Figure 21 serve to confirm that the intermediate "condenser" promotes the same effect as an actual condenser. The results show that the stages below the intermediate "condenser" stage (stage eight) experience temperature drops indicating increased internal liquid reflux rates. The stages above stage eight also show temperature drops, indicating reduced vapor flows above stage eight.

Throughout the previous discussion, the strange or unexpected responses have been ignored. In some experiments and usually for the lower stages, unexpected temperature jumps appear. Since these variations are single points, they were not considered in the discussion. These variations from the expected temperature responses are due to variations in the liquid feed flowrate or in the

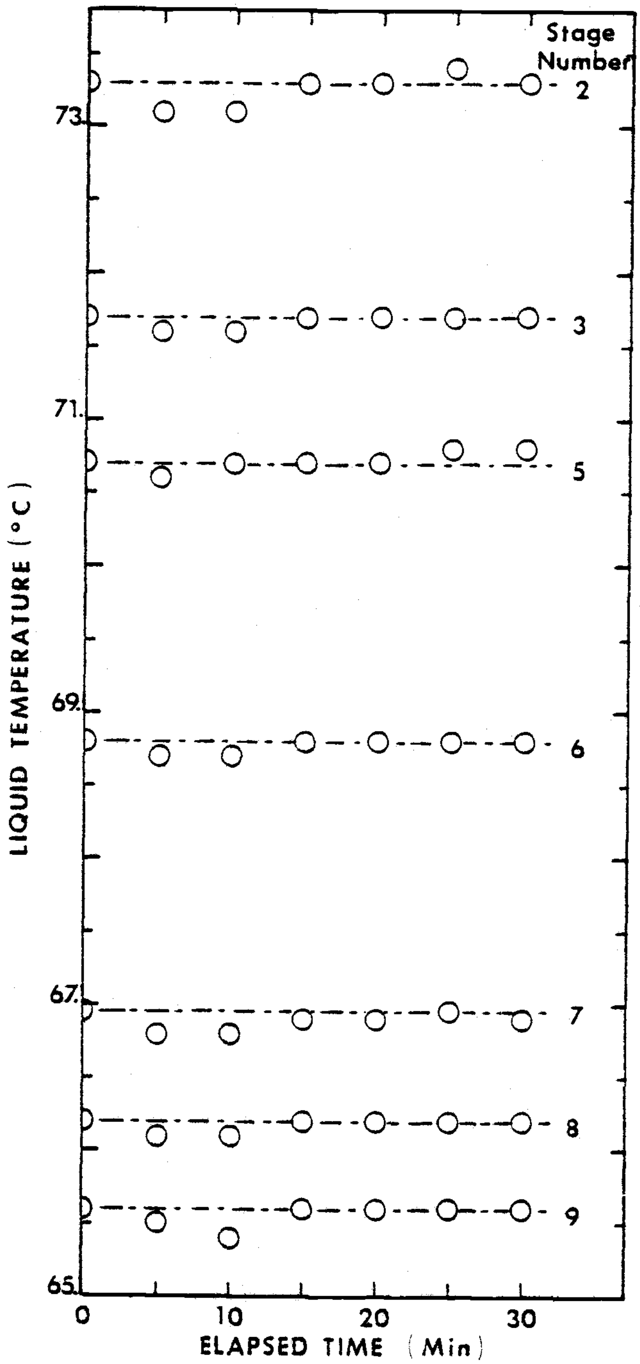



Figure 21. State Temperature Responses to a Pulse Disturbance in L".

intermediate reboiler liquid recycle flowrate. The rotameters used for all the liquid streams are inconsistent at the low flowrates required in these experiments. In addition, the air supply to the liquid pumps fluctuates. The result is further flow variations. An effort was made to minimize variations by constantly monitoring the liquid flowrates; however, in spite of this effort variations did occur.

In addition to these point disturbances, all of the dynamic responses return slowly to the initial steady-state levels. Furthermore, in some cases the stage temperatures did not return to the initial values during the duration of the experiment. The reasons for this behavior are probably as follows. First, the reboiler holdup for the experimental equipment is very large in comparison to the stage holdups. Thus the reboiler responds relatively slowly to the disturbances and, in effect, controls the ultimate response of the individual stages. Second, the effect of certain disturbances is to increase the reboiler holdup and composition, thus changing the column operating conditions. The disturbances in the feed flowrate and the external liquid reflux rate are the worst in this respect. Both of these disturbances tend to increase the reboiler holdup which, in turn, causes changes in the reboiler composition. The result of all this and the fact that the distillate and bottoms products are mixed to provide feed is that the liquid feed composition changes.

To reduce the impact of these disturbances two things were done. The liquid level in the feed pot was increased so that the feed composition could be maintained throughout an experiment. Also, the pulse duration was limited to ten minutes. These actions reduce the effect of the disturbances on the steady-state conditions. However because of the design of the feed system, some drift from steady-state will always exist. For this reason, no comparisons can reliably be made as to the relative quickness of the column responses to steady state.

# VI. DISCUSSION AND ANALYSIS OF THE RESULTS

The results presented in the previous chapter indicate that the intermediate heat exchangers moderate the responses of the heat integrated column to system disturbances. Given the inaccuracies and problems involved in the operation of the experimental column, it may be that the observed behavior is due only to the manner in which the two columns were operated. For this reason at least, the experimental response results should be compared to responses expected based on a model of the distillation system. Thus, the discussion presented in this chapter is based on the theoretical model.

The theoretical model is used here mainly to provide information in three areas. The model is first used as a tool of verification; that is, to see if the experimental response is reasonable. Second, comparison of the data generated by experiment and by simulation is used to indicate areas where more work should be done to improve the quality of the data. Finally, the model is used to critique the assumptions contained in the model itself.

In addition to the discussion based on the theoretical model, other areas are considered in this chapter. These include discussion of the errors likely present in the experimental results, a summary of the effects of intermediate heat exchangers indicated by the results, and a summary of questions which should be answered by further work on heat integrated distillation.

## Dynamic Response Model

The model presented here is based on theoretical equations given in Chapter III and is adapted as necessary to fit the experimental situation. The information presented in this section details the modifications necessary so that the model represents the experimental setup. Naturally, the solution of the set of equations making up the model is a numerical one best carried out by computer calculation. Thus, the model was applied via computer program.

## Preliminaries

Application of the model to the experimental equipment used in this work required one basic change. Since the experimental column is made of glass and is not insulated, there are significant heat losses from the column. Therefore, in modeling the experimental column the assumption of adiabatic operation cannot be made. This means that the liquid and vapor flows cannot be considered constant throughout the column. To account for the effect of heat losses then, the model must provide for changes in the liquid and vapor flows on each stage in the column.

In accounting for the heat losses, one would like the model to fit the experimental situation as closely as possible. Little information is available about heat loss from the experimental column, and the experimental efforts to determine the amount and incidence of the losses were not successful. Therefore, the heat losses were apportioned in the model based on external surface area and the amount of the loss was assumed based on the limits of the heat balance. For the experimental column, two-thirds of the system's external surface area is taken up by the bottoms boiler, bottoms pot and connections; the actual column takes up the remaining one-third. So then, in the model, two-thirds of the total heat loss is applied before the first stage and the remaining third is divided equally between the stages.

A special subroutine was used to calculate the stage liquid and vapor flows given the heat loss and the location of the intermediate heat exchangers. The intermediate boilup and vaporization provide additional discontinuities in the vapor and liquid flows. These were handled in the same manner as the heat losses, since the intermediate heat exchangers were designed to act only as sources of increased internal liquid or vapor flow.

The model was adapted for use with methanol-water system in two ways. First, the vapor-liquid equilibrium relationship was provided numerically in the computer program as a polynomial approximation. The methanol-water system can be accurately represented by an eighth order polynomial. Second, to make the simulation output compatible with the experimental results, the stage liquid concentrations as determined from solution of the model were translated to the appropriate saturated temperatures. The saturated temperatures were then the simulation output. The saturated temperature-composition curve is accurately represented by a sixth-order polynomial.

## Experimental Parameters

In order to make the results of the model as realistic as possible, measured experimental parameters were used as input to the model. The initial steady-state operating conditions indicated in Table 1 (see Chapter V) for the two experimental runs studied here, were used as input to the model. These parameters include the feed flowrate, the feed composition, the overhead product flowrate, the external liquid reflux rate, and the boilup and condensation rates. In addition, the locations of feed, intermediate boilup and intermediate condensation were incorporated into the model. The thermal state of the feed, along with the amount of the heat losses, are both conditions required by the model. Neither of these were measured experimentally and, thus, were assumed.

For the dynamic part of the model, the liquid holdups for the bottom reboiler and the individual stages are necessary information. These holdups were calculated based on the tray design, the known liquid concentrations, and the observed liquid levels at each location. The entries in Table 2 indicate the holdup values used in the model. The heat losses and subcooled feed encountered in this experiment are the source of the increased tray holdups indicated for the lower sections of the column.

Table 2. Calculated Stage Liquid Holdups

| Stage Number | Holdup (moles) |
|---|---|
| Reboiler | 1900. |
| 1 | 7.9 |
| 2 | 7.3 |
| 3 | 6.8 |
| 4 | 6.5 |
| 5 | 5.5 |
| 6 | 5.6 |
| 7 | 5.2 |
| 8 | 4.6 |
| 9 | 4.5 |
| 10 | 3.3 |

The values listed in Table 2 represent increases above the values based solely on tray design and liquid composition as follows: 40 percent for stages below the feed, 20 percent for the feed stage, 30 percent for the two stages above the feed, and 20 percent for the remaining stages except for stage ten. The value for stage ten represents a 10 percent decrease.

## Fitting Steady State

In addition to the modifications mentioned earlier, others are required so that the steady-state conditions(stage liquid and vapor concentrations) predicted by the simulation match the experimental results. The simulation results are useless unless the simulated dynamic responses start from the same point that the experimental responses do. The goal of the modifications presented here is to match as closely as possible the steady-state experimental concentrations with those concentrations predicted by the model.

Initially the model was set up to match the experimental setup with 11 differential equations for each of the stages and one for the bottom reboiler. However, this formulation could not be manipulated to match the experimental steady-state conditions. Two modifications proved to be necessary. First, the stage efficiency of the bottom reboiler (as measured from the first stage) was found to be greater than 100 percent. This impossible condition is due to the presence of substantial vapor-liquid contacting prior to the first stage; the contacting is similar to that observed in a wetted wall column. To account for this contacting in the model, an additional pseudo stage was added between the reboiler and the first stage. The pseudo stage was incorporated using a small holdup to reduce its effect on the actual stage dynamic responses.

The second necessary modification involved the top tray (the eleventh tray). The top tray is not fitted with a thermocouple, so its response cannot be measured experimentally. However, it was initially included in the model to account for the observed separation. But after noting the arrangement of this top tray in the column, it was removed from the model. The tray is positioned in such a way that the downcomers for the tray are located immediately beneath the liquid reflux entrance. This means that the top tray provided no real vapor-liquid contacting in the experimental runs and acted merely as a distributor.

The last things required by the model to fit the steady-state conditions are the individual stage efficiencies. As indicated in Chapter III, these stage efficiencies are based on the vapor compositions, therefore the stage vapor concentrations must be known for the experiments modeled. Since experimental measurements of vapor compositions and stage efficiencies proved to be unreliable, the stage vapor concentrations and stage efficiencies were determined by calculation.

The stage vapor compositions and, thus, the stage efficiencies were determined by stepping off the individual stages on the McCabe-Thiele diagram using both the experimental stage liquid concentrations and the experimental operating conditions. Since the heat losses from the column and the feed liquid thermal condition are not available for the experimental runs, the calculation procedure is an iterative one. First, either the heat losses or thermal state of the feed must be assumed to obtain the operating lines, then the calculated stage efficiencies must be used in the model to see if it matches the experimental steady-state conditions. If there is no match, the heat losses are again assumed. The final operating diagram plots may be found in Appendix D. The resultant stage efficiencies, along with the assumed heat losses and thermal state of the feed for the two experimental runs considered here, are presented in Table 3.

Table 3. Stage Efficiencies

| Stage Number | Efficiency (E) Conventional Column Experiment | Efficiency (E) Modified Column Experiment |
|---|---|---|
| Reboiler | 1.0 | 1.0 |
| Pseudo | 0.57 | 0.57 |
| 1 | 0.56 | 0.50 |
| 2 | 0.72 | 0.71 |
| 3 | 0.34 | 0.30 |
| 4 | 0.14 | 0.13 |
| 5 | 1.0 | 0.93 |
| 6 | 1.0 | 1.0 |
| 7 | 0.77 | 0.71 |
| 8 | 1.0 | 0.93 |
| 9 | 1.0 | 0.82 |
| 10 | 0.90 | 0.50 |
| q | 1.4 | 1.4 |
| Heat losses (mole/min) | 1.90 | 1.85 |

Two things should be noted from the information presented in Table 3. First, the q value (indicating the thermal state of the feed) is relatively high, indicating a very subcooled liquid. For the methanol-water system a q value of this magnitude is impossible. However, it was used in the model since it likely indicates that the heat losses were apportioned incorrectly. Secondly, the stage efficiencies in the lower half of the column are low. The low-stage efficiencies for the lower stages are probably a result of the high heat losses and possible over estimation of the boilups from the reboilers. The very low value for stage four indicates that the feed indeed is subcooled.

The steady-state temperatures indicated by the simulation are compared to the experimental steady-state values for the two experimental runs in Figures 22 and 23. From the comparison shown in these two figures, it is clear that the experimental steady-state conditions are closely matched by the model predictions.

## Theoretical Response Results

Having established the steady-state conditions, the simulated dynamic response can be generated. The simulation was performed for two experimental runs; those runs with the conventional and modified setups for the pulse disturbance in the external liquid reflux rate (LBB). These simulations were considered sufficient for the purposes of this discussion. The disturbance used in generating the simulated dynamic response is exactly the same as that used in generating the experimental dynamic responses. The disturbances are 10-minute, 20 percent positive pulses and are applied in these cases on the external liquid reflux rate.

