

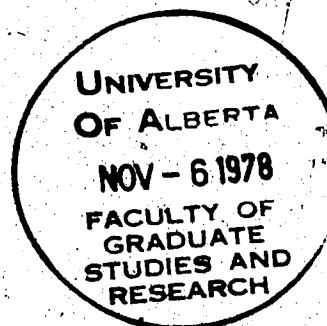
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NONLINEAR BINARY DISTILLATION COLUMN MODELS

by



ULRICH F. SIMONSMIEIER

A THESIS
SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND
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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research, for acceptance, a thesis entitled Nonlinear Binary Distillation Column Models submitted by Ulrich F. Simonsmeier in partial fulfillment of the requirements for the degree of Master of Science.

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ABSTRACT

This thesis is concerned with the development of a non-linear binary distillation model, to include the hydraulic effects of variable volume tray holdup. Verification of this model is made by comparison to open loop data obtained from a pilot plant column. In addition, other limiting assumptions are consecutively applied to the model, and effects on model behavior observed.

A literature survey presents various types of distillation models and their applications. A list of common modeling assumptions is given along with effects upon model complexity and order. A description of workers' treatment of special stages such as reboiler and condenser is included.

Mass, component, energy balances, and a linear hold-up correlation were used as a basis to develop a nonlinear model of a ten stage binary column. Two methods of numerical solution of the resulting forty equations were developed. Simulations were made for reflux and steam flow disturbances.

The model was simplified to include the assumption of constant tray holdup. Further limiting assumptions were consecutively applied and simulations were run for steam, reflux and feed flow disturbances.

Verification of the constant tray holdup model was

achieved by comparison to open loop transient data obtained from a pilot plant column. Very good agreement was shown with heat loss and efficiency used as fitting parameters. Comparison of the constant tray holdup model to the variable tray holdup model and other more simple models indicate the effects of various assumptions upon model behavior. Simulation results from the variable tray holdup model with three different holdup correlations are shown. It is shown that the choice of a holdup correlation can significantly affect the simulated column dynamics.

The final model formulation was found to lend itself easily to modification, and handled the variable holdup consideration well, providing a stable solution for a variety of cases.

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CHAPTER 1

INTRODUCTION

It has been stated by prominent chemical engineers and designers that process dynamics and control should be considered in the initial design of a process. Such a design philosophy would include the development of a mathematical process model to aid in improving process operation on the basis of dynamic and control considerations. This approach has not been widely applied, with the control system generally being designed after the equipment design has been finalized.

The desired design philosophy must combine the mathematical representation of the process system with tentative control schemes. Simulations of the controlled system behavior would lead to improved process designs. The application of this approach has been limited due to the difficulty of understanding the process physically and developing adequate mathematical models at reasonable cost. It is for these reasons that such a design philosophy has been successfully applied only to the more simple process systems.

Distillation, however, is an exception, being a relatively complex process. It has been the subject of many studies in regard to dynamic modelling, as it is one of the oldest and most widely used process units in industry. Models

of varying complexity have been derived, some having been verified by comparison to actual pilot plant data. These models include limiting assumptions that determine their rigor or completeness. A complete description of simplifying assumptions commonly used in distillation modelling is presented in a later section.

A major assumption which is made in deriving any distillation model concerns holdup and hydrodynamic lag. The primary purpose of this work was to eliminate any assumption concerning holdup or hydrodynamic lag, augmenting a fully non-linear model already incorporating a minimal number of assumptions. This proposed model describes open loop transients of tray and terminal compositions for a variety of disturbances, including changes in feed, steam and reflux flows. The model is verified by comparison to open loop data from a 8 inch diameter, 10 stage pilot plant distillation column using a methanol-water feed.

Models developed previously have given rise to favorable prediction when compared to experimental data, or have been used to simulate successfully a column under a new control scheme. However, few comparisons have been made between models of varying complexity to determine the error introduced by particular assumptions, reduction of order and/or linearization. Thus, the objective of this work was to use a completely non-linear experimentally verified model and then

modify the model by more restrictive simplifying assumptions, determining their effects upon simulated column behavior.

CHAPTER 2

LITERATURE REVIEW

2.1 ASSUMPTIONS

2.1.1 Introduction

There have been two distinct approaches to modelling distillation and other processes. The first and most elementary is fitting a transfer function model of predetermined order and complexity to open loop responses between desired input and output variables. The second, and more flexible method is the application of known physical relationships such as mixing equations or mass and energy balances to sections of the process and predicting open loop responses. The complexity of the models that would result from such an approach is determined by the assumptions employed. The most common assumptions made in distillation modelling are listed below:

1. Negligible vapor holdup and dynamics - changes in vapor rate are assumed instantaneous, and vapor holdup is considered negligible relative to liquid holdup. Use of such assumptions, which are normally justified, significantly reduces the number of equations to be solved.

2. Molar, mass or volume holdup constant - the specific assumption chosen depends upon the extent of the simplification desired. This simplification has been used in almost

all distillation models to date.

3. Liquid dynamics negligible - with this assumption it is considered that a disturbance in liquid flow rate will propagate through the column instantaneously. This may be a good assumption if the composition dynamics are very slow, or if there is only a slight dependence of holdup upon liquid flow rate.

4. Constant pressure operation - simplifies calculations in that the effect of changes in pressure on equilibrium or enthalpy considerations need not be considered. Tower pressure is under control in most processes.

5. Well mixed liquid and vapor phase in each stage - only stage efficiencies are required to establish liquid and vapor compositions on each tray.

6. Adiabatic operation or other heat loss assumption - these assumptions are only applicable if energy balances are used in the model. Adiabatic operation may be valid for well insulated or enclosed columns. Other common assumptions are those of constant heat loss or heat loss varying with composition and/or temperature.

7. Constant efficiency or some function of operating conditions - no simple correlation for predicting the efficiency of small columns exists so efficiency is used as a parameter to fit experimental data at steady state and/or during transient operation.

8. Constant molal overflow - the liquid and vapour flow rates are constant in each section of the column. This is a severe assumption and usually only introduced with minimum prior knowledge of column operating conditions.

9. Perturbation theory applies - only small excursions from steady state will be experienced which allows equilibrium and enthalpy relationships and/or conservation equations to be linearized.

10. Heat capacity of tray metal and/or column walls negligible - energy storage considered to be small compared to magnitude of other terms in enthalpy balances.

As shall be seen in later sections, all modelling work to date has included the use of at least two of the above assumptions. The accuracy of models predictions using certain of the above assumptions will also be considered in later sections.

2.1.2 Holdup and Hydrodynamic Lag

To a great extent in the literature the effect of varying holdup and hydrodynamic lag have been considered to be negligible. Physically, liquid flow from a tray is determined by the head of liquid on the tray, the amount of frothing, and to a certain extent, the pressure differential and vapour flow to the tray. The exact nature of this relationship is unknown. For conservation of mass, an increase in input liquid flow to a tray would result in an increase in liquid outflow and

possibly an increase in liquid head on the tray. As in other reservoirs with a restricted outflow, the relationship may be a lag equation

$$\frac{L(s)}{L_i(s)} = \frac{1}{\tau s + 1} \quad (2.1)$$

and the holdup dynamics would also be in the form of a lag

$$\frac{WT(s)}{L_i(s)} = \frac{K}{\tau s + 1} \quad (2.2)$$

Proposed models reported by Stainthorp and Searson¹, Scholander² and Waller³ have used expression (2.1) to relate liquid flow rates.

Many linear models assume constant liquid flow rates and holdups ($L(t) = L_0$, $WT(t) = WT_0$) upon which composition transfer functions or matrices are based. Such is the case with models developed by Osborne et al.⁴, by Sittel and Fisher⁵ and by Rosenbrock^{6,7}.

Holdup and hydrodynamic lag are treated in a different manner in the more complex nonlinear models. Liquid and vapor flows at any time are determined by mass and energy balances on each tray, taking into account accumulation or holdup changes due to density or composition changes. This eliminates liquid dynamics altogether with flows changing only with composition effects or heat effects. This approach was adopted by Huckaba et al.⁸ and by Rafal et al.⁹

in their studies.

As stated in section 2.1.1, the most common assumption made with respect to holdup is to assume the holdup to be constant on a mass, molar or volume basis. Distefano¹⁰ has altered the model of Huckaba et al.⁸ to correct tray holdup for changes in liquid and vapor rate. Holdup was updated each integration interval, but changes during each time step were not considered when solving for new liquid and vapor flow rates, resulting in neglect of liquid dynamics.

The flow of liquid from one tray to another is through a downcomer, its length approximately that of the tray spacing. In this region, liquid does not come into contact with the vapor, and should be modelled as a pure time delay in liquid flow. This approach was used in the models of Peiser and Grover¹¹ and that of Distefano¹⁰.

It is not obvious if there is a basis that should be employed regarding the use of particular assumptions since good results were obtained by previous workers using a particular model for a specific case. Only upon comparison of experimental data with simulation results from different models can a judgement be made. Even the prediction of actual holdup values for bubble cap or sieve trays involves considerable uncertainty.

2.2 TRANSFER FUNCTION MODELS

The earliest distillation column models tended to be the simplest, and transfer function models fall into this category. Transfer function models are easily obtained, by fitting to open loop data or by simple mathematical analyses of the system. Transfer function models only characterize nonlinear plants adequately provided there is no strong nonlinear system behavior. The distillation column is a nonlinear system; however, for small excursions from steady state, the transfer function model may be representative. However, with the linear model and a highly nonlinear system, modelling error became important, and with the wide availability of digital computers rather than analog computers, other system representatives were derived.

A process transfer function relates the dynamic response of an output variable to a disturbance of the input variable. For a process transfer function is given below.

$$G(s) = \frac{C(s)}{U(s)} \quad (2.3)$$

The time domain response is obtained from the inverse Laplace transform according to

$$C(t) = L^{-1}\{G(s)U(s)\} \quad (2.4)$$

In a distillation column, the variables of interest or output variables are usually the terminal compositions or compositions of the product streams. The disturbance variables are usually the feed flow, feed composition and feed enthalpy. The relations between heat input (steam flow) or reflux and bottom composition or top composition are also important for control studies.

Wahl and Harriot¹⁹ attempted to predict the time constants of these transfer functions for a 15 plate methanol-water column by fitting open loop data, and by using previously developed correlations based upon a recirculation rate and a characteristic time constant (column parameters). The correlations predicted both zeros and poles from steady state conditions.

Transfer function models relating certain tray temperatures to reflux and steam flows, determined empirically for $\pm 10\%$ step changes in flow rate, were used by Vinante and Luyben¹⁵ for decoupling control of a distillation column. Luyben and Tyreus¹⁶, using a first order hydraulic dynamics transfer function model fitted to experimental data, evaluated sidestream composition control. Other workers who have derived transfer function models are Sittel and Fisher^{21, 20}, Paul et al.³⁷, and Waller³, the latter assuming that flow lags and composition lags can be treated independently.

Another approach has been to simplify higher order or more complex models by fitting their predicted responses with transfer functions. Typical are the studies of Izawa and Morinaga¹⁷ and Beaverstock and Harriot²⁵.

2.3 STATE SPACE MODELS

State space models may be represented by Equation (2.5)

$$\dot{\underline{x}} = \underline{A} \underline{x} + \underline{B} \underline{u} \quad (2.5)$$

where $\underline{A}$ is a matrix of coefficients and $\underline{x}$ is a state vector. The state vector is a convenient way of representing important system operating variables, individually or as linear combinations of other variables. The matrix $\underline{A}$ is a matrix of coefficients, predicting trajectories of the system through state space. The other terms on the right hand side of Equation (2.5) are comprised similarly of a matrix of coefficients and a vector which may be disturbance or control terms.

State space models may be derived by mass and/or energy balance considerations. Each tray in a distillation column has composition, energy and total balances associated with it, as well as equilibrium relationships and holdup considerations, which may all be used in expressing a state space model. These relationships, however, must be linear or linearized. The state vector may be plate compositions and/or enthalpies, augmented by liquid or vapor rates or holdups,

depending upon model rigour or complexity.

Scholander² used a model based on material balances to calculate feedforward and feedback matrices for bottoms composition control. Rafal and Stevens⁹ derived optimal controls based on quadratic performance indices for a distillation system using a state space model. A model by Clear¹² used K values to predict equilibrium conditions, and heat and mass balances to describe column dynamics. Binder and Calyillo¹⁸ used a state space model to test a tracking control scheme and an adaptive-predictive control scheme. The derivation of these various control laws, and the use of other standardized techniques for model reduction are all matrix operations, and are possible because of the state space models linearity property, and the form of the model. Terris²² used a matrix technique to reduce a state space model to order 2 from 121, comparing time constants and gains of the reduced model to industrial data. Simplification of this kind may reduce computation or complexity in application but with some loss of accuracy.

Pike and Thomas²³ obtained a fourth order state space model by discretizing Laplace transfer functions and converting to a matrix form. With mathematical techniques applicable to matrices but not to Laplace functions, they calculated an optimal control strategy with two variable control.

The linearity and practicality of the state space models allow a variety of control strategies to be rigorously calculated and implemented. Unfortunately, such a model has found limited application since a distillation column is a nonlinear system. Further, without fitting of some parameters such as efficiencies and/or heat loss, even steady state agreement between calculated and experimental conditions is unlikely.

2.4 NONLINEAR MODELS

A nonlinear model consists of a system of nonlinear differential and algebraic equations. In practice, discrete methods for the solution of nonlinear differential equations are as applied to linear equations with changing coefficients; the solution for the nonlinear distillation system is obtained by solving the composition balance equations, of the form given by Equation (2.5), and updating the $\underline{A}$ matrix for changes in the state vector $\underline{x}$.

The nature of the updating technique is again dependent upon the nature of the simplifying assumptions chosen. When the nonlinear model is derived from mass, energy and component balances, such as in the work of Huckaba et al.⁸, the system of differential equations involves the liquid and vapor flow rates, vapor compositions and stage holdups. These may be established via a number of routes, and using various assumptions such as constant molal overflow and 100 percent

tray efficiency as in the work of Osborne et al.⁴. Distefano²⁴ solved for the composition coefficients, as was done by Huckaba and coworkers, but included the effect of a dead time in liquid flow from tray to tray and the effect of changing volume holdup on the tray. Svrcek²⁶ modelled a distillation column similarly but assumed constant volume but variable mass of liquid holdup on the tray. Peiser and Grover¹¹ used mass, energy and component balances to derive a model based on a 4 component system. The numerical techniques used to solve the system of equations differ from case to case.

The applications of non-linear distillation modelling have been to test proposed control schemes, or have involved comparison of dynamic simulation to experimental data. Few comparisons have been made between linear and nonlinear models, or between nonlinear models based on different assumptions. One such example can be found in the work of Rafal et al.⁹, who compared two methods of linearizing physical property relationships but found no significant difference.

To compare favorably with experimental data, a certain amount of fitting has been required. Reliable correlations for holdups or efficiencies are lacking, forcing workers to adopt a fitting approach. Distefano²⁴ developed his own empirical holdup correlation for lack of a better one. Beaverstock and Harriot²⁵ reported in their pilot plant

modelling effort that the Francis Wier formula predicted holdup values larger than measured by a factor of 2 and hydraulic time constants one third the measured values. Other workers assumed 100 percent efficiency when methods of efficiency prediction were available but supposedly did not apply in their particular application. The weakness of developing more complex models has been a lack of consistency in predicting the values of the increased numbers of parameters required in higher order models.

2.5 SPECIAL STAGES

On a macroscopic basis, all stages may be treated equally, as the general equations describing stages include special terms such as heat input or liquid and vapor drawoffs. The reboiler and condenser however, being terminal stages may be termed special as in both cases there appears a time varying heat term usually orders of magnitude greater than in "ordinary" stages. It is this heat term and special mixing considerations that have given these stages a special status.

Heat input to the reboiler involves the transfer of heat from one medium, such as steam, to the reboiler liquid. In most studies, reboiler heat duty, rather than condenser duty, is taken to be a specified process degree of freedom.

Reboiler modelling involves heat input prediction and

some mixing assumption. Peiser and Grover²⁷ used enthalpy, mass and component balances about the reboiler to obtain transfer function models relating liquid composition in the reboiler (assumed perfectly mixed) to steam and liquid in flow disturbances. Heat input was taken as a linear function of average temperature differential, and was varied with time. Distefano et al.¹⁰ also used heat balances to solve for heat input, based on steam temperature, and reboiler liquid composition. As in the work of Peiser and Grover, complete mixing was assumed. Based upon actual pilot plant measurements, Distefano et al. considered errors of 5 percent due to correlation inaccuracies to be reasonable limits for heat input prediction. Sproul and Gerster¹³, using composition balances only but including entrainment effects solved for a composition profile at various points in a reboiler system. They found that dynamics were changed by varying the entrainment factor. Svrcek²⁶ showed mathematically that for his pilot plant column, heat transfer dynamics were rapid compared to the composition dynamics, so were considered instantaneous. Heat input has also been considered constant, as in all transfer function models, and as in the matrix model used by Clear.¹²

The condenser is a special stage in that a large quantity of heat is removed. All mass entering this stage is condensed and withdrawn as product under level control except the liquid

that is returned to the column as reflux. Reflux rate and enthalpy are the two process degrees of freedom that are specified. Thus the condenser is characterized by one composition differential equation.

CHAPTER 3

MODEL DEVELOPMENT

3.1 THE EQUATIONS

3.1.1 The Distillation Column

A distillation column may be considered to be a series of mixing stages, in which the vapor and liquid streams enter, and leave accompanied by interchange of mass and energy between the phases. Condensation and flashing result in partial or total equilibrium between the vapor and liquid phases. The degree of foaming, frothing and mixing in each stage determines the amount of interfacial contact which in turn limits the extent of equilibrium achieved. On a microscopic scale, rigorous mathematical characterization of these physical processes is lacking, so the approach to modelling taken in this work was macroscopic in nature. It is assumed that complete mixing, mass transfer and heat transfer equilibrium states were instantaneously achieved, and that overall balances were satisfied.

In a distillation column, although there are many changing variables and properties, such as diffusivities and viscosities etc., the basis of column behavior can be expressed by equations in " $4n$ " variables, (where " n " is the number of stages). These are the mass circulation rates (both liquid and vapor flows from each stage), the mass

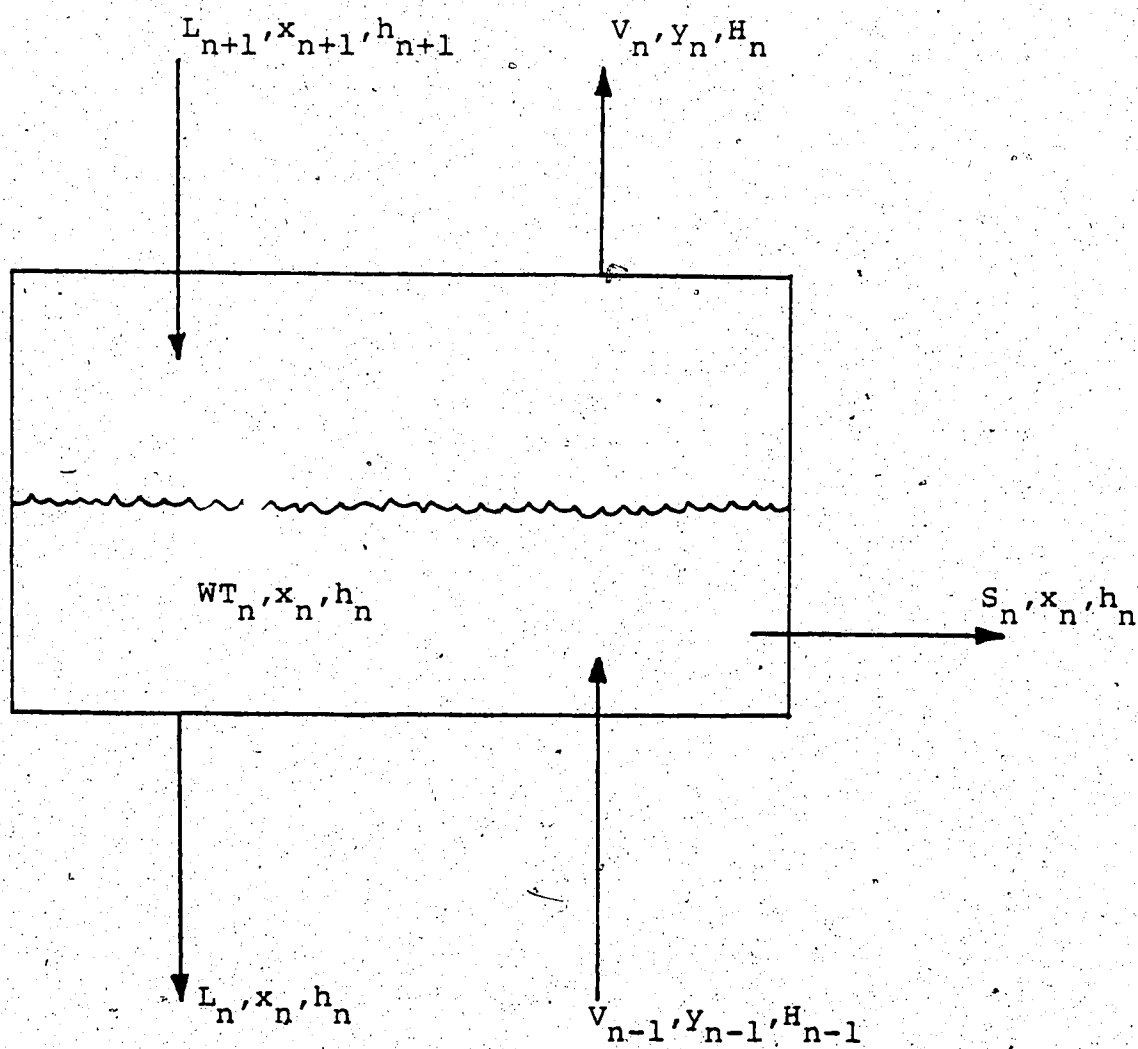


FIGURE 3.1 A Theoretical Stage

holdups on the stages, and the liquid compositions on the stages. Other variables such as density, enthalpy and accumulation rates follow directly from these base variables.

The governing equations (including liquid feed and sidestream drawoff terms) are:

mass balance

$$0 = \frac{dWT_n}{dt} = L_{n+1} - L_n + V_{n-1} - V_n - S_n + F_n \quad (3.1)$$

component balance

$$0 = \frac{dWT_n x_n}{dt} = L_{n+1} x_{n+1} - L_n x_n - V_n y_n + V_{n-1} y_{n-1} - S_n x_n + F_n x_{Fn} \quad (3.2)$$

enthalpy balance

$$0 = \frac{dWT_n h_n}{dt} = L_{n+1} h_{n+1} - L_n h_n - V_n H_n + V_{n-1} H_{n-1} - S_n h_n + F_n h_{Fn} - Q \quad (3.3)$$

The column is said to be at steady state when the rate of change of concentrations on each tray, and the rates of mass and energy accumulation, that is the left hand sides of equations (3.1), (3.2) and (3.3) are identically equal to zero.

When the system is not at steady state, the accumulation terms are not zero, but some instantaneous value, such that all balances are satisfied. Since these balances then

compromise $3n$ equations, a total balance, an energy balance, and a component balance for each tray, a further n independent equations will be needed to solve for the $4n$ base unknowns. These equations are algebraic in the form of holdup correlations.

$$WT_n = a_n L_n + b_n V_n + c_n \quad (3.4)$$

The values for a_n , b_n , c_n which may be functions of liquid and vapor density are arbitrary. No correlation exists to date which adequately represents experimental data for a wide variety of columns and operating conditions. For $b_n = 0$, the equation reduces to being a wier type correlation.

This set of equations was chosen for several reasons:

1. The equations are expressed in terms of basic chemical and physical properties and flows that must be satisfied at any instant of time.
2. There are no assumptions regarding the nature of the liquid dynamics, only that the holdup correlation is valid.
3. The equations are general and so are applicable to any column or set of equilibrium stages.

For a binary mixture, thermodynamic data or correlations necessary to determine the coefficients in equations (3.1) to (3.3) can be expressed as

$$H_n = H(y_n) \quad (3.5)$$

$$h_n = h(x_n) \quad (3.6)$$

$$Y_n = y(x_n) \quad (3.7)$$

Since the pressure in the distillation column is considered constant, no allowances for pressure dependence have been incorporated into the thermodynamic or equilibrium representations. Expression (3.7) includes not only the basic equilibrium data, but takes into account the efficiency of contact. A Murphree vapor efficiency defined by

$$E_n = \frac{Y_{n-1} - Y_n}{Y_{n-1} - Y_n^*} \quad (3.8)$$

is applied to the equilibrium vapor composition to obtain the actual vapor composition according to

$$Y_n = Y_{n-1} + E_n(Y_n^* - Y_{n-1}) \quad (3.9)$$

3.1.2 The Reboiler

The reboiler, unlike the trays, contains a relatively large liquid holdup, and has no input vapor flow but has a large heat input. Heat input is by a shell and tube heat exchanger, with condensing steam providing the energy to flash the methanol-water solution.

The heat transfer depends on the overall heat transfer coefficient, the steam temperature and the reboiler liquid temperature.

$$Q = UA(T_S - T_R) \quad (3.10)$$

The heat transfer dynamics are composed of dynamics of the temperature on the methanol-water side of the reboiler, combined with steam side temperature dynamics. The methanol-water solution temperature is a direct function of solution composition

$$T_R = \phi(x_R) \quad (3.11)$$

so temperature dynamics are identical to composition dynamics. Reboiler composition dynamics are included in the model as for any other stage in the distillation column, by calculating a component balance. The reboiler equation is

$$WT_R \frac{dx_R}{dt} = L_2 x_2 - L_1 x_1 - V_1 y_1 \quad (3.12)$$

By a total mass balance, and assuming constant (approximately) reboiler holdup by level control, the following equation

$$L_2 = L_1 + V_1$$

indicates that the reboiler time constant or reboiler residence time would be

$$\tau_R = \frac{WT_R}{L_2}$$

This time constant may be shown to be several orders of magnitude larger than the time constant on the steam side of the reboiler by examining a steam mass balance.

$$\frac{dWT_S}{dt} = S_i - S_o$$

Since there is condensate level control,

$$\frac{dWT_S}{dt} \Rightarrow 0 \quad \text{and} \quad S_i = S_o$$

The energy balance on the steam side enables solution for the steam temperature, and is expressed in equation (3.13), neglecting steam vapor holdup terms.

$$\frac{dWT_S h_S}{dt} = S_i H_{Si} - S_o h_S - Q \quad (3.13)$$

Substitution of equation (3.10) (and $S_i = S_o$) gives

$$WT_S \frac{dh_S}{dt} = S_i (H_{Si} - h_S) - UA(T_S - T_R)$$

The enthalpy of the saturated condensate may be approximated by a linear function of temperature.

$$h_S = A + BT_S$$

Substituting and gathering terms in T_S yields

$$BWT_S \frac{dT_S}{dt} = S_i (H_{Si} - A - BT_S) - UA(T_S - T_R)$$

or rearranging

$$\frac{BWT_S}{(S_i B + UA)} \frac{dT_S}{dt} + T_S = \frac{S_i (H_{Si} - A) + UA \cdot T_R}{(S_i B + UA)} \quad (3.14)$$

Expression (3.14) represents a first order system with thermal time constant

$$\tau_S = \frac{BWT_S}{S_i B + UA}$$

which when compared to the reboiler composition time constant

$$\tau_R = \frac{WT_R}{L_2}$$

is found to be several orders of magnitude smaller.

Equation (3.14) could be integrated to solve for changes in T_S , but in this work, due to the significant difference in the magnitude of the time constants, the steam temperature was assumed to reach its steady state temperature instantaneously. Neglecting the transient term in equation (3.14) yields the following equation for steam temperature.

$$T_S = \frac{S_i (H_{Si} - A) + UA \cdot T_R}{S_i B + UA} \quad (3.15)$$

To calculate the heat input at any instant, the steam temperature was calculated using equation (3.15) and the reboiler solution temperature determined from the reboiler composition. These two temperatures were then used in equation (3.10) along with the overall heat transfer coefficient to calculate the heat input Q .

3.1.3 The Condenser Model

Liquid level control in the condenser operates with a hydrostatic head type of measurement rather than direct measurement of level, so either a density change due to a

change in composition of the fluid or an actual level change, will cause a deviation from the level controller setpoint. Since mass holdup and not necessarily level is being maintained constant, the total mass balance will not contain unsteady state mass accumulation terms. On this basis, the mass balance may be expressed as

$$V_9 = R + D \quad (3.16)$$

The composition balance for the condenser may be written as

$$WT_{10} \frac{dx_{10}}{dt} = V_9 Y_9 - (R + D) x_{10}$$

or

$$WT_{10} \frac{dx_{10}}{dt} = V_9 Y_9 - V_9 x_{10} \quad (3.17)$$

Equation (3.17) was used directly in the integration step of the model solution, to give the top product composition. The reflux and distillate streams are at the same composition, feeding from the top product reservoir.

The amount of heat removal from the condenser was not included in the model, but the energy balance degrees of freedom were defined by specifying the enthalpies of all streams entering and leaving the column and by specifying heat input to the reboiler. The enthalpy of the reflux entering the column was taken to be the enthalpy of saturated

liquid at the top product composition, in the model, and was temperature controlled at close to saturated conditions, on the actual column.

3.1.4 Variable Holdup

Several correlations for flow over weirs and bubble cap tray holdups have been presented in the literature as discussed in Chapter 2. As will be shown in Chapter 5, comparison of predicted holdups with experimentally measured holdups from the distillation column used in this study show significant differences, with the measured values being lower in magnitude by as much as one half.

Attempts were made to correlate the experimentally determined holdups at different steady state conditions with liquid and vapor flow rates predicted by the constant holdup mathematical model developed in this work, but due to the scatter of data (see Appendix (F), Figure (5.10)) the correlations were not of value. However, the data did show that when plotting measured tray holdups against column feed rate, a large fraction of the total column holdup occurred below the feed tray, and furthermore, when the feed rate was increased, this proportion increased.

For these reasons, correlations presented in the literature were modified to give holdups in the same range as

measured values. The equations were of the form

$$WT_n = a_n L_n + b_n V_n + c_n$$

and the modifications were to the magnitude of the parameters a_n , b_n and c_n .

Three correlations were used in simulations in this work: in two of the three, the parameters a_n , b_n and c_n were constant from tray to tray and were not changed with time. The third correlation (Francis Weir) used parameters that varied with density changes.

Two alternate methods of allowing for variable tray holdup were attempted in this work, and are described in the "Problem Formulation" section, which follows.

3.2 PROBLEM FORMULATION

Two solution schemes to solve the $4n$ simultaneous equations were formulated. As previously mentioned, $4n$ unknowns, n compositions, n liquid flow rates, n vapor flow rates, and n tray holdups need to be specified to completely define the column dynamic state at any time. These variables must be determined in some way from the composition balance equations, total mass balance equations, energy balance equations and holdup correlations. The sequence in which these equations are solved together to define the column's state, is termed

the "problem formulation" in this work.

3.2.1 Substitution Method

3.2.1.1 Description

In the substitution method, only the composition balances were integrated; the other differential equations were treated as algebraic equations with their differential terms calculated as finite differences. Composition balance integration yielded individual tray compositions. The coefficients of the liquid and vapor composition terms were evaluated at the beginning of the time step between integration iterations. The equations integrated were arranged as follows.

$$\begin{aligned} \frac{dx_n}{dt} = \frac{1}{WT_n} (L_{n+1}x_{n+1} - L_nx_n + V_{n-1}y_{n-1} + F_nx_{Fn} \\ - V_ny_n - S_nx_n - \frac{dWT_n}{dt} \cdot x_n) \end{aligned} \quad (3.18)$$

To solve for the liquid and vapor flow rates, the enthalpy balances and total balances were solved simultaneously.

Total mass balances are given by equation (3.1)

$$\frac{dWT_n}{dt} = L_{n+1} - L_n - V_n + V_{n-1} - S_n + F_n$$

where the derivative term is evaluated from holdups of the previous time step, from a finite difference representation.

$$\frac{dWT_n^K}{dt} \approx (WT_n^K - WT_n^{K-1})/DT \quad (3.19)$$

The energy balances were rearranged to the following form

$$\begin{aligned} WT_n \frac{dh_n}{dt} + S_n h_n + h_n \frac{dWT_n}{dt} \\ = L_{n+1} h_{n+1} - L_n h_n - V_n H_n + V_{n-1} H_{n-1} \end{aligned} \quad (3.20)$$

with the enthalpy difference calculated from the finite difference representation.

$$\frac{dh_n^K}{dt} \approx (h_n^K - h_n^{K-1})/DT \quad (3.21)$$

The set of equations (3.1), (3.20) were solved by substitution for the liquid and vapor flow rates. Substituting equations (3.1) into equations (3.20), starting at stage 9, eliminates all the liquid flows, except the known reflux flow (L_{10}), so the resulting equations, in vapor flow only, may again be substituted into each other. Upon this, one equation in one vapor flow is obtained, and this equation is solved. Back substitution then gives all liquid and vapor flows for all stages. A detailed step by step procedure with detailed equations is included in Appendix (C).