Partial results for the simulated dynamic responses are presented along with the appropriate experimental response results in Figures 24 and 25. In Figure 24, the experimental simulated dynamic response results are presented for the conventional column subjected

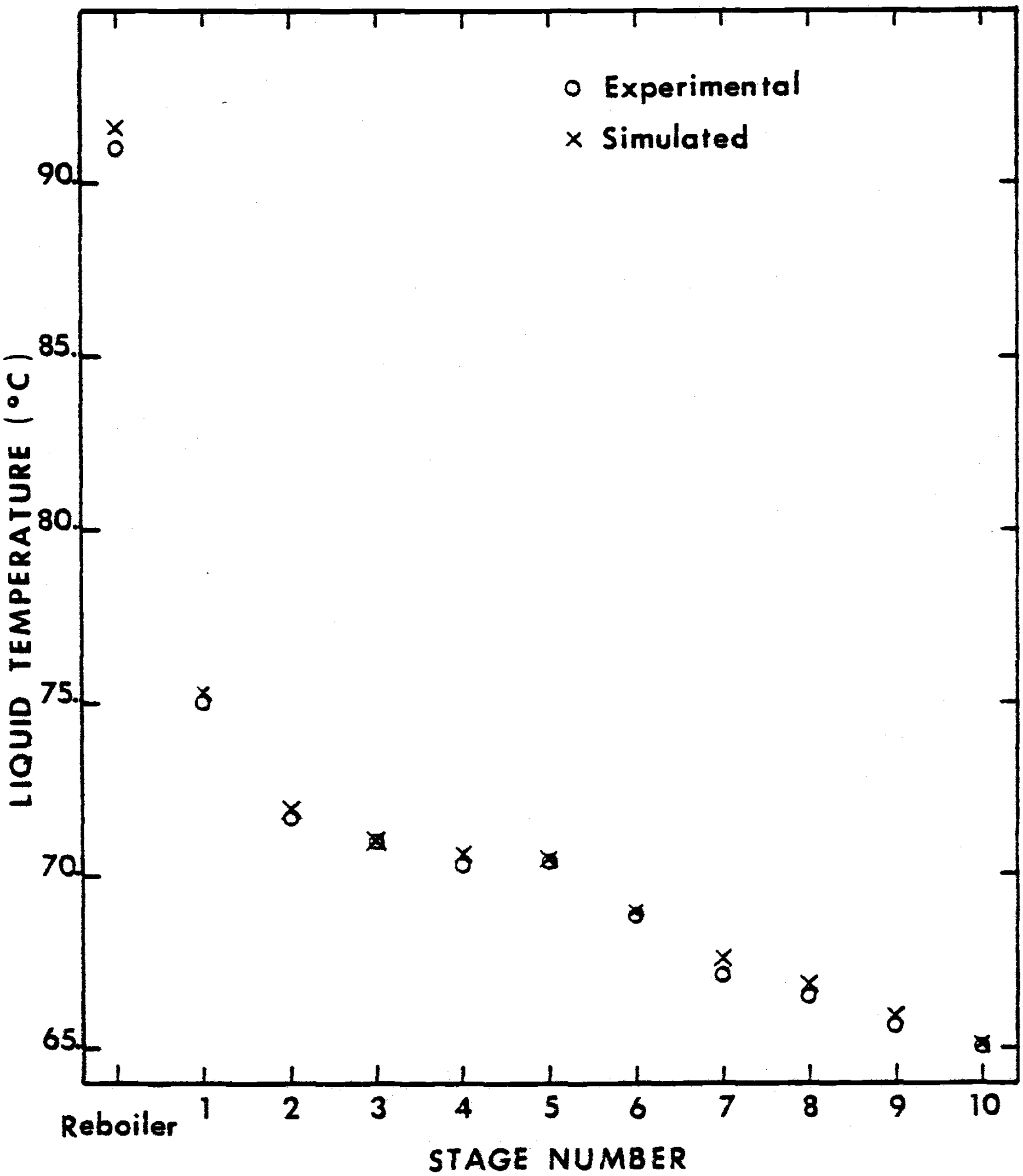



Figure 22. Steady State Stage Temperatures for the Experiment with the Conventional Column.

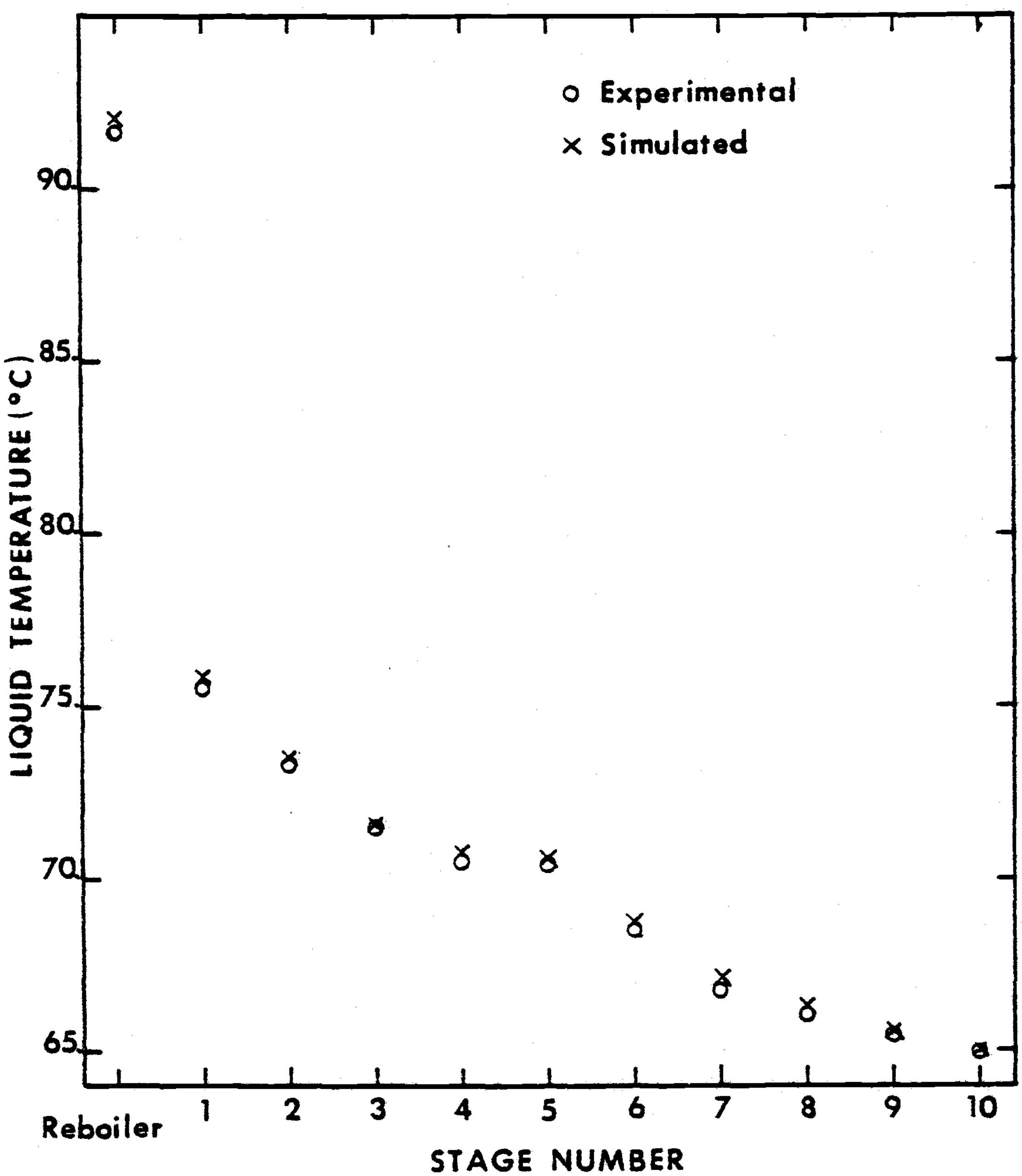



Figure 23. Steady State Stage Temperatures for the Experiment with the Modified Column.

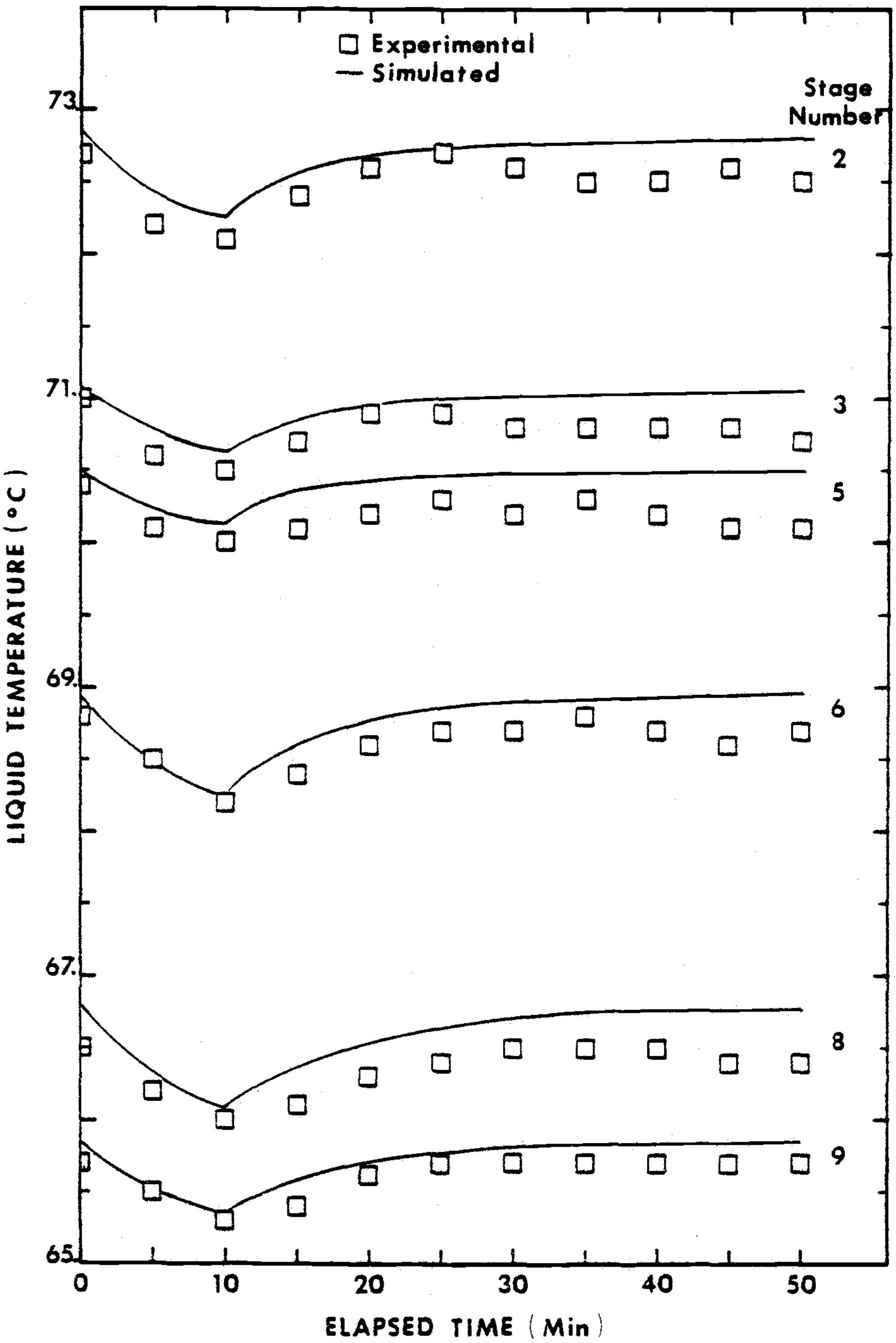



Figure 24. Conventional Column Response to a Pulse Disturbance in the Liquid Reflux Rate (LBB).

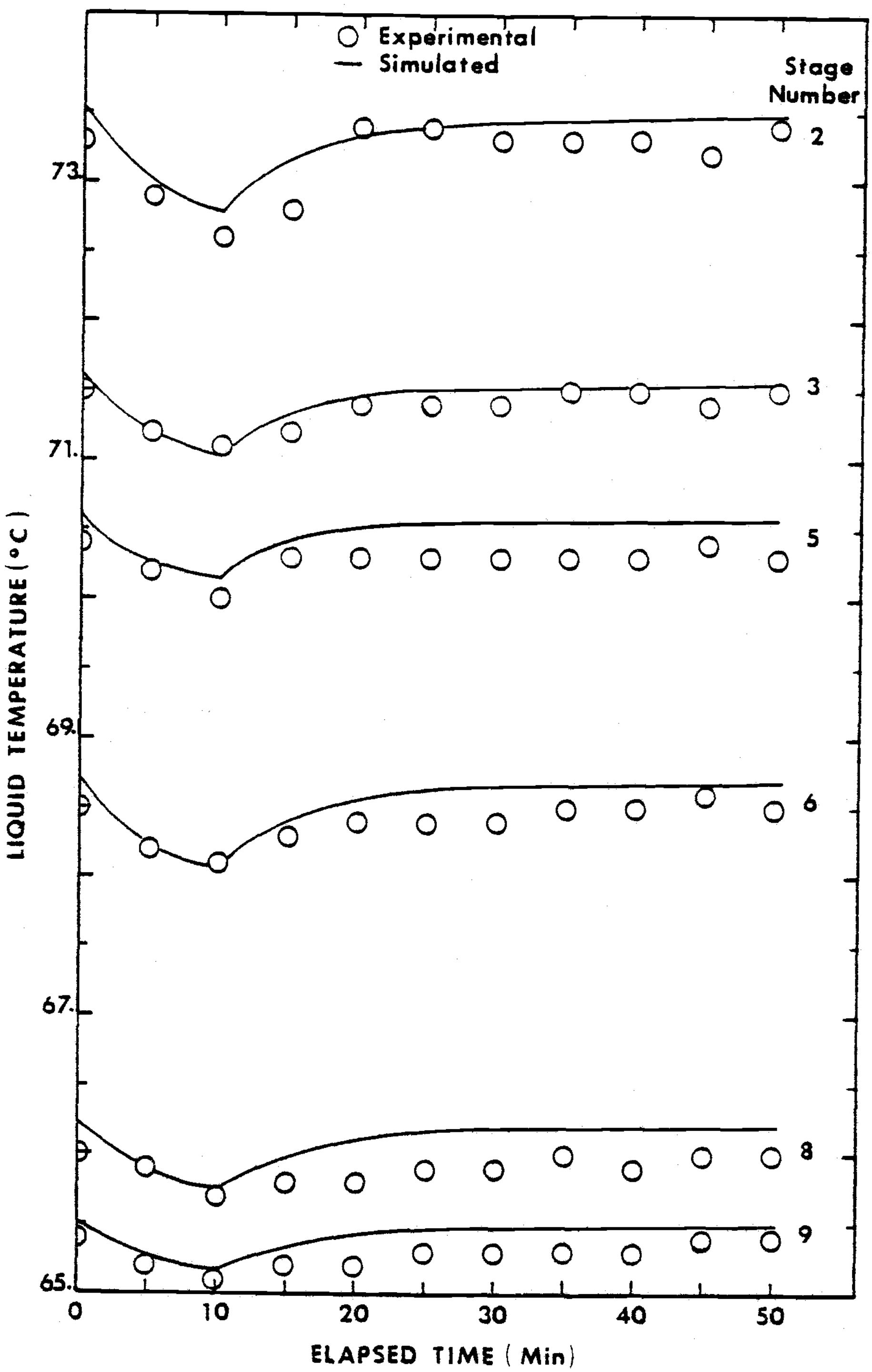



Figure 25. Modified Column Response to a Pulse Disturbance in the Liquid Reflux Rate (LBB).

to a disturbance in LBB. The results in Figure 25 are the experimental and simulated responses for the modified column. The results presented in both figures are for selected stages; complete listings of the response results along with a listing of the computer program are available in the Appendix E.

It is evident from consideration of Figures 24 and 25 that the dynamic responses generated by the computer simulation are very representative of the experimental responses. If the experimental and theoretical results are compared while taking into account both the experimental error and the slightly different starting points of the two results, it becomes evident that the two match well. In addition, it should be noted that the simulated response results reinforce the observations made earlier about the experimental results. The modified column shows deviations in the lower stages similar to those shown by the same stages in the conventional column. In the upper stages, however, the modified column shows smaller deviations than the conventional column. Thus, the simulation substantiates the response shown by the experiment.

The effect of neglecting the liquid flow transients in the model is seen in the simulated response results. The responses shown in Figures 24 and 25 have definite discontinuities at the 10-minute mark (when the pulse ends). The discontinuities are certainly not representative of the physical situation; however, a comparison of the theoretical and experimental responses shows that the experimental results are not far off the predicted result if the discontinuity is ignored. This confirms that the liquid flow transients can indeed be neglected for this experimental setup without compromising the value of the dynamic results.