Accompanying the change in liquid and vapor flows is a change in tray holdup, established from equations (3.4). As liquid accumulating on the tray has the enthalpy of the

liquid leaving the tray, all accumulation comes from the existing liquid for the enthalpy balance to be satisfied. This correction must be made using an iterative technique; holdups are calculated based on new liquid and vapor flows, accumulation rates are calculated with equation (3.21), and the exiting liquid flow is corrected for accumulation. A holdup is again calculated based on the corrected flow, and the process is continued until the difference between successive holdup calculations falls within some error criterion.

Underlying the entire solution method is the condition that the holdup derivative, upon which the calculations of current liquid and vapor flows and holdups are based, is calculated from the previous time step (equation 3.21). Since the holdup derivative is defined based on holdup values at the K and $K-1$ time instants, and is used to calculate flow rates and holdups at time step $K+1$, this naturally leads to some inaccuracies. This is overcome by using the matrix inversion method, which will be considered later in this chapter.

3.2.1.2 Stability

Numerical stability of this solution scheme can be divided into two categories, namely: the stability of the integration and the stability of the coefficient determination.

Once the problem has been formulated into a format such as

$$\underline{x}^{K+1} = \underline{A} \underline{x}^K \quad (3.22)$$

there are many standard methods to evaluate stability and obtain a stable integration for a variety of $\underline{A}$ matrices. The primary obstacle in modelling systems is to formulate the problem into a standard format. For distillation systems, this matrix is near ideal, strongly diagonally dominant, with well conditioned eigenvalues, such that a simple integration scheme such as Euler or Modified Euler will suffice for a stable solution. A detailed description of the components of this matrix, which is important in the matrix inversion method of solution, can be found in section 3.2.2.2. Furthermore, this matrix represents the composition system of the distillation column, whose eigenvalues are inherently smaller than those of the hydraulic system, which is essentially the coefficient determination system.

Although the solution for the A matrix was obtained by solving a series of algebraic equations, in essence, integrations were performed here also. However, the equations could not be formulated into a standard form expressed as

$$\underline{A}^{K+1} = f(\underline{x}^{K+1}, \underline{A}^K) \quad (3.23)$$

so only an ad hoc stability analysis could be done. The

dependence of the new value of A upon the old value comes through the enthalpy, holdup and composition derivative terms in the equations. If it were not for the inclusion of the holdup derivative, which is independent from the concentration derivative, stability of the coefficient determination step would be identical to that of the integration step, as all differential terms would be linear functions of the concentration derivative.

The solution with variable holdup resulted in a reduction in stability from the constant holdup case. It was found, that for the variable holdup case, a time step approximately 60 times smaller than for the constant holdup case was necessary for a stable solution.

The change in time step size was considered due to two effects: firstly, as previously mentioned, a relatively poor estimation of the holdup derivative was used, as only old holdup values were available for its calculation. The error incurred was necessarily reduced by using a smaller step size. Secondly, the hydraulic system, to which the tray holdups are directly related is a much faster system, as compared to the concentration system, with relatively shorter time constants. In light of this, smaller integration time steps would be expected.

The necessity of employing a very small integration

Interval provided the incentive for restructuring the formulation leading to the matrix inversion method that is considered next.

3.2.2 The Matrix Inversion Method

3.2.2.1 Description

In order to reduce solution time due to the small time increment, it was decided that a better estimate of the average tray holdup rate of change, over the integration interval, was required. An exact expression was available in the total mass balance (equation 3.1).

$$\frac{dWT_n}{dt} = L_{n+1} - L_n - V_n + V_{n-1} - S_n + F_n$$

This set of equations was integrated directly in an integration step, along with the component balances (3.2), to provide trajectories for tray compositions and tray holdups.

The vapor and liquid flow rates were again calculated in a coefficient determination step. The energy balance equations and holdup correlations were rearranged and used for this purpose. The energy balance (3.3) may be rewritten

$$h_n \frac{dWT_n}{dt} + WT_n \frac{dh_n}{dt} = L_{n+1} h_{n+1} - L_n h_n - V_n H_n + V_{n-1} H_{n-1} + F_n h_{Fn} - S_n h_n - Q \quad (3.24)$$

Replacing the rate of change of holdup using equation (3.1) and rearranging yields the equations (3.25) in the required

format

$$WT_n \frac{dh_n}{dt} + Q + S_n h_n - F_n h_{Fn} = (h_{n+1} - h_n) L_{n+1} + (h_n - H_n) V_n + (H_{n-1} - h_n) V_{n-1} \quad (3.25)$$

As opposed to the substitution method, this method uses a current value of the holdup derivative based on current values of liquid and vapor flows. By substitution into both the enthalpy balance and composition balance, the holdup derivative and liquid flow terms for a tray were eliminated from the corresponding equation (see equation 3.25). A detailed derivation is included in Appendix (C).

The holdup correlations were also rearranged (equation 3.4)

$$WT_n - c_n = a_n L_n + b_n V_n$$

When equations (3.25) are combined with equations (3.4), a matrix equation is the result,

$$\underline{FT} = \underline{TT} \begin{bmatrix} \underline{L} \\ \underline{V} \end{bmatrix} \quad (3.26)$$

where FT is the vector comprised of the left hand sides of the above mentioned equations, and TT is a matrix of coefficients which are enthalpy differences and holdup correlation parameters.

To solve for liquid and vapor flow rates, the vector $\underline{FT}$ was updated for changes in holdup, and for changes in enthalpy accumulation. Inversion of matrix $\underline{TT}$, and taking the product of the inverse and the $\underline{FT}$ vector permitted calculation of the mass flow rates from

$$\begin{bmatrix} \underline{L} \\ \underline{V} \end{bmatrix} = \underline{TT}^{-1} \underline{FT} \quad (3.27)$$

Thus, in one time step, new liquid and vapor flows were calculated, based on current compositions and holdups. Then via integration, new compositions and holdups were determined from the new mass flow rates, that is

$$\underline{x}^{K-1}, \underline{WT}^{K-1} \rightarrow \underline{L}^K, \underline{V}^K \quad (3.28)$$

$$\underline{x}^{K-1}, \underline{WT}^{K-1}, \underline{L}^K, \underline{V}^K \rightarrow \underline{x}^K, \underline{WT}^K$$

The integration step included the composition integration, according to the following expression derived in Appendix (C).

$$\underline{x}^K = (\underline{I} - \underline{DT}/2 \underline{A}^K)^{-1} (\underline{I} + \underline{DT}/2 \underline{A}^K) \underline{x}^{K-1} \quad (3.29)$$

To calculate new holdups, the first order Euler integration was used

$$\underline{WT}_n^K = \underline{WT}_n^{K-1} + \underline{DT} \left(\frac{d\underline{WT}_n}{dt} \right)^{K-1} \quad (3.30)$$

3.2.2.2 Stability

Due to the nature of the equations, rigorous stability analyses could not be made. For the integration step, but not for the coefficient determination, the equation's format was

$$\dot{\underline{x}} = \underline{A} \underline{x} \quad (3.31)$$

to which standard methods could be applied. For the integration step, the A matrix was formed as a tridiagonal matrix, its derivation being presented in Appendix (C). However, as will be shown later, it was found that the stability of the integration step, as in the substitution method, was not the controlling factor. Rather, the coefficient determination step, viewed as representing the hydraulics, limited the size of the integration interval.

It was found that using the identical holdup correlation used in the substitution method, that for the Matrix Inversion method, a time step of approximately 3 to 4 times larger could be used to obtain a stable solution. However, this time step was very dependent upon the liquid coefficient " a_n " in the holdup correlation (3.4), with a larger "a" permitting a larger time step to be used. The reason for this was evident upon inspection of the equations.

$$WT_n - c_n = a_n L_n + b_n V_n$$

A small change in holdup would change the liquid or vapor flows in order to satisfy the holdup correlation. As the enthalpy balance is also satisfied, and since liquid enthalpies are lower than those of vapor by approximately a factor of 10, the liquid flow rate would absorb most of the change. So, for example, if " a_n " were small, a change on the left hand side of equation (3.4) would cause a relatively large change in liquid flow. This change, over an integration interval, must for stability be limited by using a smaller time step.

All of the results and comparisons shown in Chapter 5 were obtained by the more stable matrix inversion method.

3.3 SIMPLIFYING ASSUMPTIONS

To compare the response of the rigorous model with dynamic responses of models based on additional assumptions, changes to the detailed model as described in the following sections, were introduced. The matrix inversion method of solution, suitably modified for the less rigorous models, was employed, as it is simpler, more straightforward and faster than the substitution method.

3.3.1 Constant Holdup

With the assumption of constant holdup, the holdup correlation equations are eliminated from the solution scheme,

and the total mass balances, equation (3.1), formerly differential equations become algebraic

$$L_{n+1} - L_n - V_n + V_{n-1} - S_n + F_n = K_n \quad (3.32)$$

Two cases of constant holdup may be assumed, constant volume holdup and constant mass holdup. For each, the right side of equation (3.32) assumes a different value. In the former case of constant volume holdup, mass accumulation will be due to variation in composition and thus a density change. As density is a linear function of composition, the following expression holds.

$$K_n = c \frac{dx_n}{dt} \quad (3.33)$$

For the assumption of constant mass holdup, it follows that

$$K_n = 0 \quad (3.34)$$

In this work, only the case of constant mass holdup was considered. Changes to implement this assumption were made to both the integration and coefficient determination systems. The integration of the total mass balance equations in the integration section was dropped. These equations were used in place of the holdup correlation equations in the solution of liquid and vapor flows. The matrix "TT" and vector "FT" from equation (3.26) were changed accordingly. Apart from these changes, the solution in each time step was carried out as for the variable holdup model.

3.3.2 Constant Liquid and Vapor Rates

The variable holdup model may be further simplified by assuming that the liquid and vapor rates do not deviate greatly from their steady state values for feed, reflux or steam flow disturbances of moderate magnitude. As holdup is a function of liquid and vapor rates, constant holdup must also be assumed.

These simplifications, which lead to a reduction of order from forty states to ten, reduces computation time, as only the integration of equations (3.31) remains. Also, matrix A remains constant except for changes in the disturbance variables.

3.3.3 Constant Molar Overflow

If the molar heats of vaporization of the two components are nearly equal, a mole of vapor condensing will cause a mole of liquid to vaporize. If heat losses from the column are neglected, then flow rates, both vapor and liquid, will be uniform throughout the stripping section and again throughout the rectifying section. The difference between the flows in the rectifying section and stripping section will be solely due to the feed flow, which is introduced between the two sections. This assumption eliminates the need for an energy balance for each tray.

Vapor dynamics were taken to be instantaneous resulting from the assumption of constant vapor holdup. Liquid dynamics, however, were handled by including the holdup correlations in the solution scheme.

Modifications to the matrix inversion solution method were as follows. In the energy balances, the vapor and liquid enthalpies, the coefficients for the vapor and liquid flow terms, respectively, were taken to be fixed values, for a composition of 50 percent MeOH. This resulted in using constant molar heats of vaporization from tray to tray. Heat input to the reboiler was calculated as before, and largely determines the vapor rate throughout the column. The holdup correlations, total and component balances were included in the solution scheme as before.

3.3.4 Adiabatic Operation

In distillation columns, there is heat loss from the column to the atmosphere by radiation and convection. A further simplifying assumption that may be employed, is the neglect of heat loss from the column. Mathematically, this results in dropping the heat loss (Q term) from the energy balance equation (3.3) yielding

$$\begin{aligned}
 W T_n \frac{dh_n}{dt} + h_n \frac{dW}{dt} &= L_{n+1} h_{n+1} - L_n h_n \\
 &- V_n H_n + V_{n-1} H_{n-1} - S_n h_n + F_n h_{Fn}
 \end{aligned}
 \tag{3.35}$$

For columns housed within buildings such as the one at the University of Alberta, heat loss is largely a function of column wall temperature. A significant portion of the total heat loss from the column was attributed to the gas chromatograph sampling system, where a long length of metal tubing, through which hot reboiler liquid was cycled, was exposed. This was a loss directly from the reboiler, amounting to approximately three to four percent of the total heat input to the column. Heat loss from the remainder of the column made up the balance, which amounted to about three to four percent of the heat input.

In the simulations, an identical heat loss for each tray was employed, with the loss selected so that the terminal compositions matched the experimental values at the initial steady state. When adiabatic operation was assumed, initial steady state conditions could not be matched, indicating the severity of this assumption.

3.3.5 Variable Heat Loss and Tray Efficiency

As a distillation column experiences upsets affecting liquid and vapor compositions on the stages, the temperatures

of the stages and thus individual tray heat losses are also affected. In the previous section, the assumption of adiabatic operation was described. An alternate assumption, rather than consider the heat loss to be constant, is to allow for a heat loss that varies with operating conditions.

The constant heat loss assumption would mathematically imply that the Q term in the energy balance, equation (3.3) is taken at a constant value over the course of the column response to a disturbance, that is

$$Q_t = Q_i \quad (3.36)$$

Rather than use a constant heat loss value, heat loss was varied directly with composition. Equation (3.37) was used to vary the heat loss with composition changes according to

$$\frac{Q_t - Q_i}{Q_f - Q_i} = \frac{x_t - x_i}{x_f - x_i} \quad (3.37)$$

where x_t is a composition of a chosen stage in the column at time t . In this work, the chosen stages were taken as the feed tray, the reboiler and the condenser. The heat loss at the initial and final steady state conditions were chosen to match the experimental steady state conditions.

Tray efficiency, like heat loss, was also allowed to vary with composition changes. Values were selected to match

experimental and simulated initial and final steady state terminal compositions. Efficiency was varied, in a manner directly analogous to that for heat loss, according to

$$\frac{E_t - E_i}{E_f - E_i} = \frac{x_t - x_i}{x_f - x_i} \quad (3.38)$$

This equation was used for both the stripping and rectifying sections, as efficiencies were taken to be uniform through a section, but different for the two sections.

3.4 NUMERICAL METHODS

Several different methods were used to integrate the composition balance equations. These equations were integrated independently in the substitution method, and as a matrix system in the matrix inversion method.

3.4.1 The Substitution Method

Equation (3.18) was expressed as below for the integration purposes for any plate other than the feed plate and with no sidestream withdrawal.

$$\frac{dx_n}{dt} = \frac{1}{WT_n} (L_{n+1}x_{n+1} - L_nx_n - V_ny_n + V_{n-1}y_{n-1} - x_n \frac{dWT_n}{dt}) \quad (3.39)$$

For the feed plate, (stage 5) the equation became

$$\frac{dx_5}{dt} = \frac{1}{WT_5} (L_6 x_6 - L_5 x_5 - V_5 y_5 + V_4 y_4 + F x_F - x_5 \frac{dWT_5}{dt}) \quad (3.40)$$

The equation for the reboiler (stage 1) became

$$\frac{dx_R}{dt} = \frac{1}{WT_R} (L_2 x_2 - L_1 x_R - V_1 y_1) \quad (3.41)$$

The equation for the condenser (stage 10) is

$$\frac{dx_{10}}{dt} = \frac{1}{WT_{10}} (V_9 (y_9 - x_{10})) \quad (3.42)$$

At any time instant, K , the general vector-matrix representation may be expressed as

$$\frac{dx_n^K}{dt} = f_n^K \quad (3.43)$$

The Euler method is summarized by equations (3.44) and (3.45)

$$\frac{dx_n^K}{dt} \approx (x_n^{K+1} - x_n^K) / DT \quad (3.44)$$

rearranging

$$x_n^{K+1} = x_n^K + DT(f_n^K) \quad (3.45)$$

In this simple integration scheme, equation (3.44), an elemental but poor approximation of the actual derivative, is employed. In calculating a transient, an error is incurred

at each time step, each error being propagated over the rest of the calculation. The modified Euler integration method offers a slight improvement over the simple Euler integration in the estimation of the derivative, and allows larger integration time intervals to be used, at the expense of more computation.

$$\frac{dx_{av}^{K+1}}{dt} = \left(\frac{dx^K}{dt} + \frac{dx^{K+1}}{dt} \right) / 2 \quad (3.46)$$

The derivatives on the right hand side of equation (3.46) evaluated at times K and K+1 are calculated using the Euler technique after which an average derivative over the time interval is calculated and used for the integration interval.

As a further attempt to better estimate the value of the composition derivative, third and fourth order Runge-Kutta techniques were used; they are outlined below. Referring to the f function described in equation (3.43), let

$$\frac{dx_n^K}{dt} = f_n(x_n^K) \quad (3.47)$$

and let

$$\begin{aligned} q_{1n} &= DT \cdot f_n(x^K) \\ q_{2n} &= DT \cdot f_n\left(x^K + \frac{1}{2} q_{1n}\right) \\ q_{3n} &= DT \cdot f_n\left(x^K + \frac{1}{2} q_{2n}\right) \\ q_{4n} &= DT \cdot f_n\left(x^K + q_{3n}\right) \end{aligned}$$

then

$$x_n^{K+1} = x_n^K + \frac{1}{6}(q_{1n} + 2q_{2n} + 2q_{3n} + q_{4n})$$

Note that equation (3.47) does not include time as an independent variable, and thus time does not enter into the fourth order Runge-Kutta solution described above, as a variable.

The more complex integration schemes such as the Runge-Kutta methods allowed the integration time interval to be increased, but involved increased computation. But, as mentioned in sections 3.2.1.2 and 3.2.2.2, the integration steps in the model solutions were not integration interval size limiting; rather, it was the step of calculating vapor and liquid flow rates and liquid holdups that forced the use of the small integration step size. The integration method, therefore was not crucial, and it was found that the simple Euler method served as well as the more complex techniques.

3.4.2 Matrix Inversion Method

As outlined in section 3.2.2, the matrix inversion formulation dealt with the integration as a system of equations expressed in matrix form as

$$\frac{dx}{dt} = \underline{A} \underline{x}$$

Use of the Euler method for integrating the system equations expressed in matrix form as given above yields

$$\begin{aligned} (\underline{x}^{K+1} - \underline{x}^K) / DT &= \underline{A}^K \underline{x}^K \\ \underline{x}^{K+1} &= (\underline{I} + DT \underline{A}^K) \underline{x}^K \end{aligned} \quad (3.48)$$

Also employed was the modified Euler method leading to

$$\begin{aligned} \frac{d\underline{x}^K}{dt} &= \underline{A}^K \left(\frac{\underline{x}^{K+1} + \underline{x}^K}{2} \right) \\ \underline{x}^{K+1} &= (\underline{I} - DT \underline{A}^K)^{-1} (\underline{I} + DT \underline{A}^K) \underline{x}^K \end{aligned} \quad (3.49)$$

The derivation is given in Appendix C.

CHAPTER 4

SIMULATION RESULTS AND DISCUSSION

4.1 RESULTS - INTRODUCTION

Results of distillation column simulations demonstrating the effects of various simplifications and assumptions are presented. The basic model for the ten stage binary column was chosen to the 30th order, in which the total mass, energy and component balances were solved simultaneously. To allow for a variable holdup, introduction of holdup correlation equations into the solution resulted in a 40th order model. Results for three cases of this model are presented in this chapter. The responses of various different models of lower complexity relative to the basic model are also presented for comparison. These are, in order of increasing complexity, the constant flow rate model, the constant molal overflow model, and the constant efficiency and heat loss model. In addition to the simulations for the basic model, for which the parameters of tray efficiency and heat loss are taken as functions of distillate composition, results are also presented for the case where the parameters are functions of feed tray composition and bottoms product composition. Finally, simulated results obtained including as heat sinks the tray metal and glass in the basic model are also presented.

The figures displayed in this chapter enable comparisons

of the effects of various assumptions upon distillation column dynamic responses. These responses further demonstrate the robust nature of the solution scheme outlined in Chapter 3, and its applicability under a variety of conditions.

A complete summary of the simulation results is presented in Table 4.1, along with an explanation of the codes used in Figures 4.1 to 4.12 inclusive.

Results are presented for step disturbances of -20 percent in feed and reflux flow rate and +10 percent in steam flow rate. Only the disturbances of reflux and steam were chosen with which to do complete comparisons for reasons of time and expense. These disturbances were chosen since it was found, as will be shown in Chapter 5, that these two disturbances exhibited the largest discrepancies between experimental and simulated responses. Furthermore, in many previous modelling studies only disturbances in feed composition or feed flow were considered.

The model using constant holdup, variable efficiency and heat loss (the 04 series, Table 4.1) is considered as the reference (base) model for purposes of comparison to predicted responses. This is a more general model than that of Huckaba et al.⁸ which is considered a general model, representing data well. Responses calculated from models of both increasing or decreasing complexity are compared with the base case responses.

TABLE 4.1

SUMMARY OF SIMULATIONS

I Reflux Disturbance
(Minus 20 percent)

Run Code	Assumptions	Figure
RO1M	1,3,12,13	4.4
RO2M	2,3,12,13	4.4
RO3M	3,12,13	4.4
RO4M	4,12,13	4.4
RO5M	5,12,13	4.6
RO6M	6,12,13	4.6
RO7M	4,7	4.9
RO8M	4,12	4.6
RO9M	4,9	4.10
RO10M	4,8	4.10
RO11M	4,12,13	4.9

II Steam Disturbance
(Plus 10 percent)

Run Code	Assumptions	Figure
SO1P	1,3,12,13	4.5
SO2P	2,3,12,13	4.5
SO3P	3,12,13	4.5
SO4P	4,12,13	4.5
SO5P	5,12	4.7
SO6P	6,12	4.7
SO7P	4,7	4.11
SO8P	4,12	4.8
SO9P	4,9	4.11
SO10P	4,8	4.11
SO12P	4,12,11	4.12
SO13P	4,12,10	4.12

III Reflux Disturbance
(Plus 20 percent)

Run Code	Assumptions	Figure
RO3P	3,12,13	4.2
RO4P	4,12,13	4.2

IV Feed Disturbance (Flow)
(Plus 20 percent)

Run Code	Assumptions	Figure
FO3P	3,12,13	4.1
FO4P	4,12,13	4.1

V Steam Disturbance
(Minus 10 percent)

Run Code	Assumptions	Figure
SO3M	3,12,13	4.3
SO4M	4,12,13	4.3

TABLE 4.2
LIST OF ASSUMPTIONS

- 1 - constant molal overflow
- 2 - constant liquid and vapor rates
- 3 - constant efficiency and heat loss
- 4 - variable efficiency and heat loss: based on top composition
- 5 - variable efficiency and heat loss: based on bottom composition
- 6 - variable efficiency and heat loss: based on feed tray composition
- 7 - variable holdup: arbitrary correlation
- 8 - variable holdup: RADEMAKER correlation
- 9 - variable holdup: FRANCIS WIER correlation
- 10 - subcooled reflux
- 11 - subcooled feed
- 12 - constant tray liquid mass holdup
- 13 - negligible heat capacity of tray glass, metal

4.2 SIMPLIFYING ASSUMPTIONS

4.2.1 Constant Efficiency, Constant Heat Loss

As mentioned in Section 3.4.4, the parameters of tray efficiency and tray heat loss were used to fit simulated responses to experimental data. Figures 4.1 to 4.3 present a comparison of predicted responses obtained by assuming constant tray heat loss and constant tray efficiency (the 03 series) with responses obtained by allowing for changes in heat loss and efficiency (the 04 series) for feed, steam and reflux disturbances. It can be seen that in the constant parameter case, the responses exhibit the same general dynamic behaviour as in the variable parameter case, but a comparison of response gains shows far greater gain values for the case of constant parameters.

This result is to be expected. Heat loss, which varies with tray temperature (or composition), varies in such a manner to reduce the effects of a disturbance; as a disturbance causes column temperatures to rise, heat loss rises to compensate, tending to reduce the effects of the disturbance. The same is true for disturbances causing temperature reductions. However, correlations relating efficiency to column parameters indicate a dependence upon flows and holdup; changes in these variables possibly do not have a damping effect. Heat loss, however, has been found to have much more effect on column operation than efficiency, and it is mainly

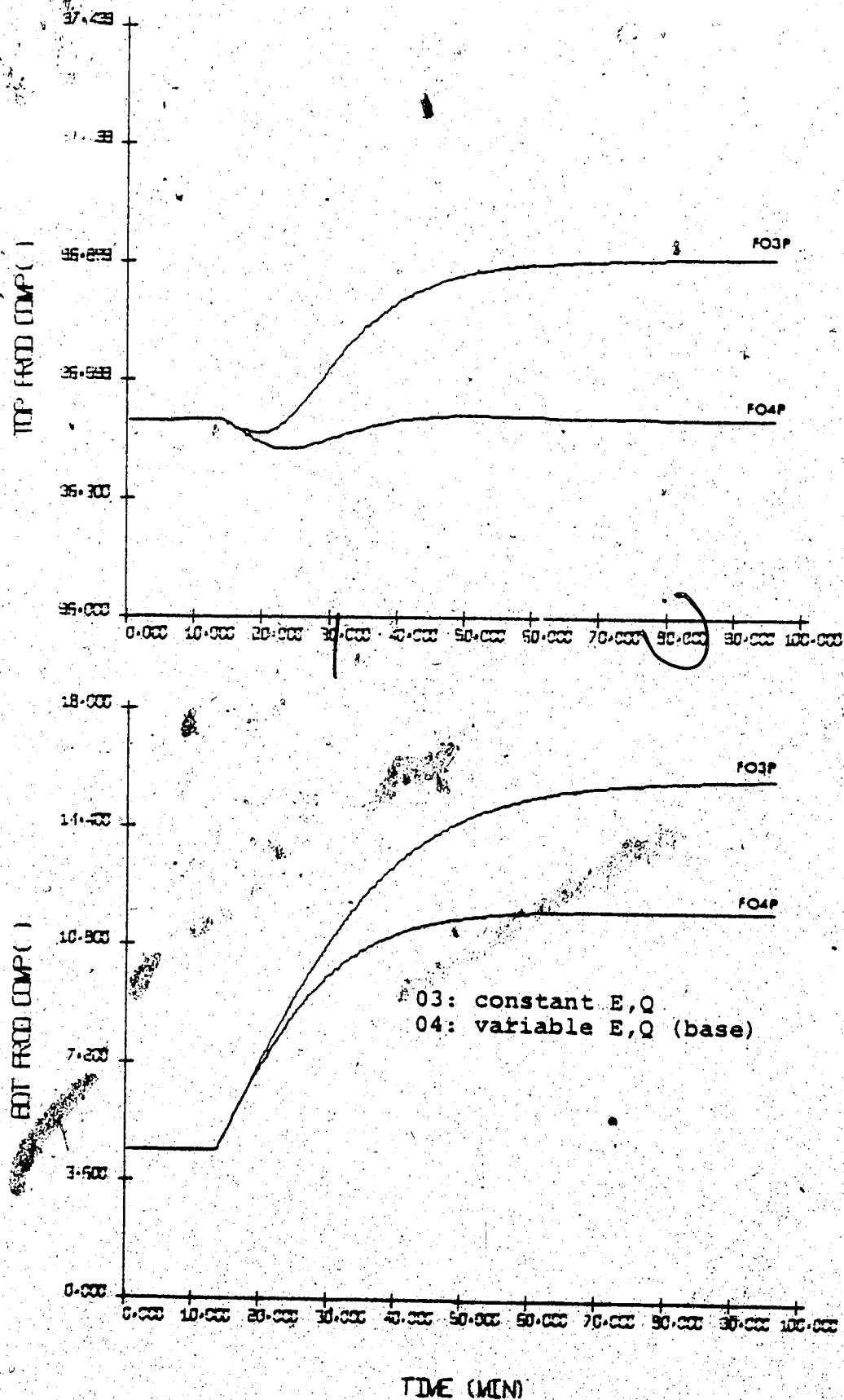


FIGURE 4.1 +20% Feed: Effects of the assumption of constant heat loss and efficiency.

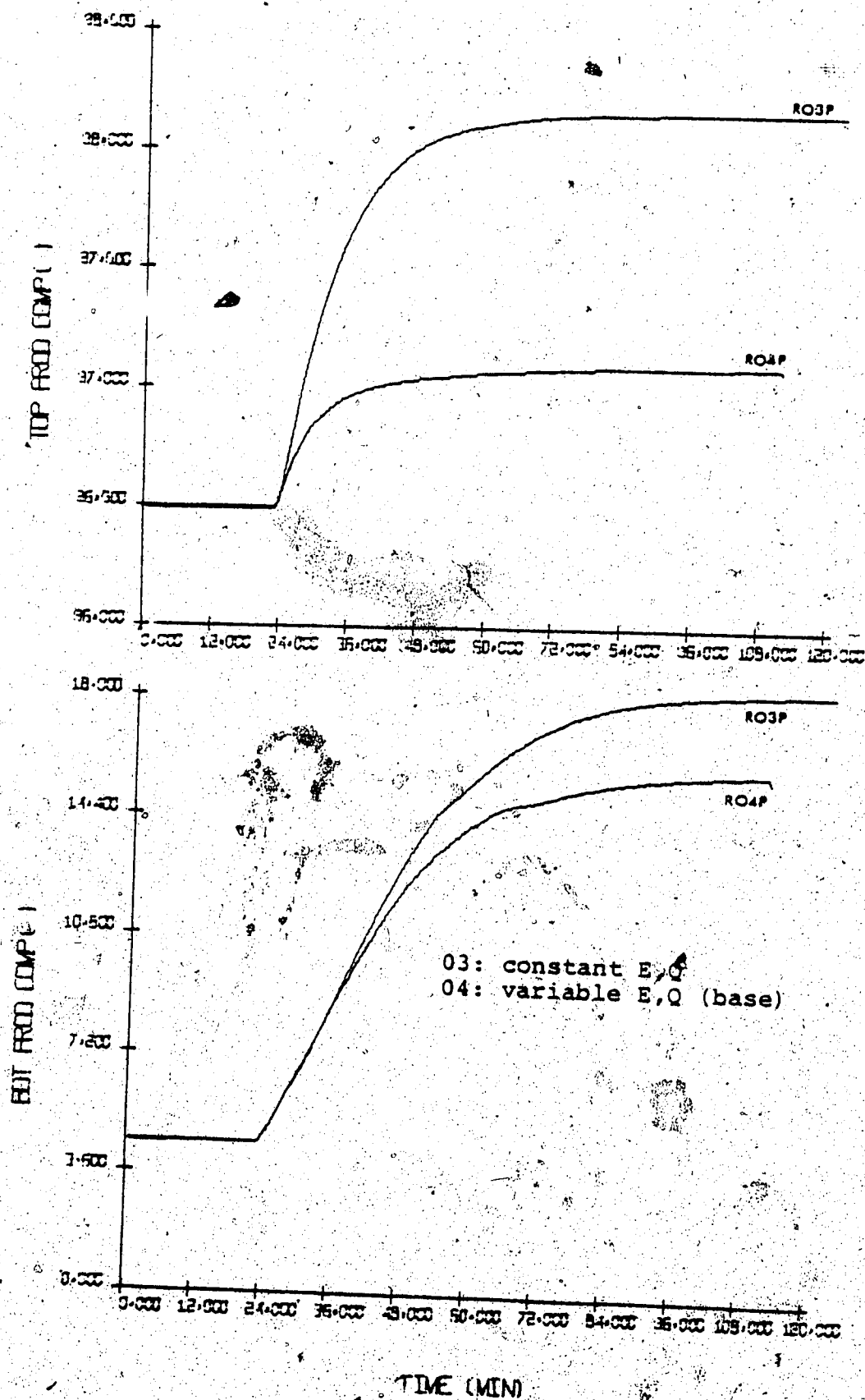


FIGURE 4.2 +20% Reflux: Effects of the assumption of constant heat loss and efficiency