## Experimental Errors

As with all experimental results, the experimental errors must be considered, particularly when a comparison is drawn between experimental and theoretical results. The errors involved in the experimental results reported in this thesis may be divided into two groups: these are the measurement errors and the setup errors. The first kind are those generated mainly by the data sampling procedures. The second kind of errors are those which arise due to the physical set-up of the experimental equipment.

The measurement errors margins are indicated in each presentation of the experimental results by the size of the symbol surrounding the actual data point. The margins used in reporting the results are $\pm$ 0.10°C in the temperature measurements and $\pm$ 0.5 minute in the time measurements. The errors in temperature sampling are due in part to the uncalibrated thermocouples and also to the variation in the temperature readings during the sampling period. The errors in sampling time are primarily the result of the manual method of data collection. Each of the 15 thermocouple channels was sampled manually at the sample time. Normally the channels could be sampled in one minute; this, however, means a $\pm$ 0.5 minute error in the sampling time. The sampling time may be reduced by automation of the data collection process (see Appendix F for details).

The error due to experimental setup are much more difficult to quantify; their sources, however, should be noted for completeness. The foremost factor affecting the quality of the experimental data is probably the column heat loss. Because of the heat loss, the liquid tray temperatures may not be uniform across the tray. In fact, measurements taken with a thermocouple inserted through the liquid sample port on the seventh stage indicate that the liquid near the wall is at least 3°C lower in temperature than the measured liquid temperature. In addition, the heat losses may also cover effects which could have been otherwise observed. Since the heat loss has exactly the opposite heat effect as the intermediate reboiler, some of the reboiler's moderating influence may be covered by the heat losses.

The other major equipment errors have been mentioned previously. These errors are those due to irregularities in valve and rotameter operation, particularly at the lower flowrates. These kinds of errors tend to move the column operating conditions from their initial steady-state values. If, in the future, data are taken to indicate the relative quickness of response, these errors will have to be reduced.

## Summary

Two summaries should be made to conclude this discussion. These are summaries of the observations made thus far and of the multitude of further questions generated by this work.

### Observations

The experimental results presented in Chapter V, along with the theoretical results presented in this chapter, make it clear that there is a difference between the dynamic responses of the conventional and heat integrated distillation systems. All of the comparisons between the experimental results for both types of columns indicate that the heat integrated column is more moderate in its internal dynamic response than is the conventional distillation column. The explanation proposed in this thesis is that the intermediate heat exchangers provide the moderating influence in the heat integrated column. This moderating influence is due to some sort of surging effect the intermediate reboiler and condenser have on the internal liquid and vapor flows.

The reboiler seems to provide the most moderating effect for disturbances related to increases in liquid flowrates down the column, while the condenser seems to have the greatest effect for disturbances related to increases in vapor flowrates up the column.

It should be mentioned that, even though a disturbance initially affects only one flowrate, removal of the disturbance usually means that the other is affected too (such was the case in the disturbance on the boilup rate, VPP). Thus, both intermediate heat exchangers may show their influence on the dynamic response, even though the initial disturbance cannot be moderated by one of the heat exchangers.

The experimental results seem to indicate that the intermediate heat exchanger closest to the disturbance will have the greatest influence on the dynamic response, assuming, of course, that the intermediate heat exchanger can influence the propagated disturbance. Examples of this kind of behavior can be found in the experimental runs with disturbances in F and LBB.

Turning now to the theoretical response results, three comments are made in summary. First, the substantial effort involved in fitting the simulation results to the experimental results was directed primarily toward indicating areas where the experiment can be improved. As can be seen from the discussion of this effort, the major deficiencies in the experimental results are related in some way to the substantial heat losses from the experimental column. It is clear that future work on the experimental equipment should be directed toward reducing the heat losses so that they do not shadow the results.

The other two comments are related more to the theoretical model than to the experimental setup. It is evident from the comparisons given here that the model can be used to predict the dynamic response for this particular setup. It is also evident that the liquid flow transients are not a problem in the experimental column and their absence in the model does not adversely impact the quality of the dynamic response results. Furthermore, the fact that the liquid flow transients can be neglected indicates that the intermediate heat exchangers cannot be acting to counteract the flow surges but rather the composition surges. Since the intermediate heat exchangers were designed to operate without holdup (and the model predictions confirm the design), it is not clear then how the heat exchangers have their effect.

Further Questions

In the course of carrying out the experimental work, a number of other questions were generated. These questions could not be addressed in this work, but they are important both as suggestions for future work and as insight into the significance of the results presented in this thesis. The individual questions are presented here in order of application to the various sections of this thesis.

The first question concerns the effect of solution nonidealities on the optimization results. What are the optimum heat loads and locations for intermediate heat exchangers in the separation of a nonideal or azeotropic mixtures? These kinds of mixtures naturally will not have easy optimization solutions; they, however, may present real opportunities for application of heat integrated distillation.

The comparisons between the conventional and modified columns in this work were done on the basis of similar column design and similar separations. The other basis for comparison is that of similar total heat duties. The question is then, what would be the result of a comparison done on a difference basis? Would the dynamic responses still be different? The indication given by these results is that the intermediate heat exchangers would still moderate disturbances, but the differences in the dynamic responses might be different than those presented here.

The feed liquid used in the experimental runs was always subcooled. This led to the exclusive influence of the intermediate reboiler on the dynamic response of some experiments. A similar situation might be expected for the condenser if the feed were partially vaporized. The results presented here, however, give no information about the effect that the thermal state of the feed has on the observed response. It may be that with a saturated feed, the effect of the intermediate heat exchangers is lessened.

The applicability of these results is greatly influenced by the heat losses present in the experimental setup. No commercial distillation column could be operated with heat losses of the magnitude encountered here. However, since both the conventional and modified columns were operated with the same heat losses, it is unlikely that the observations made here are incorrect. The heat losses do influence the dynamic response and the extent of this influence is unknown.

The ultimate questions that should be asked concerns the external control of a heat integrated column. Throughout this study, we have been concerned with the internal response of the heat integrated column. The internal response is important since it dictates the external response; however, it is the external response which is of primary concern for plant operation. In this area there are the usual questions of control strategy. Another question might be how the locations and duties of the intermediate heat exchangers influence the column's response. It may be that the optimum steady-state locations and duties are not the best ones as far as the dynamic operation of the column is concerned.

The questions presented here are by no means all that could be generated; they do, however, provide direction for future work. Further information pertaining to these concerns may be found in Appendix F.

# VII. CONCLUSIONS

In closing, three general conclusions can be made based on the information presented in this thesis. These are presented in order of their application to the text.

1. The experimental distillation equipment developed for this study can be used to generate useful dynamic response data. The distillation apparatus can be operated both with and without the intermediate heat exchangers for study of both conventional and heat integrated distillation.

2. The intermediate heat exchangers have a substantial effect on the dynamic response of distillation columns. In the cases studied here, the dynamic response of the heat integrated column (with one set of intermediate heat exchangers) is more moderate than the dynamic response of the conventional column. The observed moderation is due to the effects brought on by the intermediate heat exchangers. It is postulated that the intermediate heat exchangers have an effect similar to that of a concentration surge tank.

3. The heat losses experienced with the experimental column are relatively large. Because these losses limit the application of the results presented here, they should be reduced before further work is done using the distillation column.

# VIII. BIBLIOGRAPHY


Abrams, H., 1978. "Energy Reduction in Distillation," in Alternatives to Distillation, Inst. of Chem. Eng. Symposium Series No. 54.

Bannon, R. P. and S. Marple, 1978. "Heat Recovery in Hydrocarbon Distillation," Chem. Eng. Progr., 74(7), 41.

Benedict, M., 1947. "Multistage Separation Processes," Chem. Eng. Progr., 43(2), 41.

Chu, Ju-chin, 1950. Distillation Equilibrium Data, Reinhold Publishing Corp., New York.

Corning Glass Works, 1978. Glassplant Components Catalog, Corning, New York.

Crocker, W. A., 1976. M.S. project, Chemical Engineering Dept., Oregon State University, Corvallis, Oregon.

Duckham, H. and J. Fleming, 1976. "Better Plant Design Saves Energy," Hydrocarbon Processing, 55(7), 78.

Fitzmorris, R. E. and R. S. H. Mah, 1980. "Improving Distillation Column Design Using Thermodynamic Availability Analysis, AIChE J., 26(2), 265.

Freshwater, D. C., 1951. "Thermal Economy in Distillation," Tran. Inst. Chem. Eng., 29, 149.

________, 1961. "The Heat Pump in Multi-Component Distillation," Brit. Chem. Eng., 6, 388.

Gould, L. A., 1969. Chemical Process Control: Theory and Application, Addison and Wesley Publishing Co., Reading, Mass.

Kayihan, F., 1977. "Optimum Distribution of Heat Load in Distillation Columns Using Intermediate Condensers and Reboilers," presented 70th Annual AIChE meeting, New York.

King, C. J., 1971. Separation Processes, McGraw-Hill, Inc. New York.

Mah, R. S. H., J. J. Nicholas and R. B. Wodnik, 1977. "Distillation with Secondary Reflux and Vaporization: A Comparative Evaluation," AIChE J., 23(5), 651.

Mix, T. J., J. S. Dweck, M. Weinberg and R. C. Armstrong, 1978. "Energy Conservation in Distillation," Chem. Eng. Prog., 74(4), 49.

Null, H. R., 1976. "Heat Pumps in Distillation," Chem. Eng. Prog., 74(7), 59.

Patil, S., 1981. "Dynamics and Control of a Heat Integrated Distillation Column with Side Heat Exchangers," Chemical Engineering Department, Oregon State University, Corvallis, Oregon.

Pratt, H. R. C., 1967. Countercurrent Separation Processes, Elsevier Publishing Co., New York.

Prengle, H. W., J. R. Crump, C. S. Fang, M. Frupa, E. J. Henley and T. Wooley, 1974. "Potential for Energy Conservation in Industrial Operations," S/D-10 Governor's Energy Advisory Council, The State of Texas.

Robinson, C. S. and E. R. Gilliland, 1950. Elements of Fractional Distillation, 4th Ed., McGraw-Hill, Inc., New York.

Stephanson, R. and T. F. Anderson, 1980. "Energy Conservation in Distillation," Chem. Eng. Progr., 76(8), 68.

Tedder, D. W. and D. F. Rudd, 1978. "Parametric Studies in Industrial Distillation" (3 parts), AIChE J., 24, 303.

Timers, A. C., 1969. "International Symposium on Distillation," Inst. of Chem. Eng., Brighton, England.

Treybal, R. E., 1968. Mass Transfer Operations, 2nd Ed., McGraw-Hill, Inc., New York.

Umeda, T., K. Niida and K. Shiroko, 1979. "A Thermodynamic Approach to Heat Integration in Distillation Systems," AIChE J., 25(3), 423.

APPENDICES

# APPENDIX A
# EQUIPMENT ILLUSTRATIONS

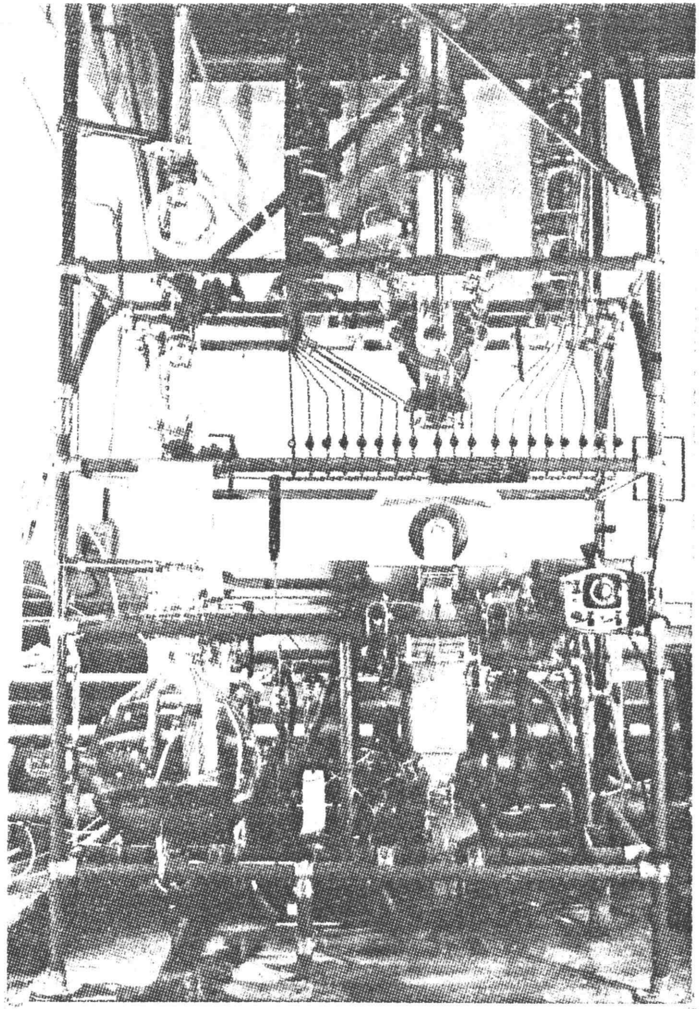


Figure A1. Front View of the Distillation Equipment.

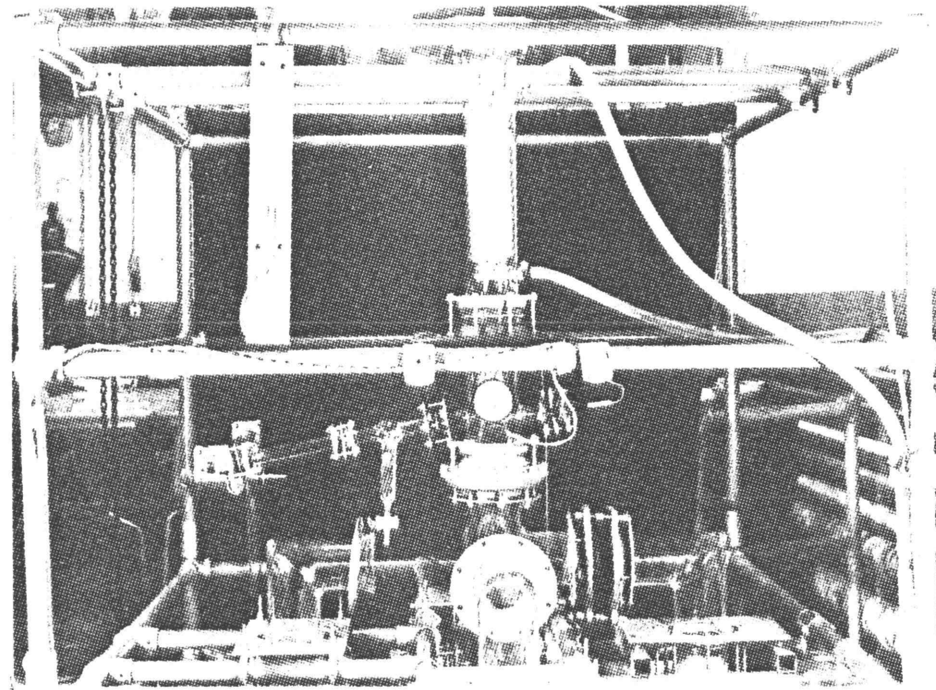


Figure A2. View of the Overhead Condenser System.