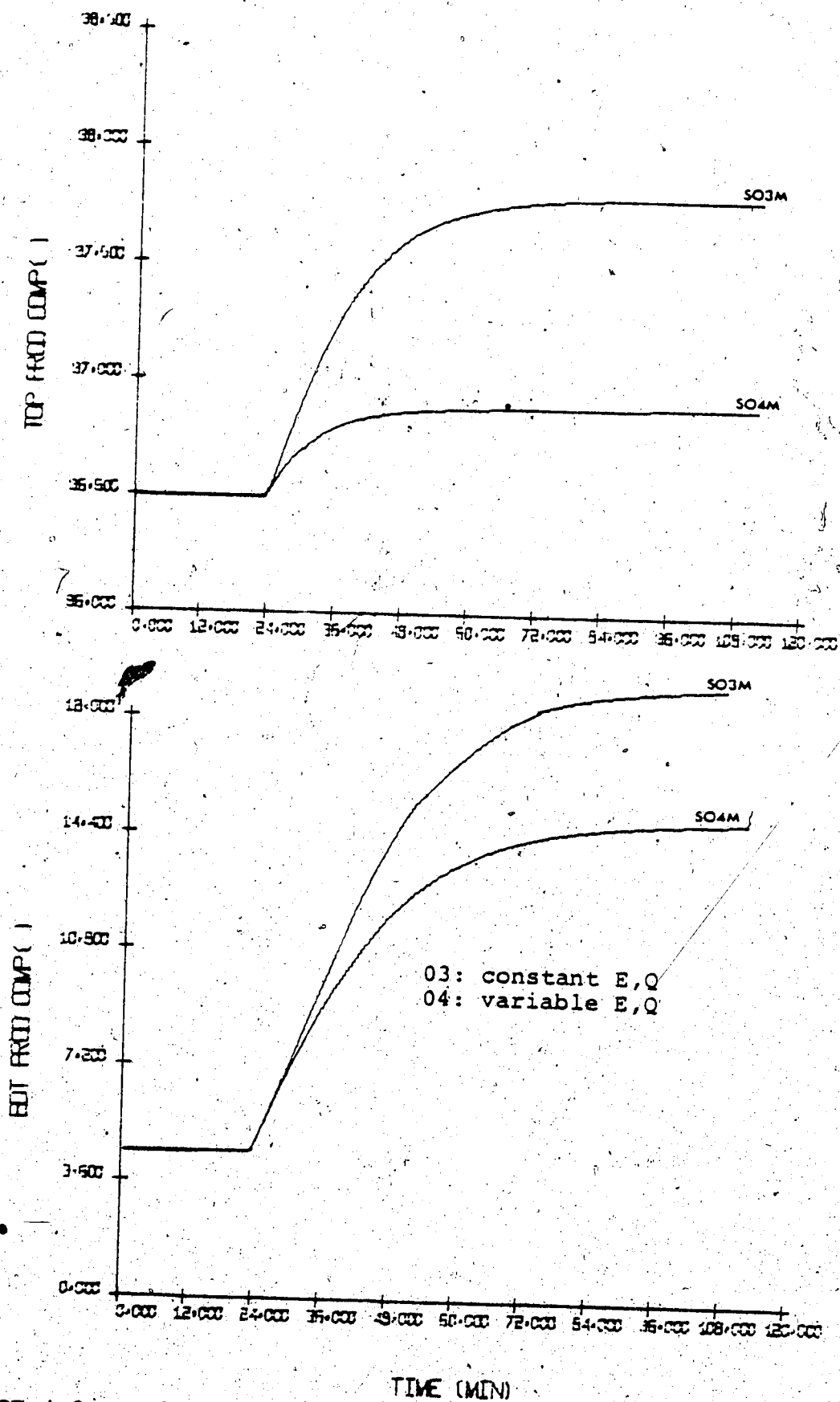


FIGURE 4.3 -10% Steam: Effects of the assumption of constant heat loss and efficiency.

the damping effect of the varying heat loss that accounts for the gain differences observed.

That the dynamics are unaffected may also be expected. Heat loss and efficiency have negligible dynamics of their own, but vary directly with column parameters such as flow and temperature. In all cases, the temperature (or composition) dynamics are slower, and thus largely determine response rates.

4.2.2 Constant Flow Profile

The assumption of constant flow profile is a severe assumption, causing liquid and vapor rates to be, not time dependent, but different from tray to tray. The effect of this assumption may be observed for the product composition responses for reflux and steam disturbances shown in Figures 4.4 and 4.5 respectively; as the 02 series.

The constant flow profile model predicts responses with lower gain and slower dynamics for both steam and reflux upsets. Such disturbances to a column cause compounded effects of flow, composition and energy. Since in the solution scheme of this model, the tray energy balance and total tray mass balance equations do not need to be solved, the resulting effect is to reduce the magnitude of the simulated column upset as shown. The error due to this simplification appears

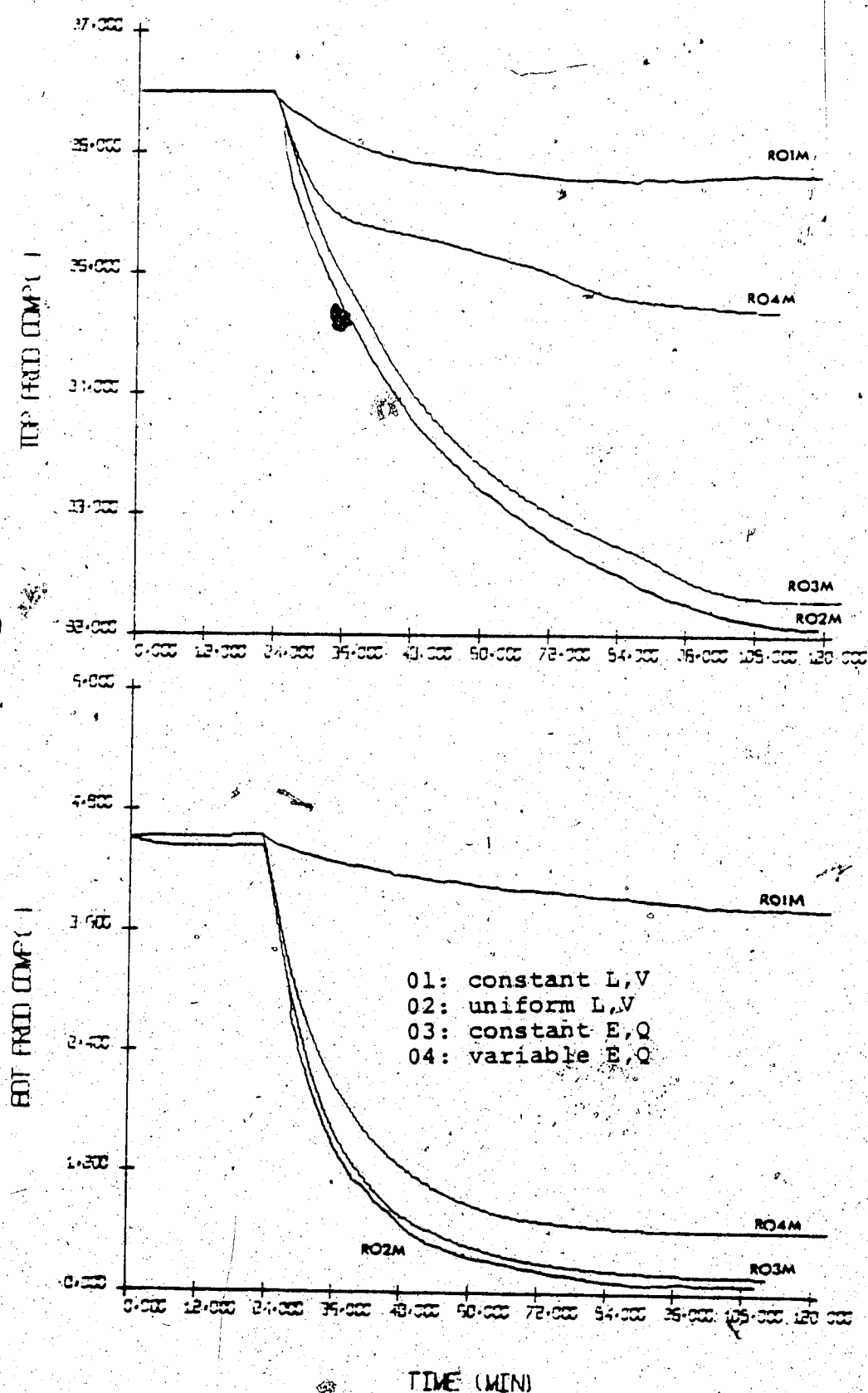


FIGURE 4.4 -20% Reflux: Effects of the simplifying assumptions of constant mass flow rate, equal molal overflow and constant heat loss and efficiency.

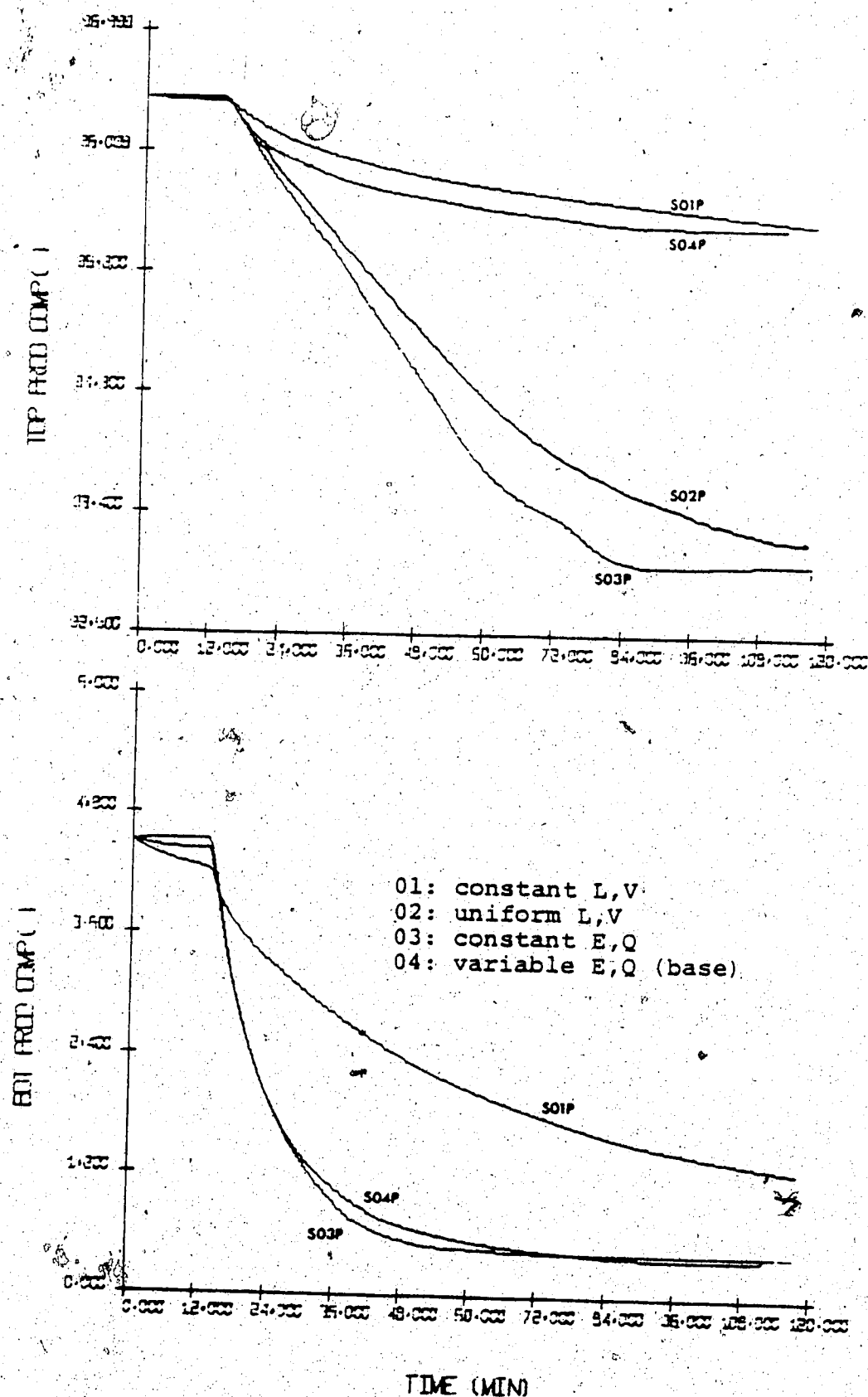


FIGURE 4.5 +10% Steam: Effects of the simplifying assumptions of constant mass flow rate, equal molal overflow and constant heat loss and efficiency.

to be much more pronounced in the bottoms composition response than for the distillate response. Although a very restrictive assumption, the error incurred by the use of this assumption may be acceptable for use in distillate control studies, where compositions do not deviate far from the initial steady state. The savings in computing time may outweigh the lack of accuracy in representing column nonlinearities.

4.2.3 Constant Molal Overflow

The constant molal overflow assumption is a severe assumption if the heats of vaporization are much different. For the methanol-water system, the heat of vaporization of water is 1.8 times the heat of vaporization of methanol. By assuming an average value of the heat of vaporization, simulations predict more vapor flow in the rectifying section of the column and less vapor flow in the stripping section, than would actually occur in the pilot plant column.

The errors due to this simplification may be seen in the 01 series in Figures 4.4 and 4.5 which show the predicted top composition responses for both reflux and steam disturbances. In the case of top composition, deviations from steady state are greater than for the case of constant liquid and vapor flow, and are close to the cases of constant efficiency and heat loss. In the constant molal overflow

case, the flow profile throughout the column will change directly with composition changes only; enthalpy effects, occurring along with composition effects in an actual column, are averaged out.

The bottoms composition responses in the case of constant molal overflow are, as before, close to the other simulated responses. This is occurring for sensitivity reasons, mentioned in the following section.

4.2.4 Heat Loss, Efficiency Functions

As previously mentioned in section 3.3.5, the parameters of tray heat loss and tray efficiency were varied with the composition of a single stage in the column, during the duration of the response. In actual operation, these parameters likely vary individually with tray compositions and flow rates. For simplification, however, these parameters of heat loss and efficiency were taken as functions of a composition at one point in the column. This point was changed to three different locations in three separate simulations: the condenser, the feed tray, and the reboiler. The effects of these changes may be observed in Figures 4.6 and 4.7.

Fitting inaccuracies are shown in the lack of agreement of compositions at the final steady state. Moving the reference location for the heat loss and efficiency functions

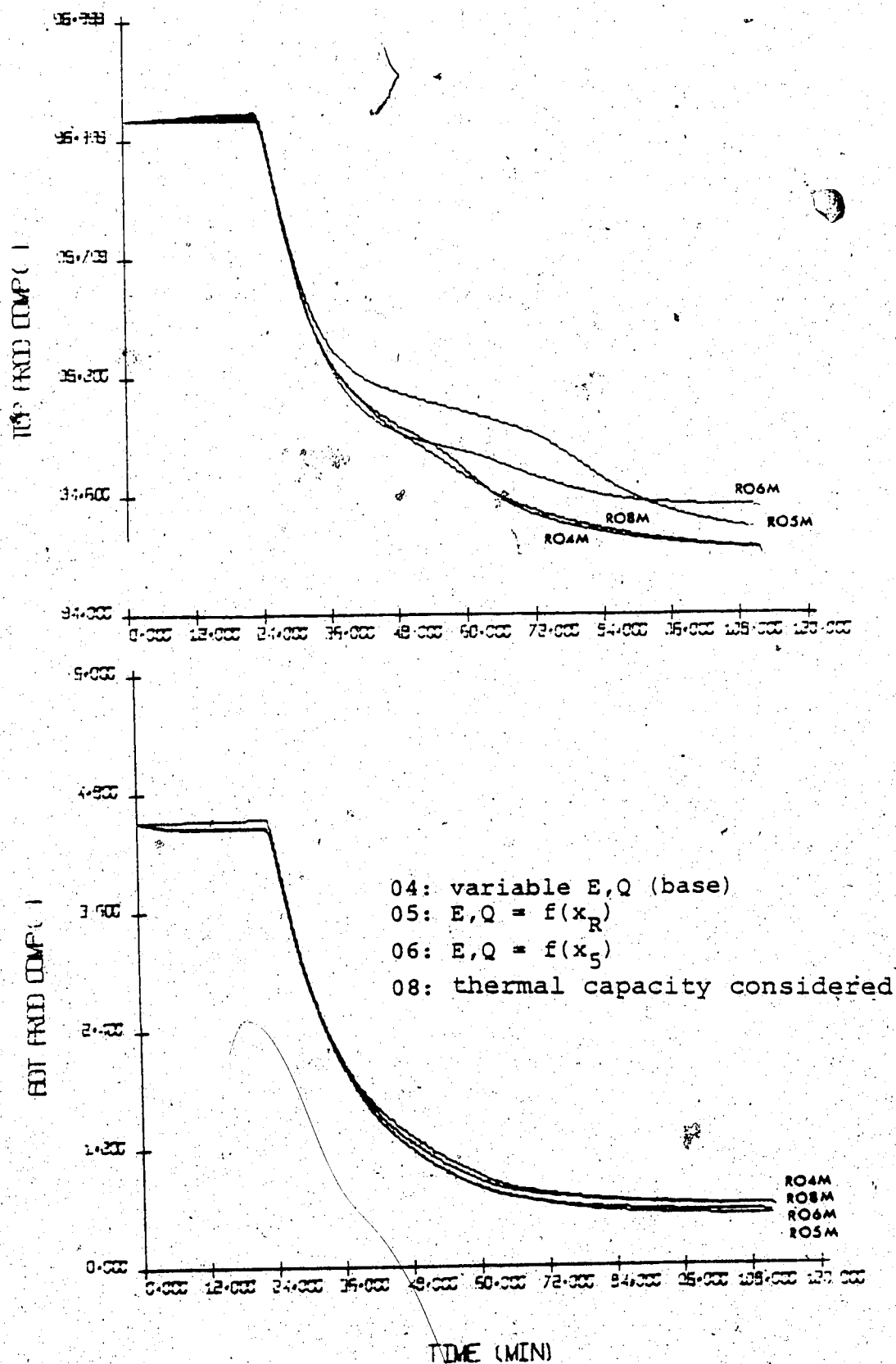


FIGURE 4.6 -20% Reflux: Effects of using other reference compositions for determining efficiency and heat loss. Effect of considering tray and glass thermal capacity.