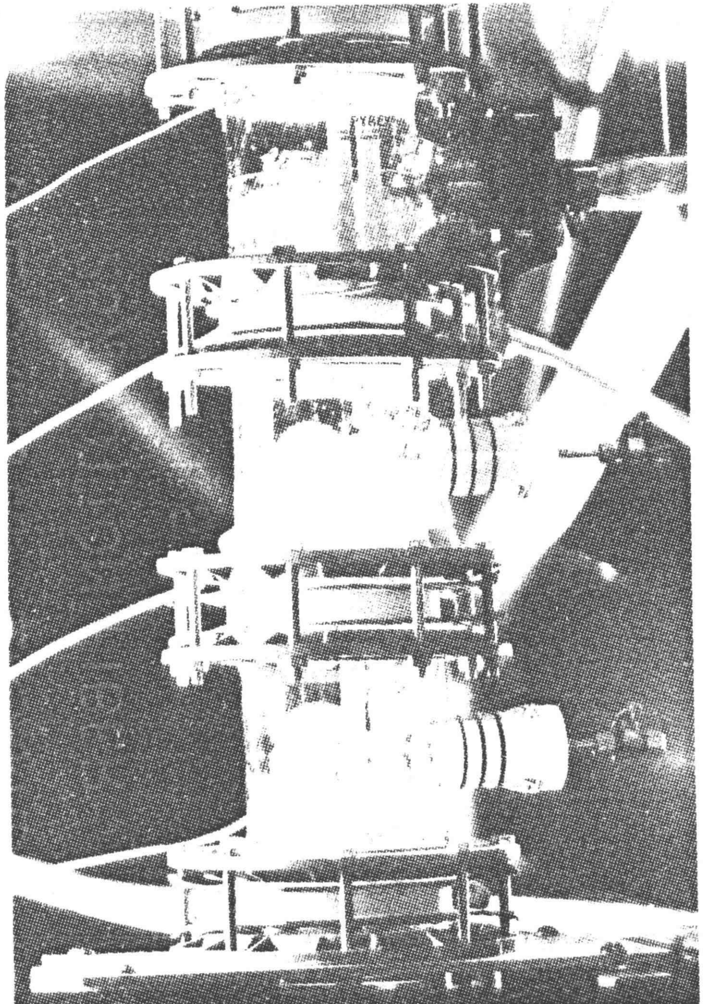


Figure A3. Close up of the Experimental Column (South Side).

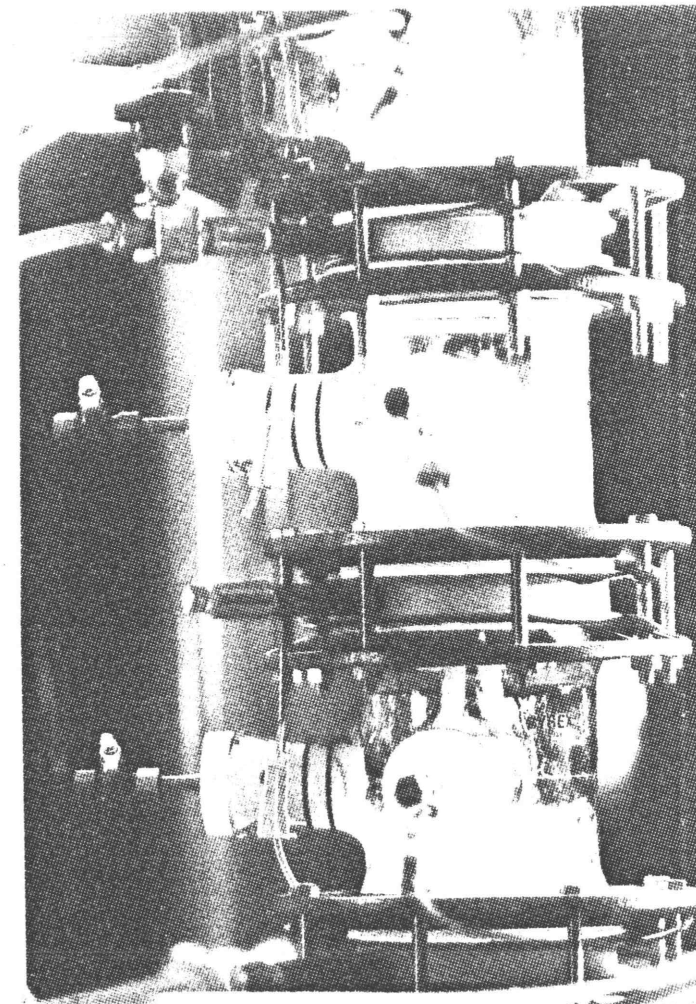


Figure A4. Close up of the Experimental Column (North Side).

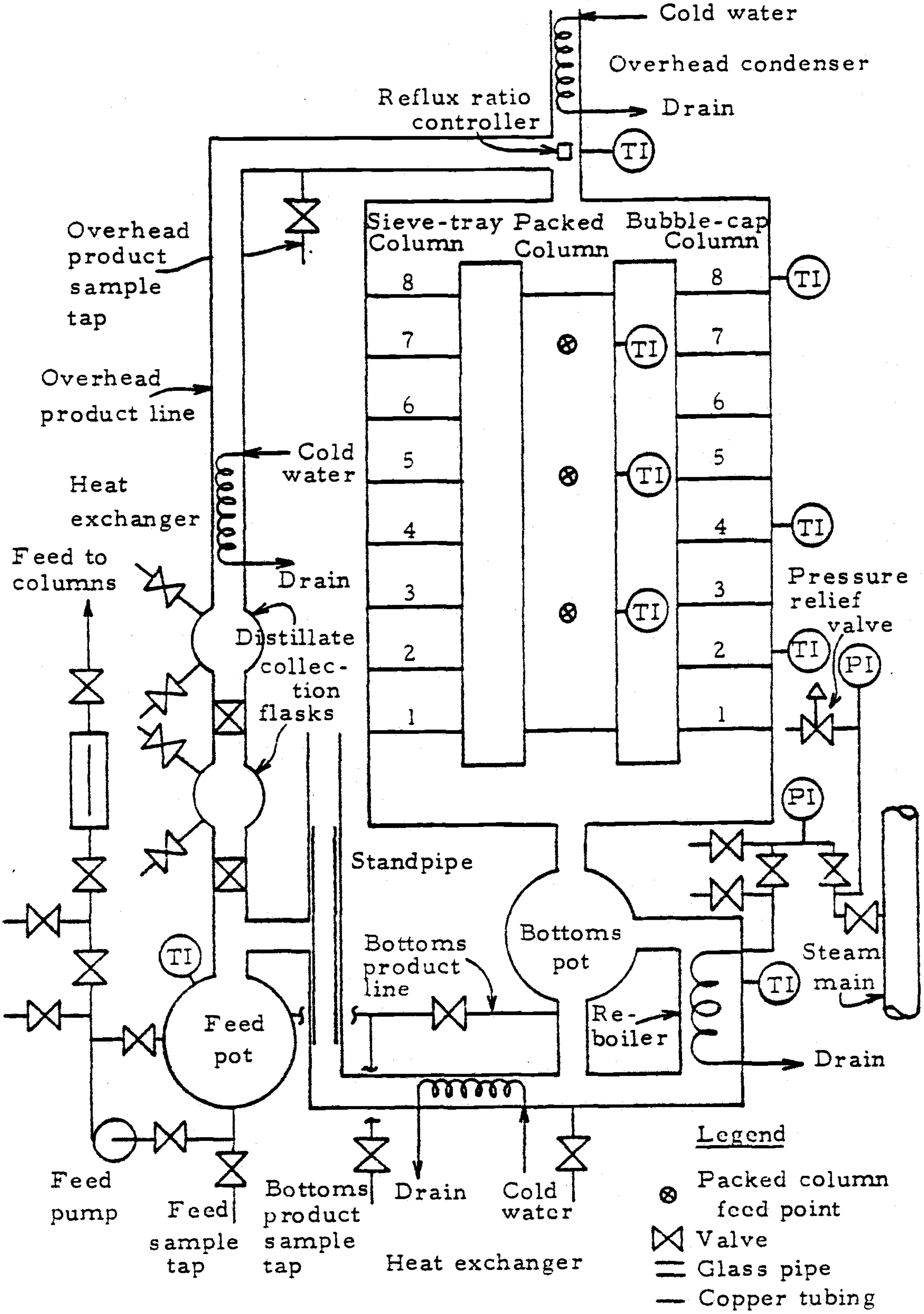



Figure A5. Schematic Diagram of the Distillation Equipment. From Crocker (1976)

# APPENDIX B

# CALIBRATION CURVES

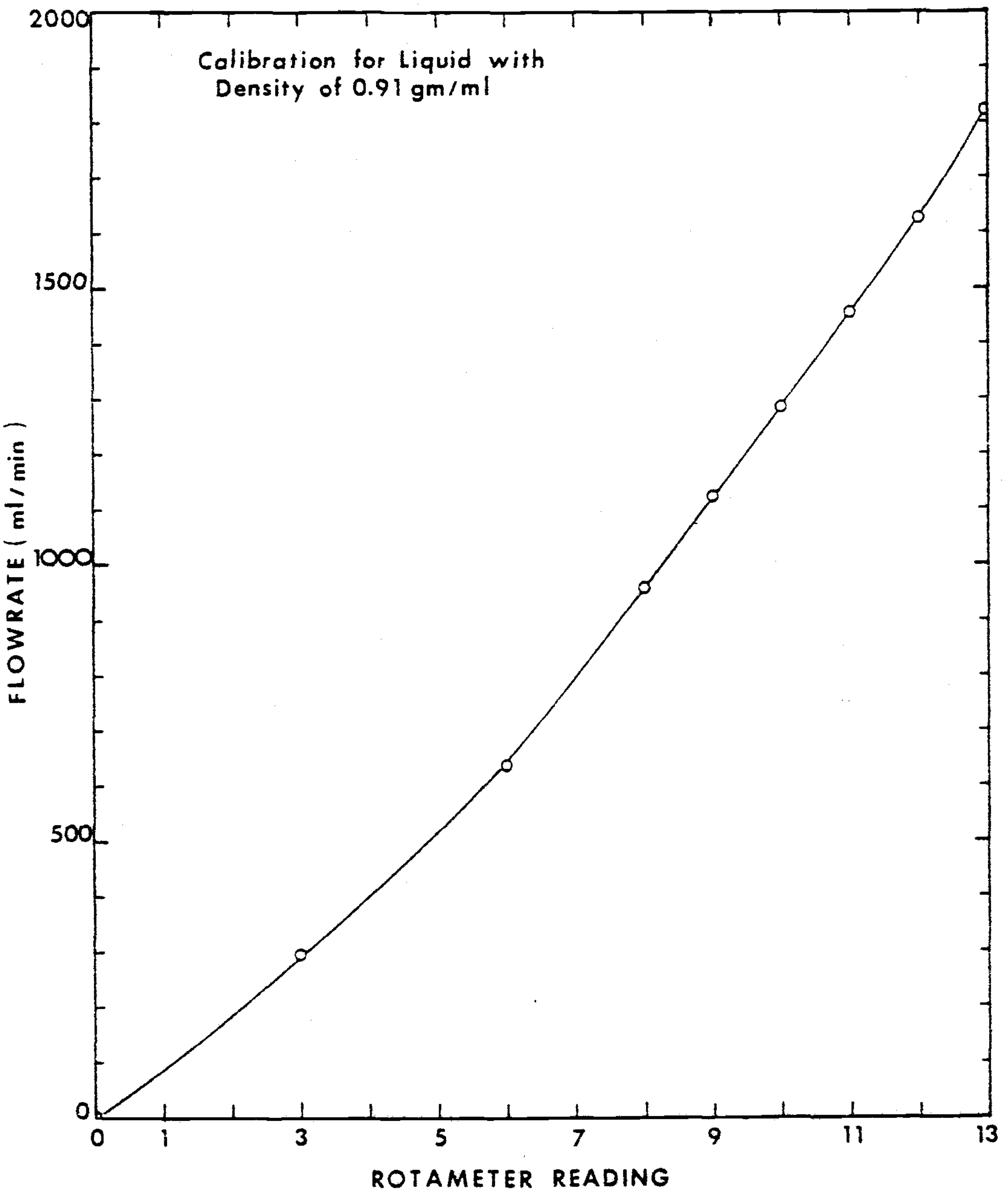



Figure B1. Rotameter Calibration Curve (Brooks Sho-Rate Rotameter).

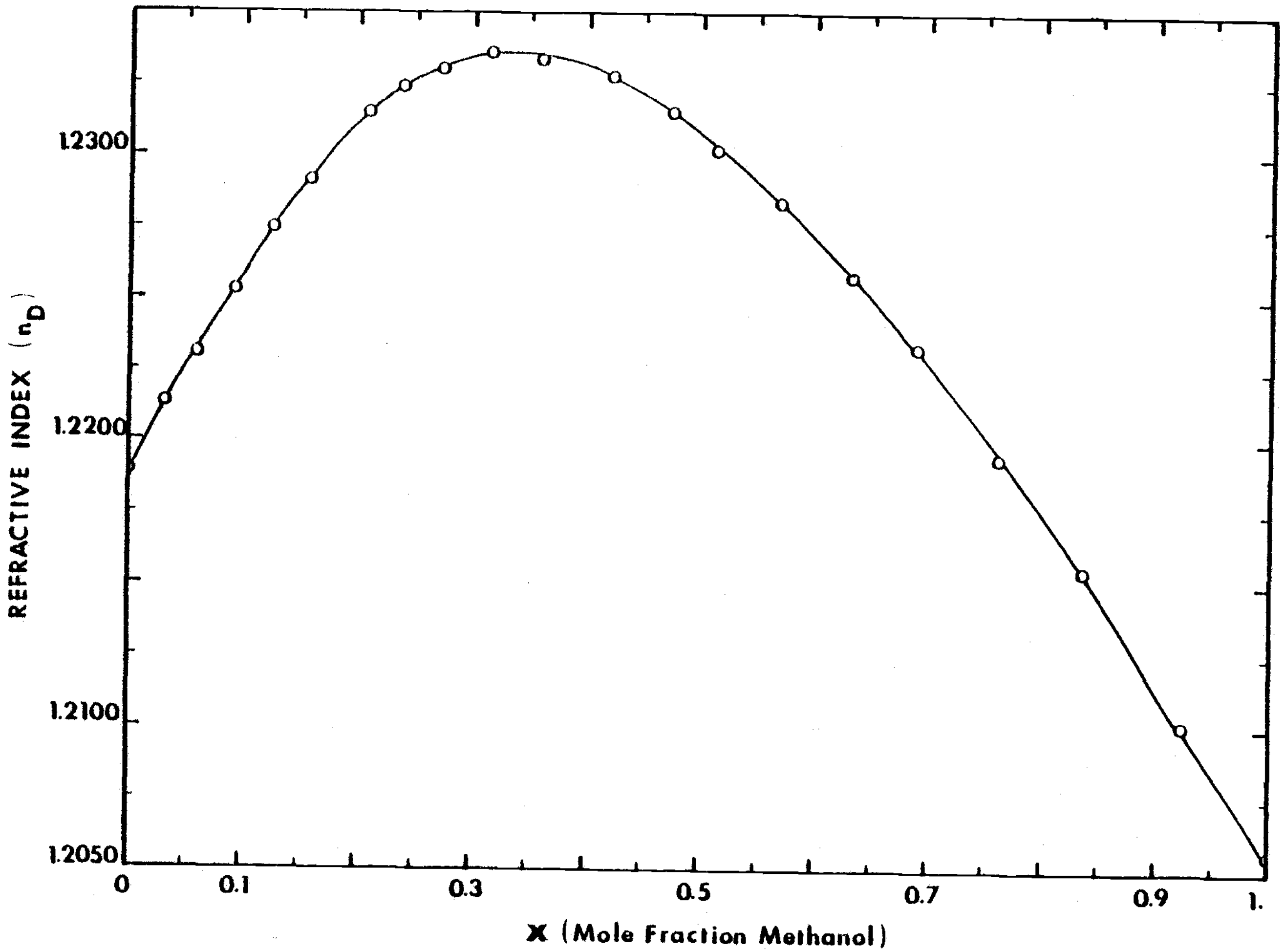



Figure B2. Index of Refraction for Aqueous Methanol Solutions at 30°C (Bausch and Lomb Refractometer).

# APPENDIX C

# OPERATING PROCEDURE

## Conventional Column

### Start Up

1. Start cooling water flow through the overhead condenser and the heat exchanger in the stand pipe line.
2. Set water flowrate through the aspirator such that the manometer indicates a pressure difference between the top column pressure and atmosphere pressure equal to -2 cm of water head.
3. With the reflux valve in the reflux position, open the main steam valve. Once the line pressure reaches 15 psig, the valves to the main and additional bottoms reboiler may be opened and the desired reboiler steam pressure chosen.
4. Switch on the digital thermometers or multipoint recorder so that stage temperatures may be monitored.
5. As the column heats up, liquid should begin accumulating on stages from the bottom up. In the case of operation with a volatile component (such as methanol), the stages accumulate liquid beginning at the top. To avoid flooding and to allow each stage to operate correctly the reboilers steam supply may have to be shut off for short periods once or twice.
6. Once all the stages are operating correctly, allow the column to operate in total reflux for approximately one hour (this insures sufficient mixing of the liquid in the reboiler).
7. Make sure that sufficient liquid fills the bottoms pot so that the bottom reboilers circulate liquid into the bottoms pot.

Operation with Feed and Off-take

1. With the column operating at total reflux, switch on the reflux ratio controller and set the stops to give the desired reflux ratio.

2. Now that the reboiler steam pressures (and thus the boilup rate) and the reflux ratio have been set, the distillate flowrate is essentially fixed. The feed and bottoms product flowrates must now be adjusted to give a material balance.

3. Measure the initial distillate flowrate and using approximate concentrations for the distillate bottoms and feed steams calculate and set the bottoms and feed flowrates. As the column operation comes to steady state the compositions of the product and feed streams must be measured along with the distillate flowrate and the feed and bottoms flowrate must again be adjusted to the appropriate levels.

4. This material balance procedure is continued until the stage liquid temperatures remain steady for 10 minutes. Due to the large reboiler holdup, the process of reaching steady state may take one hour or more.

5. Once steady state is obtained, the system may be disturbed to generate dynamic responses or liquid and vapor samples may be taken to determine stage efficiencies or liquid saturation.

Shutdown

1. Close the feed value and the bottoms product valve.

2. Shut off the feed pump and reflux ratio controller.

3. Shut the steam valves beginning with the header valve. Shut the valves for each reboiler once the pressure falls to zero.

4. After approximately 10 minutes has elapsed, shut the valves on

the water lines to the condenser and aspirator.

5. Switch off the digital thermometers and the multipoint recorder.

## Modified Column

### Startup

1. Follow the same startup procedure given for the conventional column.
2. Once the individual stages are operating in a stable way, open the air supply valve for the air-driven Micro Pumps.
3. Open the valve on the inlet liquid line to the intermediate reboiler and adjust the flow to approximately 500 ml/min. Immediately open the valve on the liquid recycle line and the regulator valve. Adjust the regulator until the pump is started and is pumping approximately 500 ml/min of liquid back to the column.
4. By adjusting the pumping rate, maintain a constant liquid level in the intermediate reboiler sufficient to cover the heat transfer coils.
5. Operate until liquid in intermediate reboiler is similar in concentration to that on the stage.

### Operation with Feed and Off-take

1. Follow steps one through four given previously for the conventional column all the while maintaining the liquid circulation through the intermediate reboiler.
2. Open the valve for water supply to the jacket side of the intermediate "condenser." With the drain valve complete open, adjust

the inlet valve to obtain a constant liquid level completely covering the heat transfer coils.

3. Once the preliminary material balance is satisfied, calculate the intermediate heat exchanger duties.

4. Open the valves on the liquid lines to and from the intermediate "condenser," and, using the regulator, start the Micro Pump and adjust the liquid flow through the "condenser" to the appropriate level.

5. Open the steam valve to the intermediate reboiler and allow the liquid to heat up. Once intermediate reboiler begins operating (i.e., the liquid is boiling), reduce the recycle liquid flowrate to maintain the constant liquid level in the reboiler.

6. The addition of intermediate boilup and condensation changes the operation of the column. Thus, the material balance must again be established.

7. Monitor the stage temperatures as well as the intermediate heat exchanger temperatures to determine when steady state is reached.

Shutdown

1. Close the steam valve on the intermediate reboiler and quickly close first the liquid inlet valve (behind the rotameter), and then the regulator valve for the liquid recycle pump.

2. Shut down the intermediate "condenser" by first closing the regulator valve, thus stopping liquid flow and then closing the valve on the liquid outlet line (behind the rotameter). Also, stop the water flow through the condenser and shut off the air supply valve at the header.

3. Follow the shutdown procedure indicated for the conventional column.

## Dynamic Operation

1. Once the column is operating at steady state, record all appropriate operating conditions, including steam pressures, flowrates, and composition.
2. Disturb the system with a 10-minute, 20% positive pulse in one of the following system parameters: F, VPP, LBB or L".
3. Record the stage and reboiler liquid temperatures at five-minute intervals from the initiation of the pulse to the end of the experiment.
4. Continue to record data until steady state is approached.
5. Shut down the system.

# APPENDIX D

# EXPERIMENTAL DATA

Table D1. Stage Liquid Temperatures for the Conventional Column: Disturbance in F

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_C$ | $T_R$ |
| 0 | 75.0 | 72.4 | 70.6 | 69.8 | 69.8 | 68.4 | 66.9 | 66.3 | 65.6 | 65.0 | $65.1^{-}$ | 91.6 |
| 5 | 74.4 | 71.8 | 70.4 | 69.8 | 69.9 | 68.5 | 67.0 | 66.4 | 65.7 | 65.1 | 65.2 | 91.6 |
| 10 | 74.2 | 71.7 | 70.3 | 69.7 | 69.9 | 68.5 | 67.0 | 66.4 | 65.7 | 65.1 | 65.2 | 91.5 |
| 15 | 74.8 | 72.3 | 70.4 | 69.7 | 69.7 | 68.3 | 66.9 | 66.3 | 65.6 | 65.0 | 65.0 | 91.3 |
| 20 | 74.9 | 72.3 | $70.5^{-}$ | $69.7^{+}$ | 69.7 | $68.3^{+}$ | 66.9 | 66.3 | 65.6 | 65.0 | 65.1 | 91.3 |
| 25 | 74.9 | 72.4 | 70.5 | 69.7 | 69.7 | 68.3 | 66.9 | 66.3 | $65.6^{-}$ | 65.0 | 65.0 | 91.3 |
| 30 | 74.9 | 72.4 | 70.5 | $69.7^{-}$ | 69.7 | 68.3 | 66.9 | 66.3 | 65.6 | 65.1 | 65.1 | $91.4^{+}$ |
| 35 | 74.9 | 72.3 | 70.5 | $69.7^{-}$ | 69.7 | 68.4 | 66.9 | 66.3 | 65.6 | 65.0 | 65.1 | 91.5 |
| 40 | 75.0 | 72.4 | 70.4 | 69.6 | $69.7^{-}$ | 68.3 | $66.9^{-}$ | 66.3 | 65.6 | 65.0 | 65.1 | 91.4 |
| 45 | 74.9 | 72.3 | 70.5 | $69.7^{-}$ | $69.7^{-}$ | 68.3 | 66.8 | 66.2 | 65.6 | 65.0 | 65.1 | 91.4 |
| 50 | 74.9 | 72.3 | 70.4 | 69.6 | 69.6 | 68.3 | 66.8 | 66.2 | 65.6 | 65.0 | 65.1 | 91.4 |

Note: Initial Steam Pressures
Main bottom reboiler 3.0 psig
Additional reboiler 4.0 psig

Table D2. Stage Liquid Temperatures for the Modified Column: Disturbance in F

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_C$ | $T_R$ | $T_{ico}$* | $T_{ici}$* | $T_{ir}$* |
| 0 | 75.5 | 73.0 | 71.0 | 70.0 | 69.9 | 68.4 | 66.7 | $66.0^{+}$ | 65.5 | 65.0 | 65.1 | 91.5 | 18.1 | 65.3 | 71.7 |
| 5 | 75.1 | 72.5 | 70.9 | 69.9 | 69.9 | 68.3 | 66.6 | 66.0 | 65.4 | 64.9 | $65.1^{-}$ | 91.4 | 18.3 | 65.3 | 71.3 |
| 10 | 75.3 | 72.5 | 70.8 | 69.9 | 69.8 | 68.3 | 66.6 | 66.0 | 65.5 | $64.9^{+}$ | $65.1^{-}$ | 91.3 | 18.2 | 65.4 | 71.2 |
| 15 | 75.3 | 72.8 | 70.9 | 69.9 | 69.8 | 68.3 | $66.6^{+}$ | 66.0 | 65.5 | 65.0 | 65.1 | 91.2 | 18.2 | 65.3 | 71.2 |
| 20 | 75.4 | 73.0 | 70.9 | 69.9 | 69.8 | 68.3 | 66.6 | 66.0 | 65.5 | 65.0 | 65.1 | 91.2 | 18.4 | 65.3 | 71.2 |
| 25 | 75.6 | 73.0 | $70.9^{+}$ | 69.9 | $69.8^{+}$ | 68.3 | 66.6 | $66.0^{+}$ | 65.5 | 65.0 | 65.1 | 91.3 | 18.2 | 65.4 | 71.3 |
| 30 | 75.4 | 73.0 | 71.0 | 69.9 | 69.9 | $68.3^{+}$ | $66.6^{+}$ | $66.0^{+}$ | 65.5 | 65.0 | 65.1 | 91.3 | 18.0 | 65.4 | 71.3 |
| 35 | 75.5 | 73.0 | 71.0 | 69.9 | 69.8 | $68.3^{-}$ | 66.6 | 66.0 | 65.5 | 65.0 | 65.1 | 91.3 | 18.3 | 65.5 | 71.3 |
| 40 | 75.4 | 73.0 | 71.0 | 69.9 | 69.9 | 68.3 | 66.6 | 66.0 | 65.5 | 65.0 | 65.1 | 91.3 | 18.1 | 65.5 | 71.3 |
| 45 | 75.4 | 72.7 | $71.0^{-}$ | 69.9 | 69.8 | 68.3 | 66.6 | 66.0 | 65.5 | 65.0 | 65.1 | 91.4 | 18.7 | 65.5 | 71.2 |
| 50 | 75.5 | 73.1 | 70.9 | 69.8 | 69.8 | $68.2^{+}$ | 66.6 | 66.0 | 65.5 | $64.9^{+}$ | 65.1 | 91.4 | 18.6 | 65.5 | 71.2 |

Note: Initial Steam Pressures
Main bottom reboiler 3.0 psig
Additional reboiler 4.0 psig
Intermediate reboiler 4.0 psig
$L'' = 300$ gm/min
$L' = 350$ gm/min
$L'_R = 260$ gm/min

*multipoint recorder reading

Table D3. Stage Liquid Temperatures for the Conventional Column: Disturbance in VPP

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_C$ | $T_R$ |
| 0 | 75.5 | 73.2 | 71.6 | 71.1 | 71.2 | 69.4 | 67.6 | 66.8 | 66.0 | 65.3 | 65.3 | 91.2 |
| 5 | 78.0 | 74.5 | 72.3 | 71.3 | 71.3 | 69.6 | 67.9 | 67.0 | 66.2 | 65.4 | 65.4 | 91.3 |
| 10 | 78.0 | 74.4 | 72.2 | 71.2 | 71.3 | 69.6 | 67.8 | 67.0 | 66.1 | 65.4 | 65.3 | 91.5 |
| 15 | 74.6 | 72.5 | 71.3 | 70.8 | 71.0 | 69.2 | 67.5 | 66.7 | 66.0 | 65.3 | 65.3 | 91.5 |
| 20 | 74.8 | 72.6 | 71.3 | 70.8 | 70.9 | 69.2 | 67.4 | 66.7 | 65.9 | 65.2 | 65.2 | 91.5 |
| 25 | 74.9 | 72.7 | 71.3 | 70.8 | 71.0 | 69.2 | 67.4 | 66.7 | 65.9 | 65.2 | 65.2 | 91.3 |
| 30 | 75.1 | 72.8 | 71.4 | 70.8 | 70.9 | 69.2 | 67.4 | 66.7 | 65.9 | 65.2 | 65.2 | 91.1 |
| 35 | 75.3 | 72.9 | 71.4 | 70.8 | 71.0 | 69.3 | 67.5 | 66.7 | 65.9 | 65.2 | 65.2 | 91.1 |
| 40 | 75.1 | 72.8 | 71.3 | 70.7 | 70.9 | 69.1 | 67.4 | 66.7 | 65.9 | 65.2 | 65.2 | 90.9 |
| 45 | 75.3 | 72.9 | 71.3 | 70.7 | 70.9 | 69.2 | 67.5 | 66.7 | 65.9 | 65.2 | 65.2 | 91.0 |
| 50 | 75.3 | 72.9 | 71.3 | 70.8 | 70.9 | 69.2 | 67.4 | 66.7 | 65.9 | 65.2 | 65.2 | 90.9 |

Note: Initial Steam Pressure
Main bottom reboiler 3.0 psig
Additional reboiler 3.0 psig

Table D4. Stage Liquid Temperatures for the Modified Column: Disturbance in VPP

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_R$ | $T_C^*$ | $T_{ici}^*$ | $T_{ico}^*$ | $T_{ir}^*$ |
| 0 | 75.4 | 73.3 | 71.8 | 70.8 | 70.7 | 68.8 | 67.0 | 66.2 | 65.6 | 65.0 | 90.9 | 64.9 | 65.6 | 17.0 | 72.7 |
| 5 | 77.5 | 74.6 | 72.3 | 71.1 | 70.9 | 69.1 | 67.2 | 66.4 | 65.7 | 65.1 | 91.2 | 64.8 | 65.7 | 17.6 | 73.2 |
| 10 | 77.7 | 74.6 | 72.4 | 71.1 | 70.9 | 69.1 | 67.2 | 66.4 | 65.7 | 65.1 | $91.3^{+}$ | 64.9 | 65.7 | 17.5 | 73.1 |
| 15 | 75.2 | 73.2 | 71.9 | 70.8 | 70.8 | 68.9 | 67.0 | 66.3 | $65.6^{+}$ | 65.0 | 91.3 | 64.8 | 65.6 | 17.5 | 73.0 |
| 20 | 75.5 | 73.2 | 71.6 | 70.7 | 70.7 | 68.8 | 67.0 | 66.2 | 65.6 | 65.0 | 91.1 | 64.9 | 65.6 | 17.7 | 72.7 |
| 25 | 75.3 | 73.3 | 71.7 | 70.7 | 70.7 | 68.8 | 67.0 | 66.2 | 65.6 | 65.0 | 91.2 | 64.9 | 65.6 | 17.7 | 72.7 |
| 30 | 75.3 | 73.2 | 71.6 | 70.7 | 70.7 | 68.8 | 67.0 | 66.2 | 65.6 | 65.0 | $91.1^{+}$ | 64.9 | 65.5 | 17.6 | 72.5 |
| 35 | 75.5 | 73.3 | 71.7 | 70.7 | 70.7 | 68.7 | 66.9 | 66.2 | 65.6 | 65.0 | 90.9 | 64.9 | 65.5 | 17.0 | 72.5 |
| 40 | 75.3 | 73.1 | 71.7 | 70.6 | 70.6 | 68.8 | $66.9^{+}$ | 66.2 | 65.6 | 65.0 | 90.9 | 64.9 | 65.4 | 16.3 | 72.4 |
| 45 | 75.4 | 73.2 | 71.6 | 70.6 | 70.7 | 68.8 | 67.0 | 66.2 | 65.6 | 65.0 | 90.9 | 64.9 | 65.4 | 16.3 | 72.3 |
| 50 | 75.5 | 73.3 | 71.6 | 70.7 | 70.6 | 68.7 | 66.9 | 66.2 | 65.6 | 65.0 | 90.8 | 64.8 | 65.4 | 16.3 | 72.3 |