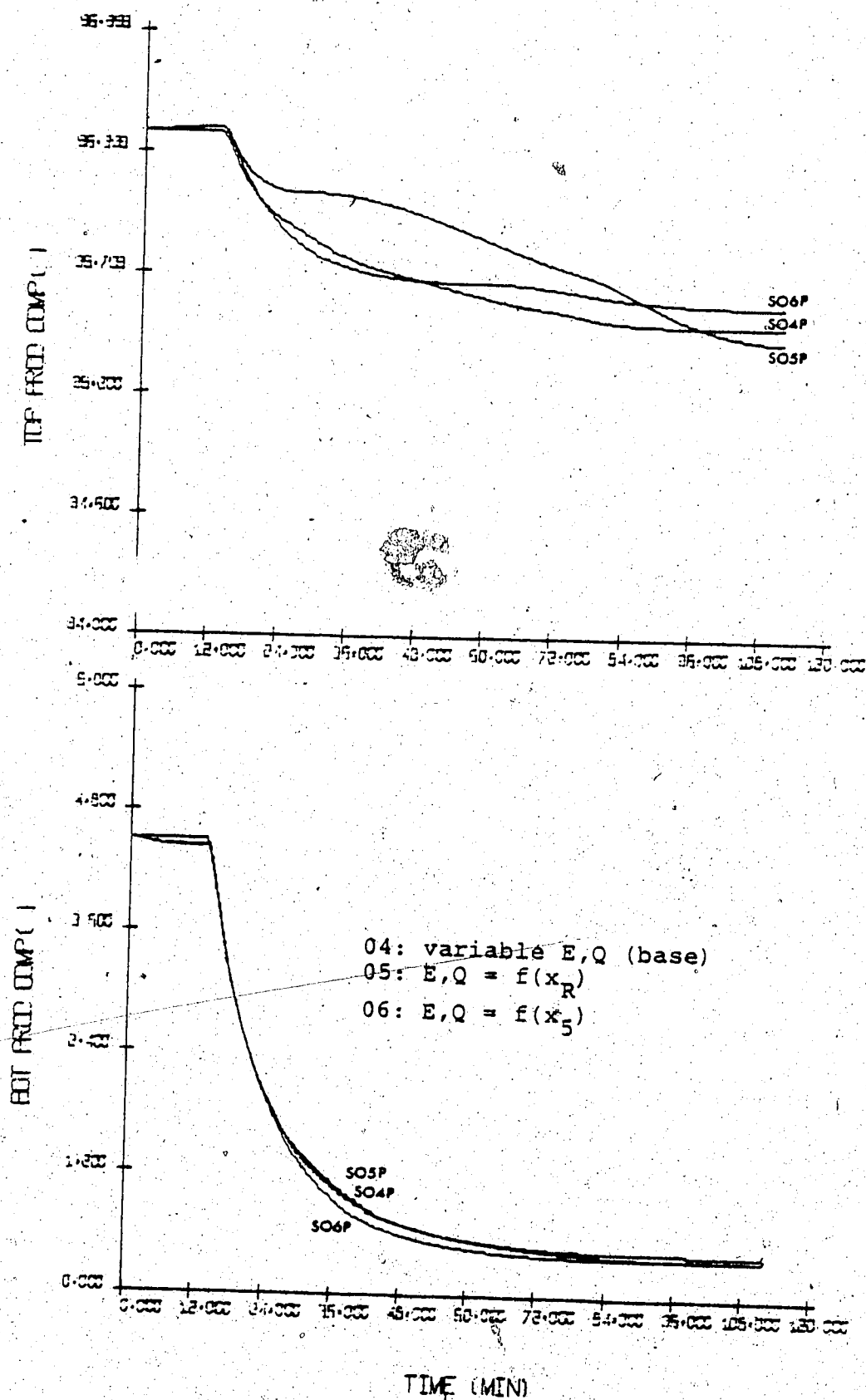


FIGURE 4.7 +10% Steam: Effects of using other reference compositions for determining efficiency and heat loss.

will only change response dynamics and not gains. For initial and final steady states of simulations of different cases of the same model to agree, identical initial and final efficiency and heat loss values must be used for all cases. The parameters, P , of heat loss and efficiency, were varied as expressed by Equation 4.1:

$$P_t = P_i + \frac{(x_t - x_i)}{(x_f - x_i)} (P_f - P_i) \quad (4.1)$$

Although the desired final efficiencies and heat loss were chosen to match the final experimental steady state compositions, and were constant for all three simulations, calculated parameters at the final steady state (P_t , $t = t_f$) did not exactly match the desired final parameters, and caused the discrepancies.

A trial and error method was used to obtain final steady state efficiency and heat loss terms that would enable matching the simulated terminal compositions to experimental ones. A summary of efficiencies and heat loss values that permitted matching for each disturbance is presented in Table 4.3.

However, it is still valid to make qualitative observations as to how dynamics are altered by changing the reference location. In all cases, the dynamic behavior of the bottom composition responses are almost identical to the base case

TABLE 4.3

SIMULATION HEAT LOSS AND EFFICIENCY

INITIAL HEAT LOSSES		INITIAL EFFICIENCIES	
Trays	400 joules/sec	Stripping	.93
Reboiler	1000 joules/sec	Rectifying	.78

DISTURBANCE	FINAL HEAT LOSS (j/s)		FINAL EFFICIENCIES	
	Trays	Reboiler	Stripping	Rectifying
+10% steam	700	1000	.95	.90
-10% steam	200	800	.85	.72
+20% reflux	190	1000	.80	.65
-20% reflux	600	1000	.85	.72
+20% feed	250	800	.93	.78
-20% feed	650	1000	.93	.85

(04) series). Disturbances examined forced the bottoms composition into an insensitive region, at approximately 0.5% methanol, in the pinch region of the equilibrium curve. As a result, the gains were minimally affected. In addition, the relatively high circulation rate in the stripping section and the large holdup in the reboiler combine to make the reboiler composition dynamics dominant. These dynamics largely depend upon reboiler holdup so not much change from model to model would be expected.

The effect of a changing reference composition location does however, significantly affect the top composition dynamics. The three different reference locations for varying heat loss

and efficiency exhibit three distinct effects that are common to both steam and reflux disturbances. With heat loss and efficiency varying with feed tray composition (06 series), distillate composition responds most rapidly, since the feed tray reacts quickly to disturbances, the feed tray composition being in a very sensitive region of the equilibrium curve. With heat loss and efficiency varying with bottoms composition (05 series) distillate composition dynamics show the dynamics of two effects; the flow of energy dynamics cause the rapid reaction of the bottom product composition to the flow disturbance. As the bottom product composition more closely reaches its final steady state, the composition effects working their way slowly through the column, react more slowly at the condenser, eventually trimming all compositions to their final steady state values.

The smoothest of the three responses, as is to be expected, occurs for the case of heat loss and efficiency varying with distillate composition (04 series) which seems to have the slowest response to disturbance in all cases.

The top product composition responses for negative reflux flow disturbances, however, exhibit an unexpected nonlinear type of behavior that is not typical of the experimental responses (as seen in Chapter 5). It is considered that by letting heat loss and efficiency vary with a certain composition in the distillation column, the dynamics of that particular stage are directly introduced and amplified in the dynamics of other

compositions in the column, as the simulated responses show. In addition, the assumption of uniform heat loss and uniform efficiency in the rectifying and stripping sections cause the simulated column to adopt unnatural flow and composition profiles, perhaps also contributing to the different curve shapes.

A model of further complexity could be developed, with heat loss and efficiency varying individually with tray compositions or temperatures. The fitting involved in such a case, however, would be a very complex and time consuming procedure.

4.2.5 Thermal Capacity of Tray Metal and Glass

The possibility that heat effects due to the energy storage in the tray metal and glass could be significant, was examined. The modifications to the model to include this effect involved the addition of one extra term in the energy balance equation, the last term shown in Equation (4.2).

$$\frac{dT_n}{dt} = L_{n+1}h_{n+1} + V_{n-1}H_{n-1} - L_n h_n - V_n H_n - QLP - \sum_i (c_p \cdot m) \frac{dT_n}{dt} \quad (4.2)$$

The plots of predicted composition responses allowing for this effect are shown in Figures 4.6 and 4.8 (the 08 series). Comparison of the responses in these figures with those of the base case show such effects to be negligible, for the pilot plant column used in this study. Changes in the responses may be observed as slight smoothing, evident upon close examination. Also evident are the marginally slower overall dynamics. Both of these effects are expected, an additional heat reservoir having a damping effect, and may be observed in both distillate and bottoms composition responses.

4.2.6 Variable Holdup

Responses of models including the assumptions of variable holdup are presented in comparison with the base case (the 04 series) in Figures 4.9 and 4.10 for reflux disturbances Figure 4.11 for the steam disturbance. Three different hold-up correlations were used. One of those chosen was used by Rademaker et al.³⁰ in their work in distillation column modeling; the Francis Wier formula for flow over rectangular wiers was used in other simulations. The third correlation

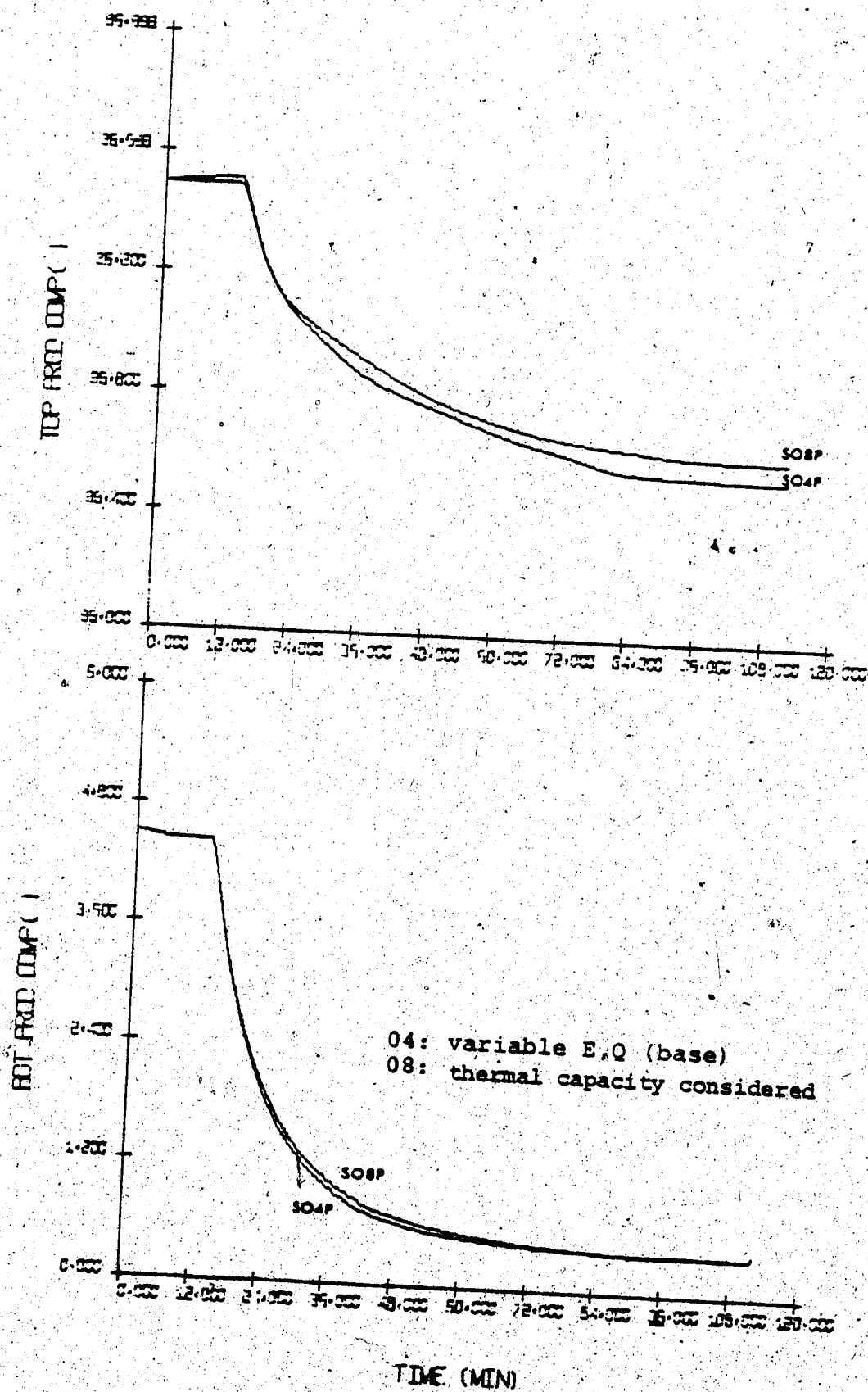


FIGURE 4.8 +10% Steam: Effect of considering tray and glass thermal capacity

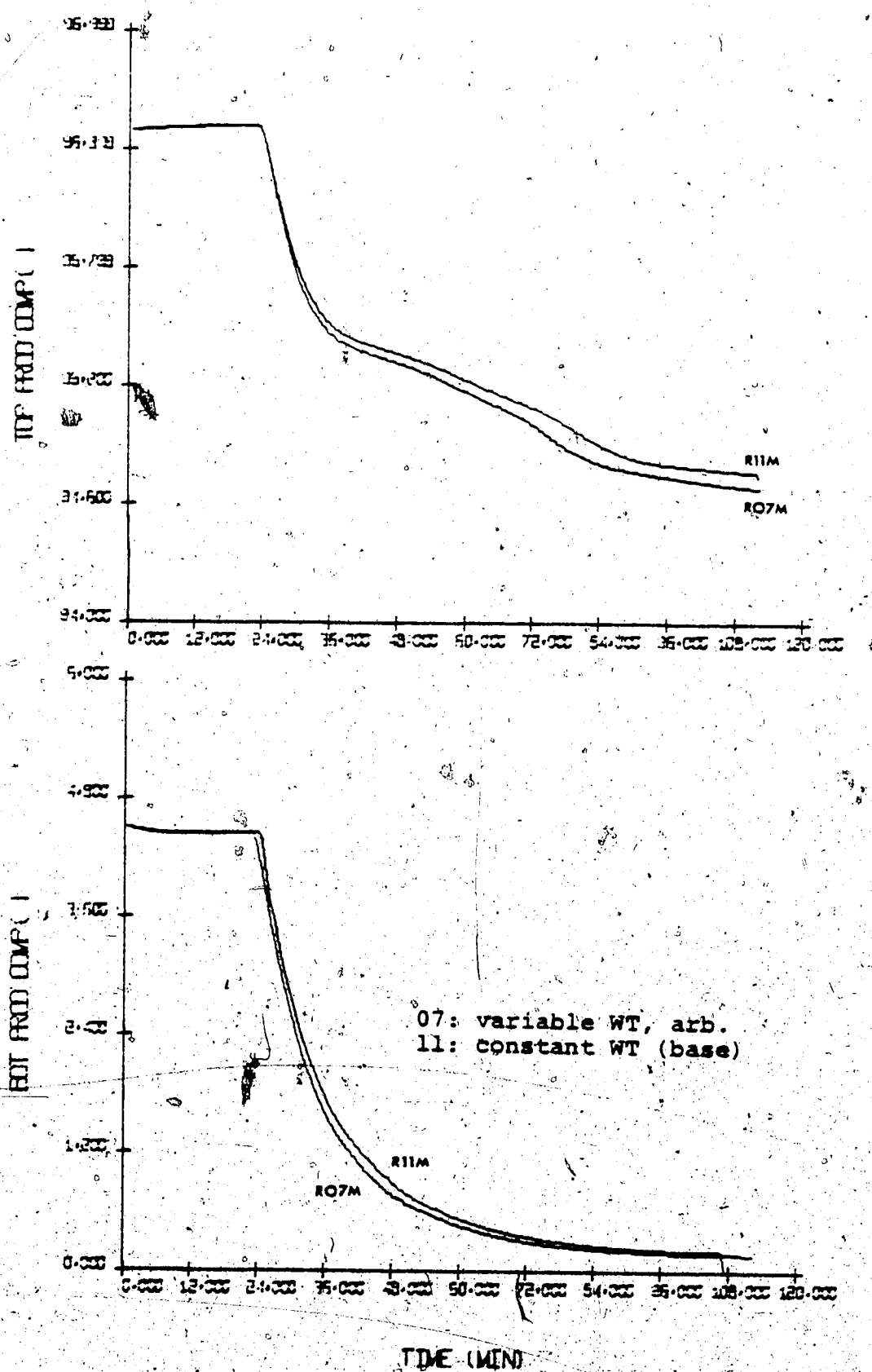


FIGURE 4.9 -20% Reflux: Effects of the assumption of variable holdup according to arbitrary holdup correlation.

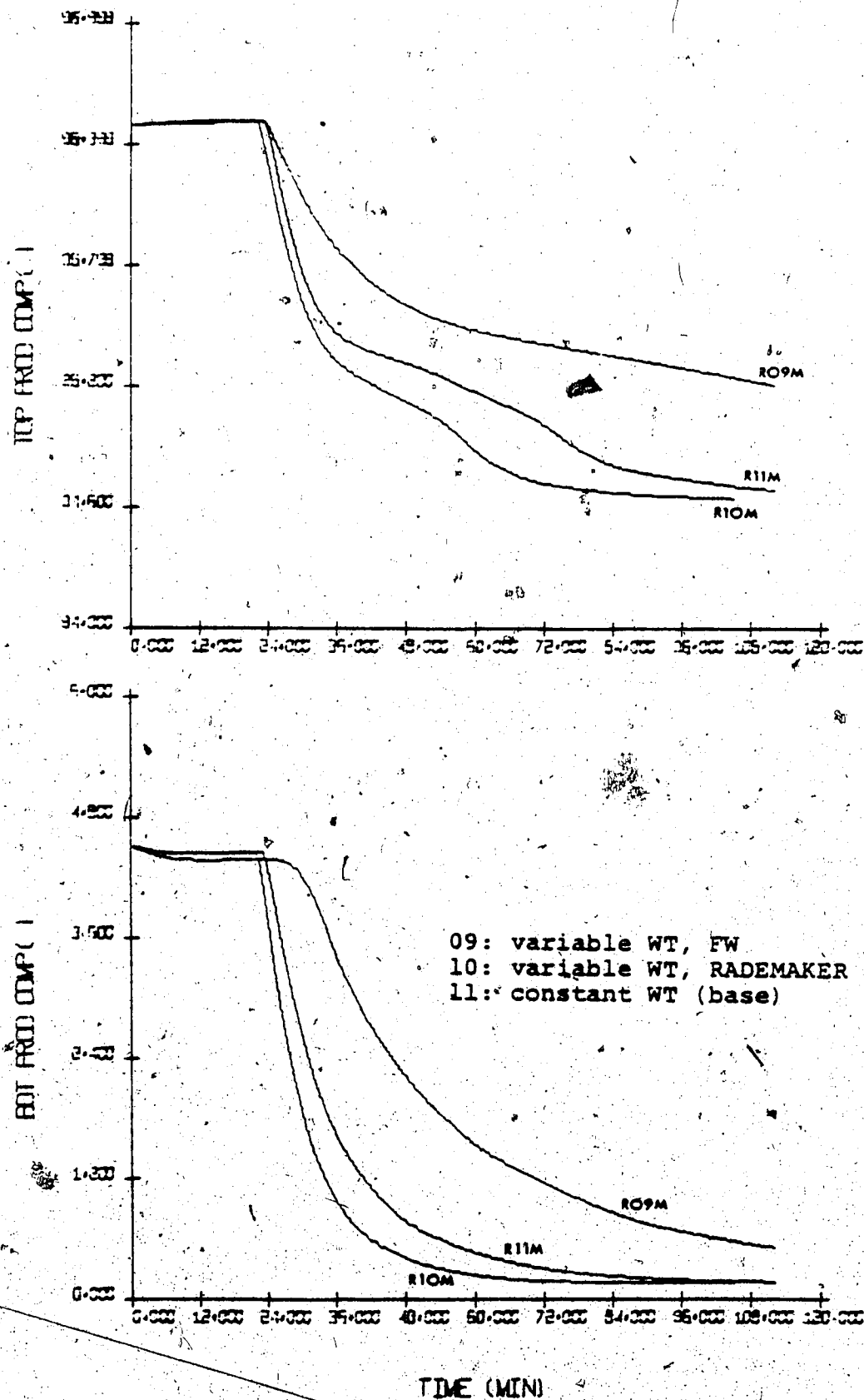


FIGURE 4.10 -20% Reflux: Effects of the assumption of variable holdup according to the FRANCIS WIER, RADEMAKER holdup correlations.

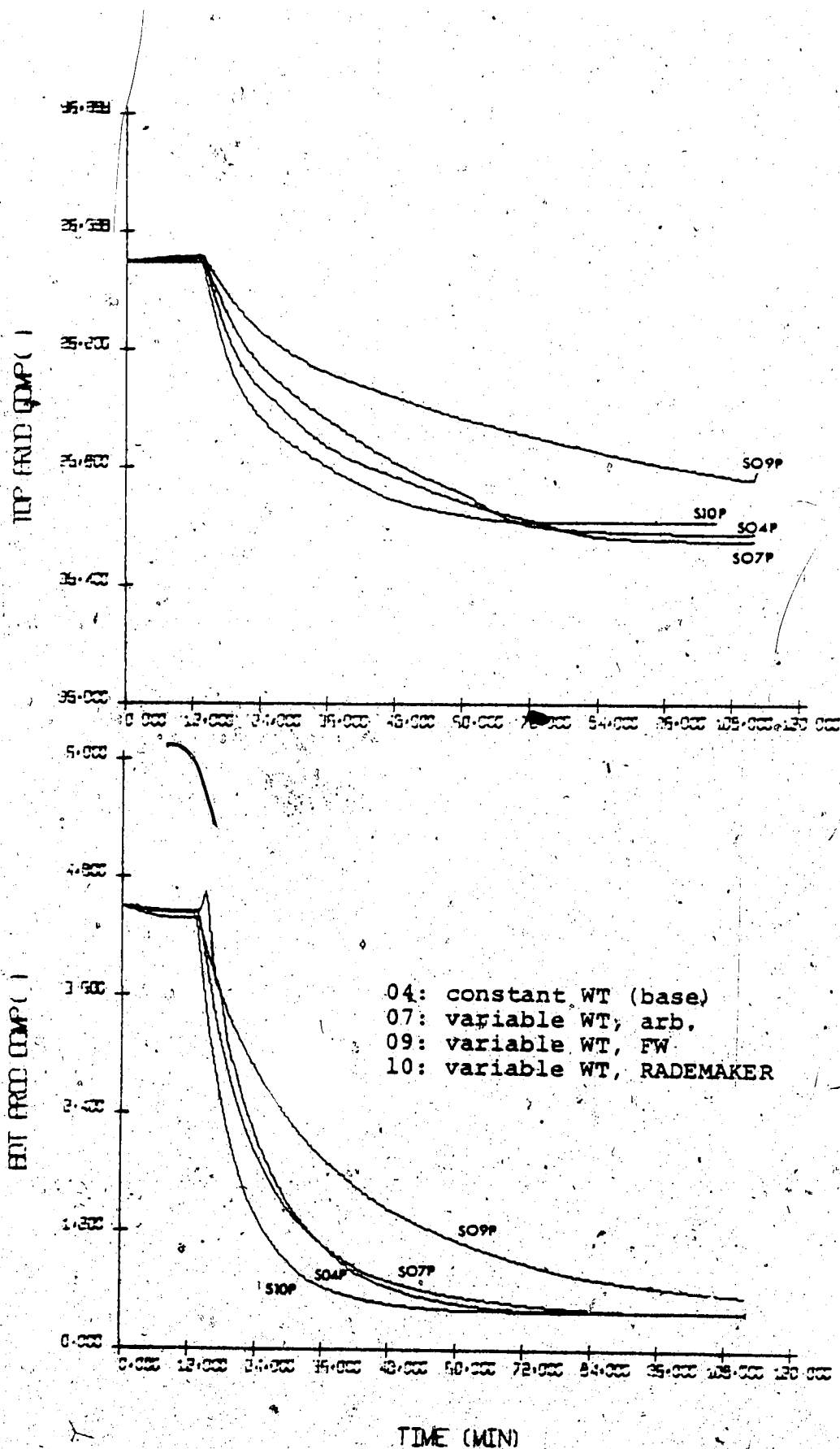


FIGURE 4.11 +10% Steam: Effects of the assumption of variable holdup according to the arbitrary, FRANCIS WIER, RADEMAKER holdup correlations.

was, as the other two, of the general form presented in Equation (4.3), but with arbitrarily chosen parameters a_n , b_n and c_n .

$$WT_n = a_n L_n - b_n V_n - c_n \quad (4.3)$$

For the Rademaker correlation, but not for the third (arbitrary parameters) holdup correlation, the coefficient of the vapor flow, b_n , had a larger magnitude than the liquid coefficient, a_n . This was to have an important effect upon model trajectories; the basis for the selection of coefficient values is presented in section 3.1.4 and in Appendix C. The vapor coefficient, b_n , may be shown to be null in the Francis wier correlation, flow over a wier assumed to have no dependence upon vapor flow. Table 4.4 presents a summary of the coefficients of Equation (4.3) used in this study.

A comparison of the simulations with holdups based on the third correlation (arbitrary parameters, the 07 series) with the simulations of the base case shows very little effect of the variable holdup assumption upon column dynamics. From the bottoms composition response due to a reflux disturbance, dynamics appear to have been slowed down slightly, as is also the case, but more apparent in the steam upset simulation (07 vs 04). However, in Figure 4.10, including the Rademaker correlation with smaller coefficients in the holdup equation resulted in the prediction of responses with

TABLE 4.4
HOLDUP CORRELATION PARAMETERS

Holdup Correlation	Series	Liquid Coefficient a_n	Vapor Coefficient b_n	Constant c_n
RADEMAKER ET AL	10	2.2	-1.54	1500
FRANCIS ¹ WIER	9	$1.39\rho L^{1/3}$	0	1585ρ
ARBITRARY	7	10	-20	1500

¹Correlation depends upon the following parameters

L - liquid flow (g/s)

ρ - tray liquid density (g/l)

Derivation of the Francis wier expression is presented in
Appendix F.

faster dynamics. In Figure 4.11, the same observation may be made with regard to the steam disturbance. Use of the Francis Wier formula (the 09 series) resulted in slower dynamics for both steam and reflux upsets.

These observations are explained by considering the effects of the coefficients " a_n " and " b_n " in Equation (4.3) upon the holdup and flow rates when a disturbance takes place. Flow rates and rates of change of holdup at any time are governed by the total mass and enthalpy tray balances, while absolute holdups and flow rates are related by Equation (4.3). As the enthalpy of tray holdup and liquid leaving the tray are identical, a disturbance which forces the holdup to change will quickly cause the liquid flow to change to satisfy the enthalpy balance. To what extent, and at what rate depends upon the holdup correlation coefficients.

The three sets of equations mentioned above, used in the solution of flow rates and holdups, are solved simultaneously and it is difficult to describe the propagation mechanism. The initial disturbance will cause a change in liquid flow from a particular tray, a change in vapor flow, and a change in the rate of accumulation of holdup, with the magnitude of the changes determined by the values of holdup coefficients and the enthalpies throughout the column. To determine the exact nature of the individual influences of these parameters

would involve a more complete set of simulated runs, for a spectrum of disturbance types and coefficient values. For this reason, no quantitative relationships of dynamics and coefficient values can be presented here. Rather, some examples will show that coefficient sign, magnitude and vapor to liquid coefficient ratio are important.

Figure 4.11 shows the response of the distillation column to a steam disturbance, an upset which initially causes mainly an increase in vapor rate. Generally, flow changes occur more quickly than holdup changes; because of this and the opposite signs of the coefficients of the Rademaker correlation, including the Rademaker correlation in the solution speeds up simulated dynamics. In the short term, because of slowly reacting holdups, the nature of Equation (4.5) would cause a vapor rate increase to predict a liquid rate increase, as a result of including this equation in the model solution. This results in an increased mass circulation rate, spreading the disturbance effects more quickly, and speeding up column dynamics.

Figure 4.10 shows the effects of holdup coefficient magnitude upon column response. Large holdup correlation coefficients such as used in Francis weir correlation, or such as the arbitrarily chosen coefficients ensure holdups sensitive to liquid and vapor rates. In a disturbance,

holdups change faster and further, absorbing or damping the liquid flow changes, and slowing the rate of column response (see section 3.2.2.2).

Figure 4.9 demonstrates the significance of the ratio of liquid coefficients to vapor coefficients. For a reflux disturbance, almost no effect is shown in column response by assuming the arbitrary parameters represent holdup behavior, where for the steam a distinct effect is shown. In both types of disturbances there are increases in vapor and liquid flow, but in different proportions. The coefficients happened to be chosen in such a way, that the vapor and liquid flow rate terms in equation (4.3) were not changed by tray holdup changes to affect column dynamics significantly. For other disturbances, however, these coefficients had a pronounced effect upon column dynamics.

The Francis weir correlation predicts a zero dependence of liquid holdup upon vapor flow, and thus vapor effects are eliminated. Furthermore, the correlation also predicts absolute holdup values approximately double the average measured values, thus giving tray time constants that are double actual values. This results in a severe damping of liquid flow and composition dynamics as can be seen in Figures 4.10 and 4.11.

It is intended that this discussion serve to show that

coefficient magnitude, sign, and ratio are important in determining simulated column dynamics. The speed of the dynamics may be altered by choosing different sets of holdup correlation coefficients.

In conclusion, the effects of allowing a variable volume holdup in the model will depend upon the holdup correlation chosen. Simulation results in this work have shown these effects to cause an increase or a decrease in the distillation column models rate of response to a disturbance. The results also show that the solution scheme of the nonlinear distillation column model developed in this work is stable and valid for a variety of holdup correlations.

4.2.7 Enthalpy Reductions

The feed and reflux enthalpies were reduced by 5 kJ/kg each in independent runs, to determine the effect upon column dynamics. The results of these changes would be to raise the initial steady state methanol compositions on all trays. The results, presented in Figure 4.12 for steam disturbances, indicate little change in dynamics.