Note: Initial Steam Pressures

| | |
|---|---|
| Main bottom reboiler | 3.0 psig |
| Additional reboiler | 3.0 psig |
| Intermediate reboiler | 4.0 psig |

$L'' = 300$ gm/min
$L' = 400$ gm/min
$L'_R = 290$ gm/min

*multipoint recorder reading

Table D5. Stage Liquid Temperatures for the Conventional Column: Disturbance in LBB

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_C$ | $T_R$ |
| 0 | 75.0 | 72.7 | 71.0 | 70.3 | 70.4 | 68.8 | 67.2 | 66.5 | 65.7 | 65.1 | 65.2 | 91.0 |
| 5 | 74.4 | 72.2 | 70.6 | 70.0 | 70.1 | 68.5 | 66.8 | 66.2 | 65.5 | 64.9 | 65.1 | 90.9 |
| 10 | 74.8 | 72.1 | 70.5 | 69.9 | 70.0 | 68.2 | 66.6 | 66.0 | 65.3 | 64.8 | 65.0 | 90.8 |
| 15 | 74.9 | 72.4 | 70.7 | 70.0 | 70.1 | 68.4 | 66.9 | 66.1 | 65.4 | 64.8 | 64.9 | 90.5 |
| 20 | 75.0 | 72.6 | 70.9 | 70.1 | 70.2 | 68.6 | 67.0 | 66.3 | 65.6 | 65.0 | 65.0 | 90.5 |
| 25 | 75.0 | 72.7 | 70.9 | 70.1 | 70.3 | 68.7 | 67.1 | 66.4 | 65.7 | 65.1 | 65.1 | 90.5 |
| 30 | 75.0 | 72.6 | 70.8 | 70.1 | 70.2 | 68.7 | 67.2 | 66.5 | 65.7 | 65.1 | 65.1 | 90.5 |
| 35 | 75.0 | 72.5 | 70.8 | 70.1 | 70.3 | 68.8 | 67.1 | 66.5 | 65.7 | 65.1 | 65.2 | 90.6 |
| 40 | 75.0 | 72.5 | 70.8 | 70.1 | 70.2 | 68.7 | 67.1 | 66.5 | 65.7 | 65.1 | 65.1 | 90.5 |
| 45 | 74.8 | 72.6 | 70.8 | 70.0 | 70.1 | 68.6 | 67.1 | 66.4 | 65.7 | 65.1 | 65.1 | 90.5 |
| 50 | 74.8 | 72.5 | 70.7 | 70.0 | 70.1 | 68.7 | 67.0 | 66.4 | 65.7 | 65.1 | 65.2 | 90.5 |

Note: Initial Steam Pressures
Main bottom reboiler 4.0 psig
Additional reboiler 4.0 psig

Table D6. Stage Liquid Temperatures for the Modified Column: Disturbance in LBB

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_C$ | $T_R$ | $T_{ir}^*$ | $T_{ici}^*$ | $T_{ico}^*$ |
| 0 | 75.5 | 73.3 | 71.5 | 70.5 | 70.4 | 68.5 | 66.7 | 66.0 | 65.4 | 64.9 | 65.0 | 91.7 | 72.2 | 65.3 | 18.0 |
| 5 | 75.2 | 72.9 | 71.2 | 70.2 | 70.2 | 68.2 | 66.5 | 65.9 | 65.2 | 64.7 | 64.9 | 91.6 | 72.0 | 65.3 | 18.0 |
| 10 | 75.2 | 72.6 | 71.1 | 70.1 | 70.0 | 68.1 | 66.4 | 65.7 | 65.1 | 64.6 | 64.8 | 91.4 | 71.7 | 65.2 | 18.0 |
| 15 | 75.3 | 72.8 | 71.2 | 70.3 | 70.3 | 68.3 | 66.6 | 65.8 | 65.2 | 64.7 | 64.8 | 91.3 | 71.6 | 65.3 | 18.3 |
| 20 | 76.0 | 73.4 | 71.4 | 70.3 | 70.3 | 68.4 | 66.6 | 65.8 | 65.2 | 64.7 | 64.8 | 91.1 | 71.7 | 65.3 | 18.0 |
| 25 | 75.5 | 73.4 | 71.4 | 70.3 | 70.3 | 68.4 | 66.6 | 65.9 | 65.3 | 64.8 | 64.9 | 91.2 | 71.7 | 65.3 | 18.2 |
| 30 | 75.5 | 73.3 | 71.4 | 70.3 | 70.3 | 68.4 | 66.7 | 65.9 | 65.3 | 64.8 | 64.9 | 91.2 | 71.8 | 65.4 | 18.3 |
| 35 | 75.5 | 73.3 | 71.5 | 70.4 | 70.3 | 68.5 | 66.7 | 66.0 | 65.3 | 64.8 | 64.9 | 91.2 | 71.8 | 65.5 | 18.1 |
| 40 | 76.2 | 73.3 | 71.5 | 70.4 | 70.3 | 68.5 | 66.7 | 65.9 | 65.3 | 64.8 | 64.9 | 91.2 | 71.9 | 65.5 | 18.1 |
| 45 | 75.3 | 73.2 | 71.4 | 70.5 | 70.4 | 68.6 | 66.7 | 66.0 | 65.4 | 64.8 | 64.9 | 91.3 | 71.8 | 65.5 | 18.3 |
| 50 | 75.5 | 73.4 | 71.5 | 70.4 | 70.3 | 68.5 | 66.7 | 66.0 | 65.4 | 64.9 | 65.0 | 91.3 | 71.9 | 65.5 | 18.2 |

Note: Initial Steam Pressures

| | |
|---|---|
| Main bottom reboiler | 3.0 psig |
| Additional reboiler | 4.0 psig |
| Intermediate reboiler | 4.0 psig |

$L'' = 300$ gm/min

$L' = 400$ gm/min

$L'_R = 300$ gm/min

*multipoint recorder reading

Table D7. Stage Liquid Temperatures for the Modified Column: Disturbance in L"

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ | $T_R$ | $T_C$* | $T_{ici}$* | $T_{ico}$* | $T_{ir}$* |
| 0 | 75.5 | 73.3 | 71.7 | 70.7 | 70.7 | 68.8 | $66.9^+$ | 66.2 | 65.6 | 65.0 | 91.1 | 65.0 | 65.6 | 17.5 | 72.7 |
| 5 | 75.2 | 73.1 | 71.6 | 70.6 | 70.6 | 68.7 | 66.8 | 66.1 | 65.5 | 64.9 | 91.0 | 64.9 | 65.4 | 16.5 | 72.7 |
| 10 | 75.0 | 73.1 | 71.6 | 70.7 | 70.7 | 68.7 | 66.8 | 66.1 | 65.4 | 64.9 | 90.9 | 64.9 | 65.4 | 18.5 | 72.7 |
| 15 | 75.2 | 73.3 | 71.7 | 70.7 | 70.7 | 68.8 | 66.9 | 66.2 | 65.6 | 65.0 | 91.0 | 64.9 | 65.4 | 17.2 | 72.7 |
| 20 | 75.5 | 73.3 | 71.7 | 70.7 | 70.7 | 68.8 | 66.9 | 66.2 | 65.6 | 65.0 | 91.1 | 64.9 | 65.5 | 17.4 | 72.7 |
| 25 | 75.7 | 73.7 | 71.7 | 70.8 | 70.8 | 68.8 | $67.0^-$ | 66.2 | 65.6 | 65.0 | 91.0 | 64.9 | 65.5 | 17.6 | 72.7 |
| 30 | 75.5 | 73.3 | 71.7 | 70.8 | 70.8 | 68.8 | 66.9 | 66.2 | 65.6 | 65.0 | 91.0 | 64.9 | 65.5 | 17.4 | 72.7 |

Note: Initial Reboiler Steam Pressures
- Main bottom reboiler 3.0 psig
- Additional reboiler 3.0 psig
- Intermediate reboiler 4.0 psig

L" = 300 gm/min (initially)
L' = 400 gm/min
$L'_R$ = 320 gm/min

*multipoint recorder reading

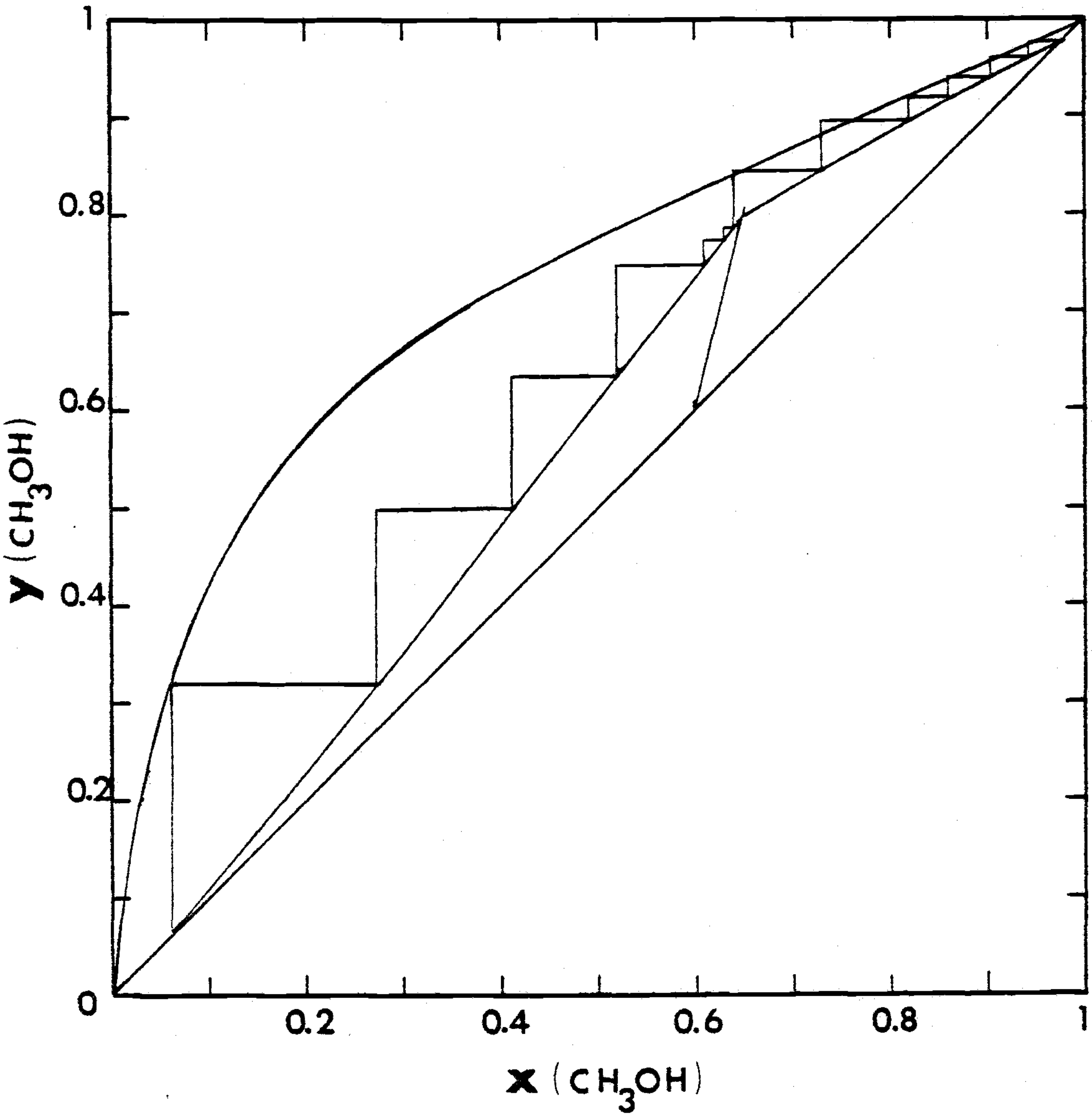



Figure D1. McCabe-Thiele Diagram for the Conventional Column (Steady State for the Experiment with Pulse in LBB).

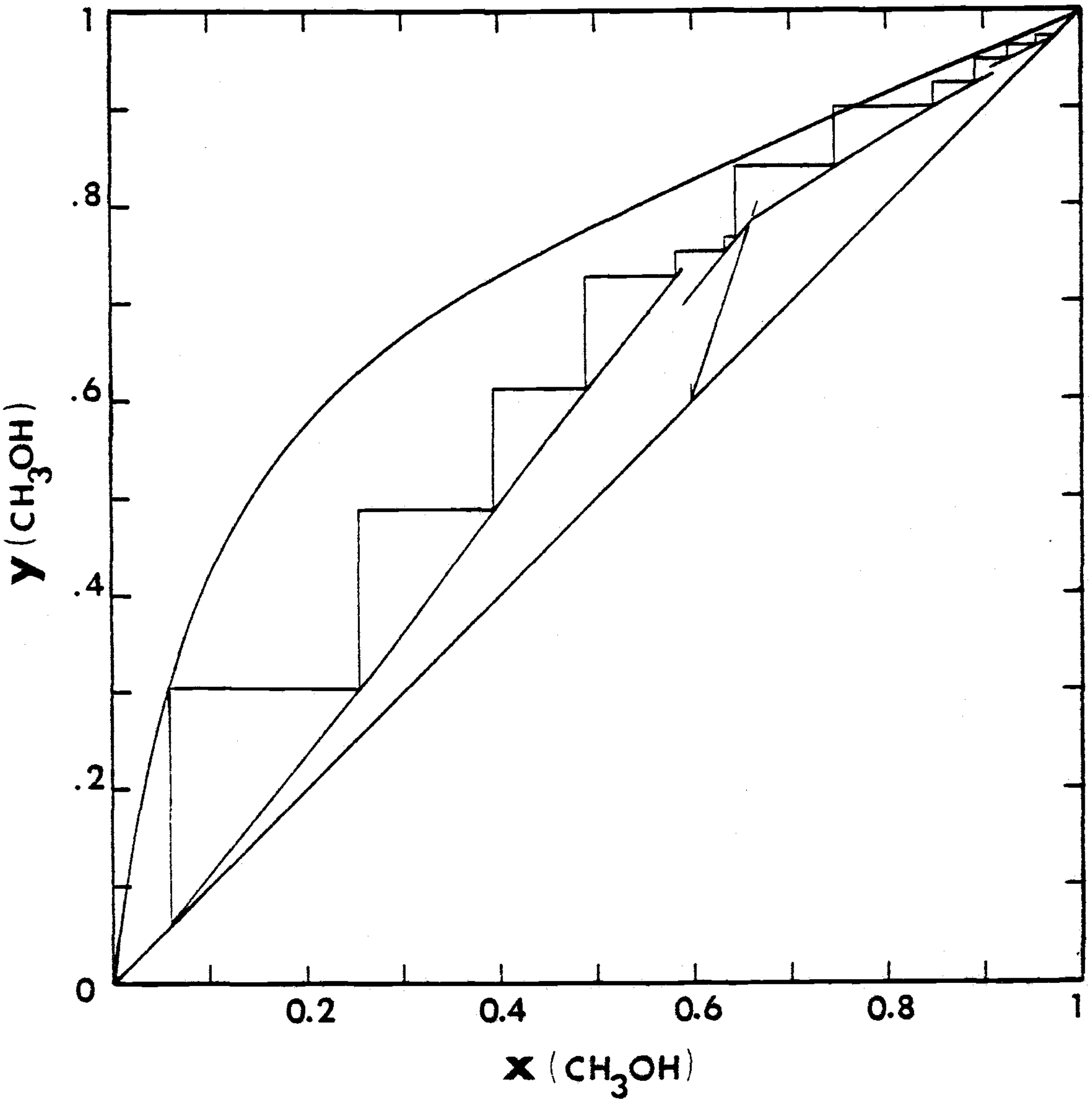



Figure D2. McCabe-Thiele Diagram for the Modified Column (Steady State for the Experiment with Pulse in LBB).

# APPENDIX E

## COMPUTER PROGRAM AND OUTPUT

```
      PROGRAM DISTNLM(INPUT,OUTPUT,TAPE5,TAPE6=OUTPUT,TAPE7)
      REAL LPP,LBB
      EXTERNAL FCN1
      DIMENSION DX(50),C(24),W(50,12),XA(50),X1(50),TEMP(50)
      COMMON/STG/JF,JIB,JIC
      COMMON/PAR/VPP,LPP,HL,VBB,LBB,
     1           B,FL(50),FV(50),SV,SL
      COMMON/VAR/H0,H,F,Z,HDP(50),Y(50),Q
      DATA (X1(I),I=1,12)/.057,.249,.401,.511,.604,.628,.635,.722,
     1                    .796,.843,.895,.937/
      DATA NW,TOL,IND,T/50,0.0001,1,0./
      DATA (HDP(I),I=2,12)/1.,7.9,7.3,6.8,6.5,5.5,5.6,5.2,
     1                      4.6,4.5,3.3/
C
C     SPECIFY THE COLUMN PARAMETERS
C
      H0=1900.
      HDP(1)=H0
      H=5.
      F=5.5
      D=3.2
      R=1.
      Q=1.4
      Z=0.597
      SV=0.
      SL=0.0
      HL=1.9
      LBB=3.2
      VPP=10.5
      VBB=VPP+(1.-Q)*F+SV-SL-HL
      LPP=LBB+SL-SV+Q*F+HL
      B=LPP-VPP
      DA=VBB-LBB
      DLBB=.2*LBB
      DVPP=0.
      DSL=0.
      DSV=0.
      DF=0.
      DZ=0.0
C
C     SPECIFY THE TOTAL NO. OF STAGES, FEED STAGE, INTERSTAGE
C     REBOILER AND CONDENSOR STAGE NUMBERS
C
      N=12
      JF=7
      JIB=5
      JIC=10
      XD=YEQLM(X1(N))
C
C     PRINT OUT THE INITIAL STEADYSTATE CONDITIONS DESCRIBING THE
C     WHOLE COLUMN
C
      CALL CISSC(X1(1),XD,DA,R,N)
C
C     PRINT THE CHANGES IN LOAD
C
      CALL CHLOAD(DF,DZ,DLBB,DVPP,DSL,DSV)
      TEND=0.
      WRITE(6,10)X1(1),(X1(I),I=3,N)
   10 FORMAT(/30X,*THE INITIAL STAGE COMPOSITIONS ARE:*,/
     1  22X,12(2X,F5.4))
      WRITE(6,30)
   30 FORMAT(/10X,*TIME(MIN)*,7X,*TB*,5X,*T1*,5X,*T2*,5X,
     1  *T3*,5X,*T4*,5X,*T5*,5X,*T6*,5X,*T7*,5X,*T8*,
     2  5X,*T9*,5X,*T10*,/)
      DO 35 IS=1,N
   35 CALL STD(X1(IS),TEMP(IS))
      WRITE(6,40)TEND,TEMP(1),(TEMP(I),I=3,N)
      WRITE(7,40)TEND,TEMP(1),(TEMP(I),I=3,N)
   40 FORMAT(10X,F6.3,6X,11(1X,F6.2))
      TEND=2.0
```

```
C
C     SET THE COLUMN PARAMETERS TO DISTURBANCE CONDITIONS.
C
      ICT=0
 80   LBB=LBB+DLBB
      VPP=VPP+DVPP
      SL=SL+DSL
      SV=SV+DSV
      Z=Z+DZ
      F=F+DF
      LPP=LBB+SL-SV+Q*F+HL
      VBB=VPP+(1.-Q)*F+SV-SL-HL
      DA=VBB-LBB
      B=LPP-VPP
      R=LBB/DA
      CALL FLOWS(N,LPP,VPP,SV,SL,HL,FL,FV)
      DO 60 L=1,10,2
      CALL DVERK(N,FCN1,T,X1,TEND,TOL,IND,C,NW,W,IER)
      DO 55 IS=1,N
 55   CALL STD(X1(IS),TEMP(IS))
      WRITE(6,40)TEND,TEMP(1),(TEMP(II),II=3,N)
      WRITE(7,40)TEND,TEMP(1),(TEMP(II),II=3,N)
      T=TEND
 60   TEND=TEND+2.0
      ICT=ICT+1
      IF(ICT.LE.1)GO TO 65
      IF(T.GE.50.) GO TO 70
      DF=0.
      DZ=0.
      DLBB=0.
      DVPP=0.
      DSL=0.
      DSV=0.
      GO TO 80
 65   DF=-DF
      DZ=-DZ
      DLBB=-DLBB
      DVPP=-DVPP
      DSL=-DSL
      DSV=-DSV
      GO TO 30
C
C     PRINT THE NEW STEADY STATE CONDITIONS
C
 70   WRITE(6,50)
 50   FORMAT(/10X,*THE NEW STEADY STATE CONDITIONS ARE*)
      WRITE(6,75)X1(1),(X1(I),I=3,N)
 75   FORMAT(/30X,*STAGE COMPOSITIONS:*,/22X,12(2X,F5.4))
C     CALL CISSC(X1(1),Y(N),DA,R,N)
      STOP
      END
C
C     THIS SUBROUTINE PRINTS THE CHANGE IN LOAD
C
      SUBROUTINE CHLOAD(A,B,C,D,E,F)
      WRITE(6,10)A,B,C,D,E,F
 10   FORMAT(/10X,*THE CHANGES IN LOAD ARE*,/10X,*DF     =*,F7.3,
     1        /10X,*DZ     =*,F7.3,/10X,*DLBB   =*,F7.3,
     2        /10X,*DVPP   =*,F7.3,/10X,*DSL    =*,F7.3,
     3        /10X,*DSV    =*,F7.3)
      RETURN
      END
C
C     THIS SUBROUTINE DESCRIBES THE INITIAL STEADY STATE
C     CONDITIONS OF THE COLUMN
C
      SUBROUTINE CISSC(XB,XD,DA,R,N)
      COMMON/STG/JF,JIB,JIC
      COMMON/PAR/VPP,LPP,HL,VBB,LBB,
     1           B,FL(50),FV(50),SV,SL
      COMMON/VAR/HO,H,F,Z,HDP(50),Y(50),Q
      WRITE(6,10)N,JF,JIB,JIC
 10   FORMAT(/10X,*NUMBER OF PLATES*,22X,I5,/10X,
     2       *FEED PLATE(FROM BOTTOM)*,15X,I5,/10X,
     3       *INTERMEDIATE REBOILER PLATE*,11X,I5,/10X,
     4       *INTERMEDIATE CONDENSOR PLATE*,10X,I5)
```

```
      WRITE(6,20)Z,XD,XB
   20 FORMAT(/10X,*FEED COMPOSITION*,24X,F7.3,/10X,
     1       *TOP PRODUCT COMPOSITION*,17X,F7.3,/10X,
     2       *BOTTOMS PRODUCT COMPOSITION*,13X,F7.3)
      WRITE(6,30)F,DA,B,LBB,VPP,SV,SL,HL
   30 FORMAT(/10X,*FEED RATE, MOLES/MIN*,20X,F7.3,/10X,
     1       *DISTILLATE RATE, MOLES/MIN*,14X,F7.3,/10X,
     2       *BOTTOMS PRODUCT RATE, MOLES/MIN*,9X,F7.3,/10X,
     3       *REFLUX RATE, MOLES/MIN*,18X,F7.3,/10X,
     4       *MAIN BOIL-UP RATE, MOLES/MIN*,12X,F7.3,/10X,
     5       *INTERM. BOIL-UP RATE, MOLES/MIN*,9X,F7.3,/10X,
     6       *INTERM. COND. LOAD, MOLES/MIN*,11X,F7.3,/10X,
     7       *TOTAL HEAT LOSS, MOLES/MIN*,14X,F7.3)
      WRITE(6,40)R,H,HO
   40 FORMAT(/10X*REFLUX RATIO*,28X,F7.3,/10X,
     1       *AVG. PLATE HOLD UP, MOLES/PLATE*,9X,F7.3/10X,
     2       *REBOILER HOLD UP, MOLES*,16X,F8.3)
      RETURN
      END
C
C     THIS SUBROUTINE DESCRIBES THE DIFFERENTIAL EQUATIONS
C     FOR THE NON-LINEAR MODEL
C
      SUBROUTINE FCN1(N,T,X1,XP)
      REAL LPP,LBB
      COMMON/STG/JF,JIB,JIC
      COMMON/VAR/HO,H,F,Z,HDP(50),Y(50),Q
      COMMON/PAR/VPP,LPP,HL,VBB,LBB,
     1           B,FL(50),FV(50),SV,SL
      DIMENSION X1(50),Y1(50),XP(50),E(12)
      DATA (E(I),I=1,12)/1.,.57,.56,.72,.34,.14,1.,1.,.77,1.,1.,.9/
      DO 10 I=1,N
   10 Y1(I)=YEQLM(X1(I))
      Y(1)=Y1(1)
      DO 15 II=2,N
   15 Y(II)=Y(II-1)+E(II)*(Y1(II)-Y(II-1))
      XP(1)=(FL(1)*X1(2)-VPP*Y(1)-B*X1(1))/HDP(1)
      NM=N-1
      DO 20 J=2,NM
      KP=J+1
      KM=J-1
      XP(J)=(FL(KP)*X1(KP)-FL(J)*X1(J)+FV(KM)*Y(KM)-FV(J)*Y(J))/HDP(J)
   20 CONTINUE
      I=JF-1
      K=JF+1
      J=JF
      XP(J)=(FL(K)*X1(K)-FL(J)*X1(J)-FV(J)*Y(J)+FV(I)*Y(I)+F*Z)/HDP(J)
      XP(N)=(LBB*Y(N)-FL(N)*X1(N)+FV(NM)*Y(NM)-FV(N)*Y(N))/HDP(N)
      RETURN
      END
C
C     THIS SUBROUTINE DETERMINES THE SATURATED LIQUID TEMPERATURES
C
      SUBROUTINE STD(X,T)
      DIMENSION CST(7)
      DATA (CST(I),I=1,7)/98.7191,-150.1635,493.4604,-1083.0034,
     1  1402.2762,-977.0760,280.2922/
      T=CST(1)
      DO 10 J=1,6
      JP=J+1
      XJ=FLOAT(J)
   10 T=T+CST(JP)*X**XJ
      RETURN
      END
C
C     THIS FUNCTION EVALUATES THE EQUILLIBRIUM VAPOR CONCNS.
C
      FUNCTION YEQLM(X3)
      DIMENSION CT(9)
      DATA (CT(I),I=1,9)/.0194,6.331,-33.061,117.9321,-273.2466,
     1                   402.7804,-360.9695,178.3382,-37.1236/
      YEQLM=CT(1)
      X3A=ABS(X3)
      DO 10 J=1,8
      JP=J+1
      XJ=FLOAT(J)
   10 YEQLM=YEQLM+CT(JP)*X3A**XJ
      RETURN
      END
```

```
C
C     THIS SUBROUTINE EVALUATES THE LIQUID AND VAPOR FLOWS
C
      SUBROUTINE FLOWS(N,XLP,XVP,SV,SL,HL,FL,FV)
      COMMON/STG/JF,JIB,JIC
      COMMON/VAR/HO,H,F,Z,HDP(50),Y(50),Q
      DIMENSION FL(50),FV(50)
      XLB=2.*HL/3.
      XL=HL/(FLOAT(N-1)*3.)
      FV(1)=XVP-XLB
      FL(1)=XLP
      FV(2)=FV(1)-XL
      FL(2)=FL(1)-XLB
      DO 10 I=3,JIB
      IM=I-1
      FL(I)=FL(IM)-XL
      FV(I)=FV(IM)-XL
 10   CONTINUE
      FV(JIB)=FV(JIB-1)+SV-XL
      JP=JIB+1
      FL(JP)=FL(JIB)+SV-XL
      FV(JP)=FV(JIB)-XL
      JP=JP+1
      DO 20 II=JP,JF
      IM=II-1
      FL(II)=FL(IM)-XL
      FV(II)=FV(IM)-XL
 20   CONTINUE
      FV(JF)=FV(JF-1)+(1.-Q)*F-XL
      JP=JF+1
      FL(JP)=FL(JF)-Q*F-XL
      FV(JP)=FV(JF)-XL
      JP=JP+1
      DO 30 IJ=JP,JIC
      IM=IJ-1
      FL(IJ)=FL(IM)-XL
      FV(IJ)=FV(IM)-XL
 30   CONTINUE
      FV(JIC)=FV(JIC-1)-SL-XL
      JP=JIC+1
      FL(JP)=FL(JIC)-SL-XL
      FV(JP)=FV(JIC)-XL
      JP=JP+1
      DO 40 M=JP,N
      IM=M-1
      FL(M)=FL(IM)-XL
      FV(M)=FV(IM)-XL
 40   CONTINUE
      RETURN
      END
```

Table E1. Simulation Results for the Conventional Column; Pulse Disturbance in LBB.

| Elasped Time (min) | Liquid Temperatures ($^{o}$C) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_R$ | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ |
| 0.000 | 91.58 | 75.31 | 72.89 | 71.10 | 70.66 | 70.53 | 68.99 | 67.66 | 66.81 | 65.89 | 65.22 |
| 2.000 | 91.55 | 74.97 | 72.65 | 70.95 | 70.52 | 70.37 | 68.74 | 67.42 | 66.58 | 65.71 | 65.10 |
| 4.000 | 91.51 | 74.77 | 72.50 | 70.83 | 70.42 | 70.28 | 68.57 | 67.22 | 66.40 | 65.58 | 65.03 |
| 6.000 | 91.46 | 74.63 | 72.39 | 70.75 | 70.34 | 70.21 | 68.44 | 67.07 | 66.27 | 65.49 | 64.99 |
| 8.000 | 91.41 | 74.53 | 72.31 | 70.68 | 70.29 | 70.17 | 68.34 | 66.95 | 66.17 | 65.43 | 64.96 |
| 10.000 | 91.36 | 74.45 | 72.25 | 70.64 | 70.25 | 70.13 | 68.27 | 66.87 | 66.10 | 65.38 | 64.93 |
| 12.000 | 91.33 | 74.70 | 72.42 | 70.74 | 70.37 | 70.28 | 68.47 | 67.02 | 66.24 | 65.48 | 65.00 |
| 14.000 | 91.32 | 74.84 | 72.54 | 70.84 | 70.45 | 70.35 | 68.59 | 67.16 | 66.36 | 65.57 | 65.04 |
| 16.000 | 91.31 | 74.93 | 72.61 | 70.89 | 70.50 | 70.39 | 68.68 | 67.27 | 66.46 | 65.64 | 65.08 |
| 18.000 | 91.30 | 74.99 | 72.66 | 70.93 | 70.53 | 70.42 | 68.75 | 67.36 | 66.53 | 65.69 | 65.11 |
| 20.000 | 91.30 | 75.03 | 72.69 | 70.96 | 70.56 | 70.44 | 68.80 | 67.42 | 66.59 | 65.73 | 65.13 |
| 22.000 | 91.29 | 75.05 | 72.71 | 70.98 | 70.57 | 70.46 | 68.84 | 67.47 | 66.64 | 65.77 | 65.15 |
| 24.000 | 91.29 | 75.08 | 72.73 | 71.00 | 70.59 | 70.47 | 68.87 | 67.51 | 66.68 | 65.79 | 65.16 |
| 26.000 | 91.29 | 75.09 | 72.75 | 71.01 | 70.60 | 70.48 | 68.89 | 67.54 | 66.70 | 65.81 | 65.17 |
| 28.000 | 91.29 | 75.10 | 72.76 | 71.02 | 70.61 | 70.48 | 68.91 | 67.57 | 66.73 | 65.83 | 65.18 |
| 30.000 | 91.29 | 75.11 | 72.76 | 71.02 | 70.61 | 70.49 | 68.92 | 67.59 | 66.74 | 65.84 | 65.19 |
| 32.000 | 91.29 | 75.12 | 72.77 | 71.03 | 70.62 | 70.49 | 68.93 | 67.60 | 66.76 | 65.85 | 65.19 |
| 34.000 | 91.29 | 75.13 | 72.78 | 71.03 | 70.62 | 70.50 | 68.94 | 67.61 | 66.76 | 65.86 | 65.20 |
| 36.000 | 91.29 | 75.13 | 72.78 | 71.04 | 70.62 | 70.50 | 68.95 | 67.62 | 66.77 | 65.86 | 65.20 |
| 38.000 | 91.29 | 75.13 | 72.78 | 71.04 | 70.62 | 70.50 | 68.95 | 67.62 | 66.78 | 65.87 | 65.20 |
| 40.000 | 91.29 | 75.14 | 72.78 | 71.04 | 70.63 | 70.50 | 68.96 | 67.63 | 66.78 | 65.87 | 65.21 |
| 42.000 | 91.29 | 75.14 | 72.79 | 71.04 | 70.63 | 70.51 | 68.96 | 67.63 | 66.79 | 65.87 | 65.21 |
| 44.000 | 91.30 | 75.14 | 72.79 | 71.04 | 70.63 | 70.51 | 68.96 | 67.64 | 66.79 | 65.88 | 65.21 |
| 46.000 | 91.30 | 75.14 | 72.79 | 71.04 | 70.63 | 70.51 | 68.96 | 67.64 | 66.79 | 65.88 | 65.21 |
| 48.000 | 91.30 | 75.14 | 72.79 | 71.04 | 70.63 | 70.51 | 68.97 | 67.64 | 66.79 | 65.88 | 65.21 |
| 50.000 | 91.30 | 75.15 | 72.79 | 71.04 | 70.63 | 70.51 | 68.97 | 67.64 | 66.79 | 65.88 | 65.21 |

Table E2. Simulation Results for the Modified Column: Pulse Disturbance in LBB.

| Elapsed Time (min) | Liquid Temperatures (°C) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $T_R$ | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $T_6$ | $T_7$ | $T_8$ | $T_9$ | $T_{10}$ |
| 0.000 | 92.10 | 75.83 | 73.55 | 71.60 | 70.77 | 70.61 | 68.72 | 67.08 | 66.24 | 65.51 | 65.03 |
| 2.000 | 92.08 | 75.45 | 73.28 | 71.42 | 70.59 | 70.41 | 68.45 | 66.86 | 66.06 | 65.39 | 64.96 |
| 4.000 | 92.04 | 75.20 | 73.07 | 71.26 | 70.47 | 70.30 | 68.28 | 66.69 | 65.93 | 65.30 | 64.92 |
| 6.000 | 91.99 | 75.01 | 72.93 | 71.15 | 70.39 | 70.23 | 68.17 | 66.58 | 65.85 | 65.25 | 64.89 |
| 8.000 | 91.94 | 74.89 | 72.83 | 71.08 | 70.33 | 70.18 | 68.09 | 66.50 | 65.79 | 65.21 | 64.87 |
| 10.000 | 91.88 | 74.79 | 72.76 | 71.03 | 70.29 | 70.14 | 68.03 | 66.44 | 65.74 | 65.18 | 64.85 |
| 12.000 | 91.86 | 75.05 | 72.96 | 71.17 | 70.43 | 70.32 | 68.25 | 66.59 | 65.86 | 65.25 | 64.89 |
| 14.000 | 91.84 | 75.23 | 73.11 | 71.29 | 70.52 | 70.39 | 68.37 | 66.72 | 65.95 | 65.31 | 64.92 |
| 16.000 | 91.83 | 75.36 | 73.22 | 71.37 | 70.58 | 70.45 | 68.46 | 66.81 | 66.02 | 65.36 | 64.95 |
| 18.000 | 91.82 | 75.44 | 73.28 | 71.42 | 70.63 | 70.48 | 68.52 | 66.87 | 66.08 | 65.40 | 64.97 |
| 20.000 | 91.81 | 75.50 | 73.33 | 71.46 | 70.65 | 70.51 | 68.57 | 66.92 | 66.11 | 65.42 | 64.98 |
| 22.000 | 91.81 | 75.54 | 73.36 | 71.49 | 70.67 | 70.52 | 68.60 | 66.96 | 66.14 | 65.44 | 64.99 |
| 24.000 | 91.81 | 75.57 | 73.39 | 71.51 | 70.69 | 70.54 | 68.62 | 66.98 | 66.16 | 65.46 | 65.00 |
| 26.000 | 91.81 | 75.59 | 73.40 | 71.52 | 70.70 | 70.55 | 68.64 | 67.00 | 66.18 | 65.47 | 65.01 |
| 28.000 | 91.81 | 75.60 | 73.41 | 71.53 | 70.71 | 70.55 | 68.65 | 67.01 | 66.19 | 65.48 | 65.01 |
| 30.000 | 91.81 | 75.61 | 73.42 | 71.53 | 70.71 | 70.56 | 68.66 | 67.02 | 66.20 | 65.48 | 65.01 |
| 32.000 | 91.81 | 75.62 | 73.43 | 71.54 | 70.71 | 70.56 | 68.66 | 67.03 | 66.20 | 65.49 | 65.02 |
| 34.000 | 91.81 | 75.63 | 73.43 | 71.54 | 70.72 | 70.56 | 68.67 | 67.04 | 66.21 | 65.49 | 65.02 |
| 36.000 | 91.81 | 75.63 | 73.44 | 71.55 | 70.72 | 70.57 | 68.67 | 67.04 | 66.21 | 65.49 | 65.02 |
| 38.000 | 91.81 | 75.63 | 73.44 | 71.55 | 70.72 | 70.57 | 68.67 | 67.04 | 66.21 | 65.49 | 65.02 |
| 40.000 | 91.81 | 75.64 | 73.44 | 71.55 | 70.72 | 70.57 | 68.68 | 67.05 | 66.21 | 65.50 | 65.02 |
| 42.000 | 91.82 | 75.64 | 73.44 | 71.55 | 70.72 | 70.57 | 68.68 | 67.05 | 66.21 | 65.50 | 65.02 |
| 44.000 | 91.82 | 75.64 | 73.45 | 71.55 | 70.72 | 70.57 | 68.68 | 67.05 | 66.21 | 65.50 | 65.02 |
| 46.000 | 91.82 | 75.64 | 73.45 | 71.55 | 70.73 | 70.57 | 68.68 | 67.05 | 66.22 | 65.50 | 65.02 |
| 48.000 | 91.82 | 75.64 | 73.45 | 71.55 | 70.73 | 70.57 | 68.68 | 67.05 | 66.22 | 65.50 | 65.02 |
| 50.000 | 91.82 | 75.64 | 73.45 | 71.55 | 70.73 | 70.57 | 68.68 | 67.05 | 66.22 | 65.50 | 65.02 |

# APPENDIX F

## FUTURE WORK

Two loose ends should be tied up for those who might do more work on the experimental equipment developed for this thesis. Some preliminary development work has been done both on automation of the data sampling and on the control setup necessary for process control experiments. This work is summarized here.

As indicated in the text, manual sampling of the temperature data contributes to the experimental error. Automatic data sampling could both reduce the experimental error and free the operator to maintain better control over the experiment itself. Given the availability of a Nova 840 minicomputer with real time capabilities in the Chemical Engineering Department and the Analog Devices 2036 digital thermometers available in the experimental setup, it is possible that the two could be interfaced for remote data sampling.

In addition to standard manual operation, the AD 2036 digital thermometers have been designed to accept a computer generated binary coded decimal (BCD) channel input and also have the capability to output for computer use the contents of the channel in BCD form. Since the digital thermometers only have six channels, three of the meters are required to handle all of the temperature inputs from the experimental setup. Connection of all three meters with the minicomputer requires some multiplexing of inputs and outputs. The preliminary electronics to do this have been developed for one of the meters by John McKeon and are available in the Chemical Engineering Department.

The distillation equipment could also be developed for process control work. A number of things are available and should be noted here.

1. The Honeywell controller mentioned in the text could be used in

process control experiments. The controller has, in addition to the standard PID controller settings, the capability for operation as a computer station with direct digital control (DDC).

2. The reflux ratio controller also has a computer operated mode (in addition to the manual mode) and could be interfaced with the minicomputer for DDC of the reflux ratio.

3. Control valves have already been placed on the steam lines to the main bottom reboiler and to the intermediate reboiler. These control valves are both ½-inch Taylor air-to-open valves with ¼-inch orifices.

With these instruments and valves, one could control the conventional column. However, if it were desired to do control experiments on the modified column with feed forward or feed back control on the intermediate heat exchangers, the intermediate "condensation" rate must also be controlled. Preliminary work indicates that a control valve on the air supply line to the Micro Pump motor could be used to control the liquid flowrate through the "condenser." If the "condenser" has sufficient heat transfer area so that the liquid always approaches the cooling water temperature regardless of flowrate, then the "condensation" rate can be controlled by controlling the liquid flow. Using a Research Control Valve (trim size J) positioned on the air supply line to the Micro Pump, the liquid flow data presented in Figure F1 were collected. The air supply pressure for the data indicated in the figure was maintained at an average value of 50 psig. It is clear from Figure F1 that the liquid flowrate can be controlled by manipulation of the air flow to the Micro Pump motor.

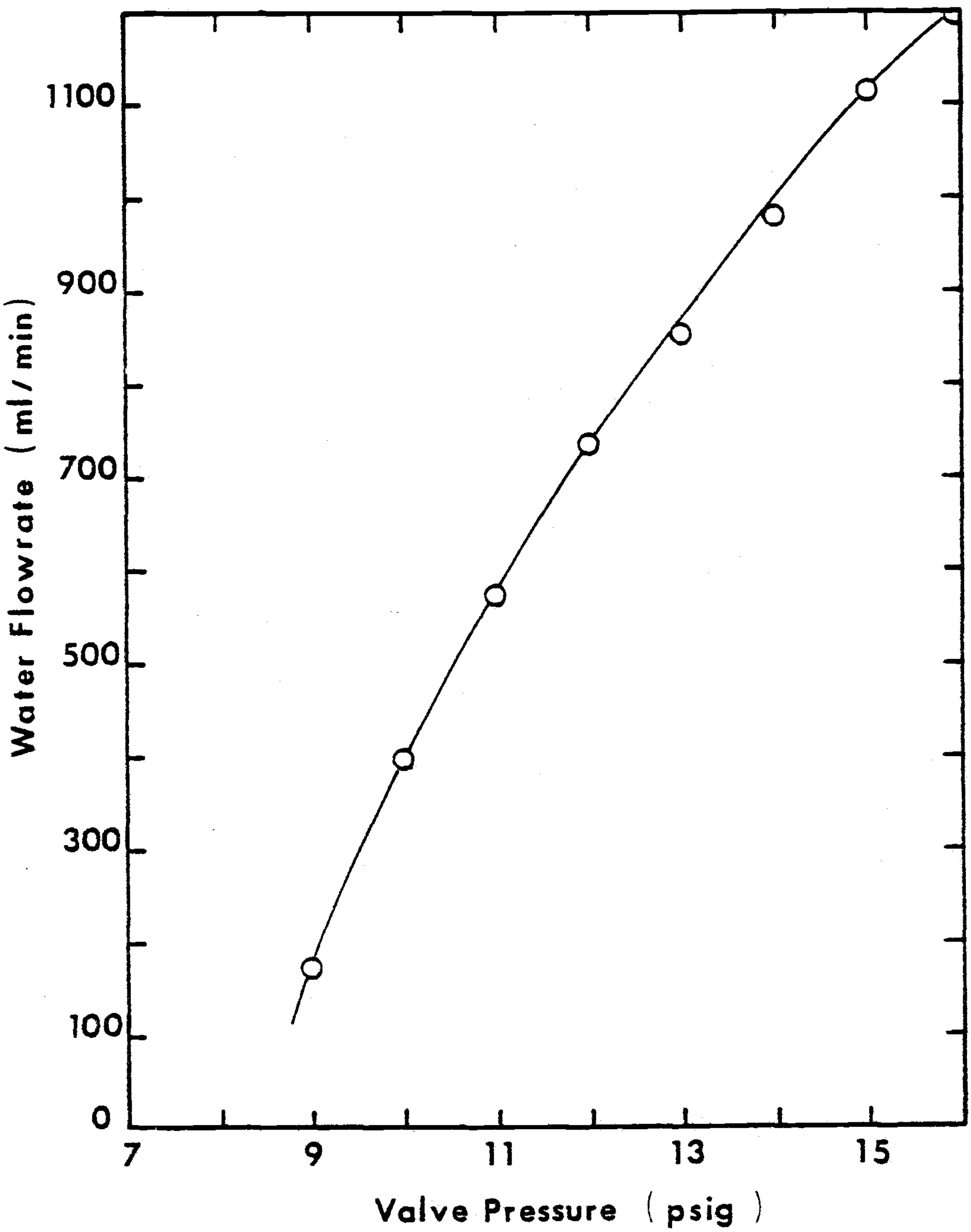



Figure F1. Liquid Flowrates for the Research Control Valve (Trim J) - Micro Pump Combination.