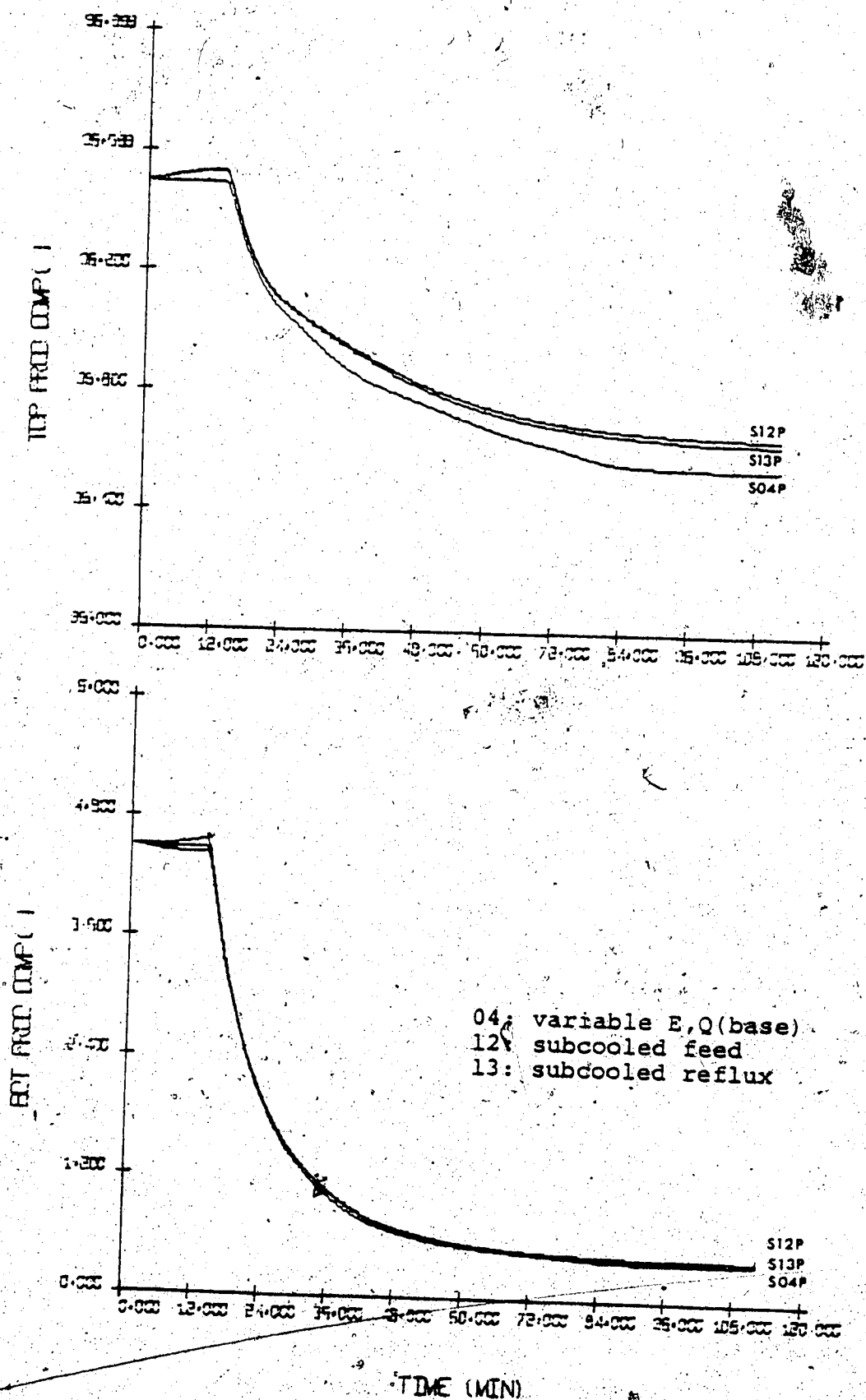


FIGURE 4.12 +10% Steam: Effects of steam disturbance upon column with subcooled feed and reflux streams.

CHAPTER 5

MODEL VERIFICATION RESULTS AND DISCUSSION

5.1 EQUIPMENT

5.1.1 The Column

In order to verify composition transients predicted by the mathematical model, open loop tests were conducted on a pilot scale distillation column separating a methanol-water feed. The column configuration, shown schematically in Figure 5.1, is essentially the same as that used by recent control study workers (28, 44, 47) at the University of Alberta, except for changes in bottoms composition measurement system as described in section 5.1.2.

The 8 tray, 9 inch diameter column is fully instrumented, all pertinent variables being measured or controlled. The walls are glass enabling viewing and estimation of froth heights. The stainless steel bubble cap trays were built at the University of Alberta, to accommodate thermocouples and fluid circulation for composition measurement purposes. The column is equipped with a total condenser and thermosyphon reboiler. Constant liquid level in the condenser and reboiler is maintained by adjusting product flow rates, distillate at the top and bottoms from the reboiler. The feed rate, reflux rate and steam rate, which are the major manipulated and/or disturbance variables are measured and controlled at their

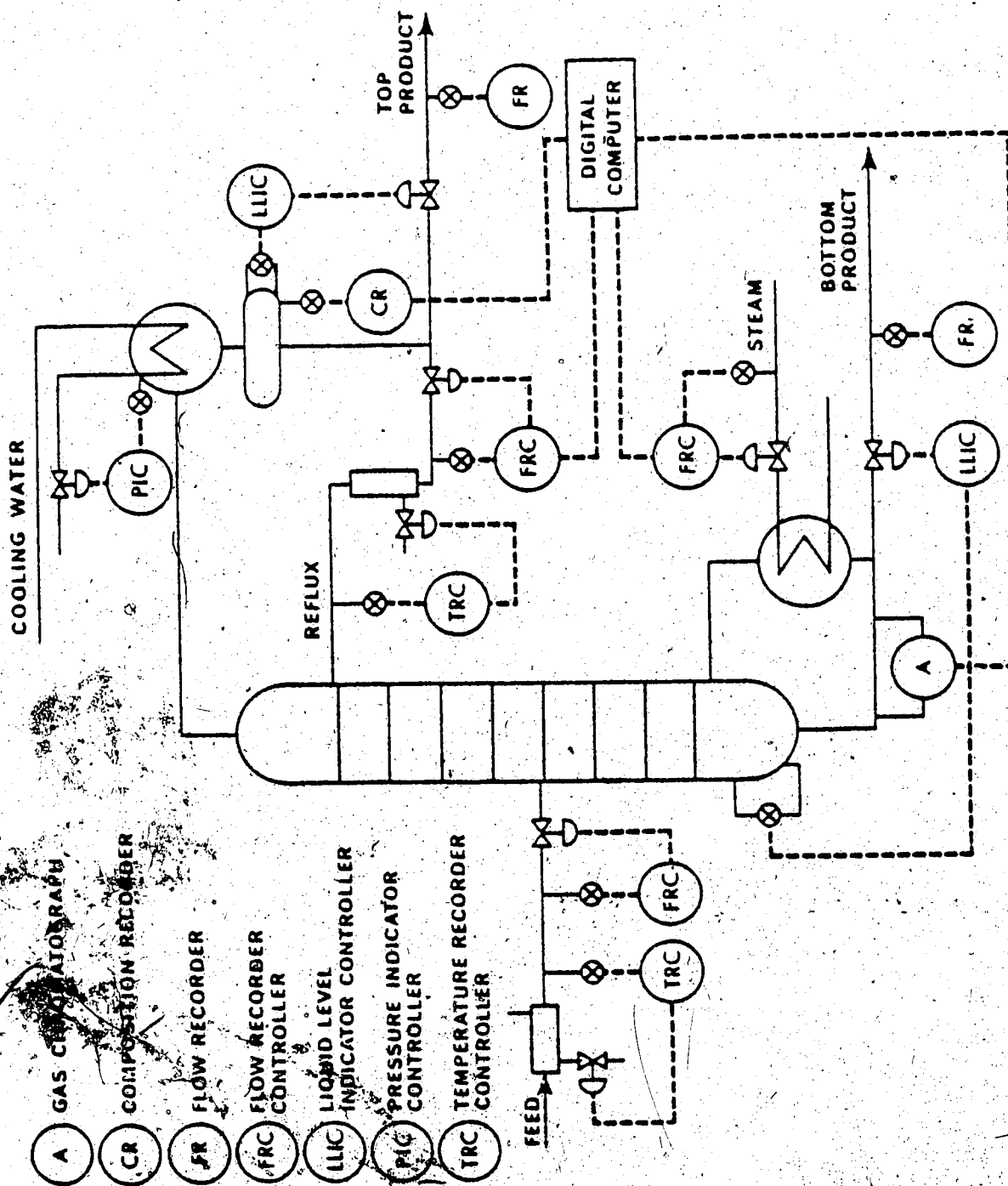


FIGURE 5.1 DISTILLATION COLUMN AND CONTROL LOOPS

set points. Cooling water flow rate to the condenser is employed to regulate tower pressure. Also controlled are the inlet temperatures of the feed and reflux streams. Plate liquid temperatures, cooling water temperatures, steam and condensate temperatures, product temperatures and several temperatures in the reboiler are monitored. The top product composition, high in methanol content was measured by a capacitance probe, and was controlled by an electronic controller cascaded to reflux flow rate. An IBM 1800 digital computer with a Direct Digital Control package provided control and data acquisition, keeping historical records of important measurements.

An online gas chromatograph, used to measure the bottoms composition at 4 minute intervals, was driven by the 1800 computer. It was equipped with an automatic liquid sampling valve, a heater, and a special liquid circulation loop. A detailed description is described in Appendix E.

The feed solution was continuously recycled from a large storage tank, containing a sufficient quantity to prevent feed composition drift during the course of a run.

5.1.2 Bottoms Composition Measurement

As methanol-water mixtures high in water content can not be analysed reliably by a capacitance system, an alternate

method was found. This consisted of a Hewlett Packard model 5722A chromatograph with an automatic liquid sampling valve and a product circulation system, as shown schematically in Figure 5.2.

The gas chromatograph is a discrete analysis system; a sample is injected into a separation column at periodic intervals. A carrier gas draws the sample in gaseous form over a bed of porous material, having an adsorption selectivity to the components of the sample. As a result, the components are physically separated as the gas passes through the bed, and pass out of the bed as peaks of concentration, normally distributed about a characteristic elution time. These impurities in the carrier gas cause a change in heat transfer properties of the combined gas, and passing the mixture over heated metal filaments will cause a temperature rise or fall of the filaments, altering their electrical conductivity. The change in filament resistance is converted to a change in voltage and from the voltage signal, ratios of peak sizes determine relative quantities of components in the sample. A sample voltage signal is presented in Figure 5.3 for a sample containing approximately 5 percent methanol. The upper plot of Figure 5.4 shows only the methanol peak of the same sample, showing an absence of noise.

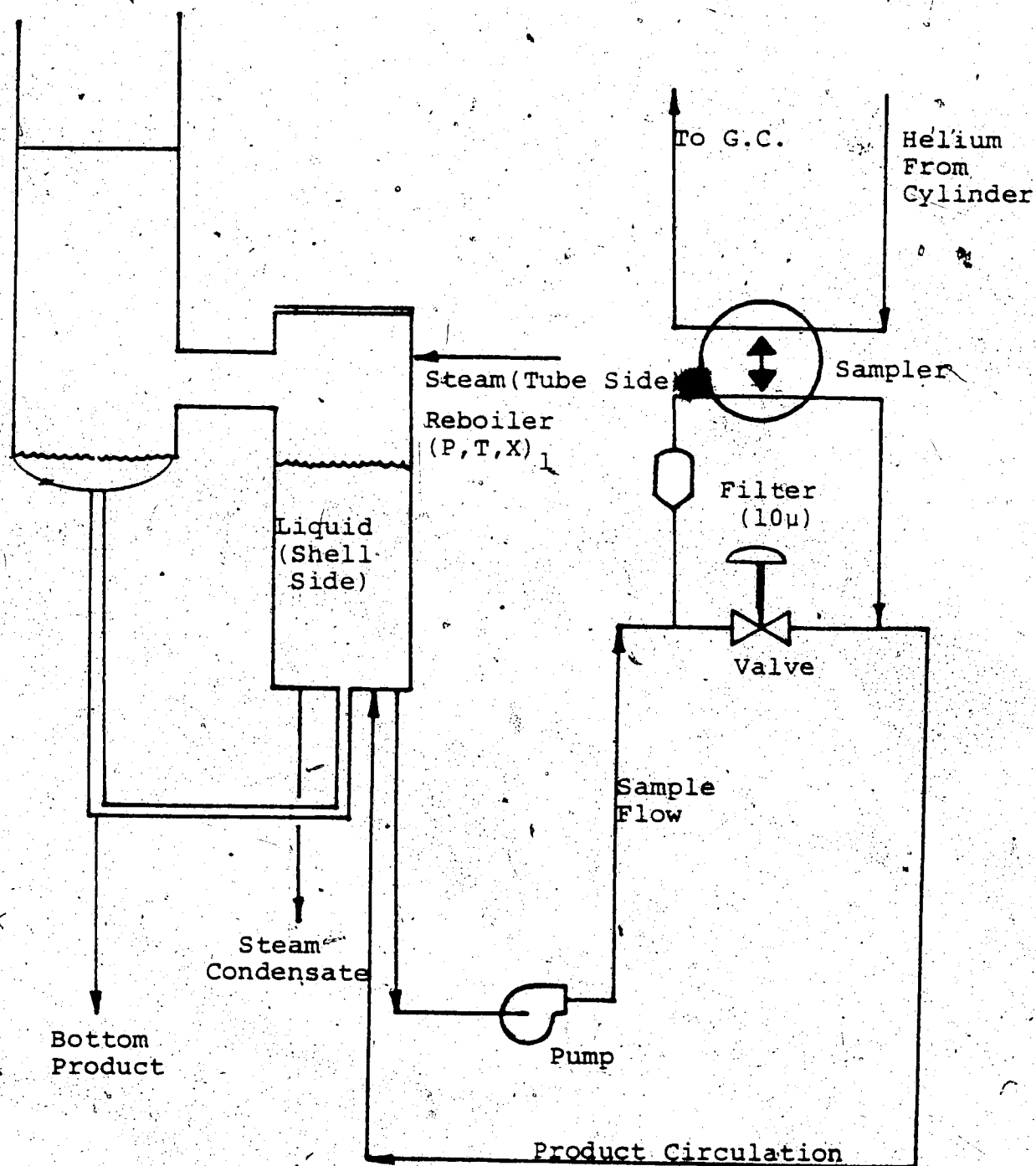


FIGURE 5.2 Bottoms Product Sampling System

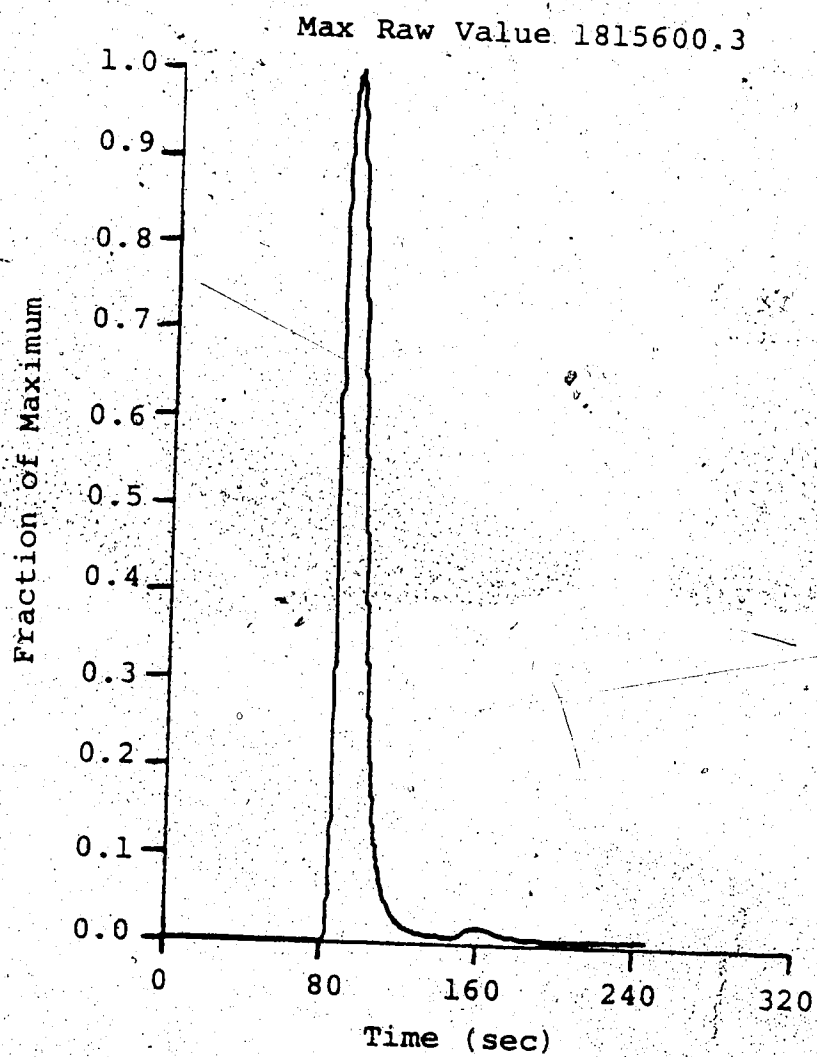


FIGURE 5.3 Sample Chromatogram (5% MeOH)

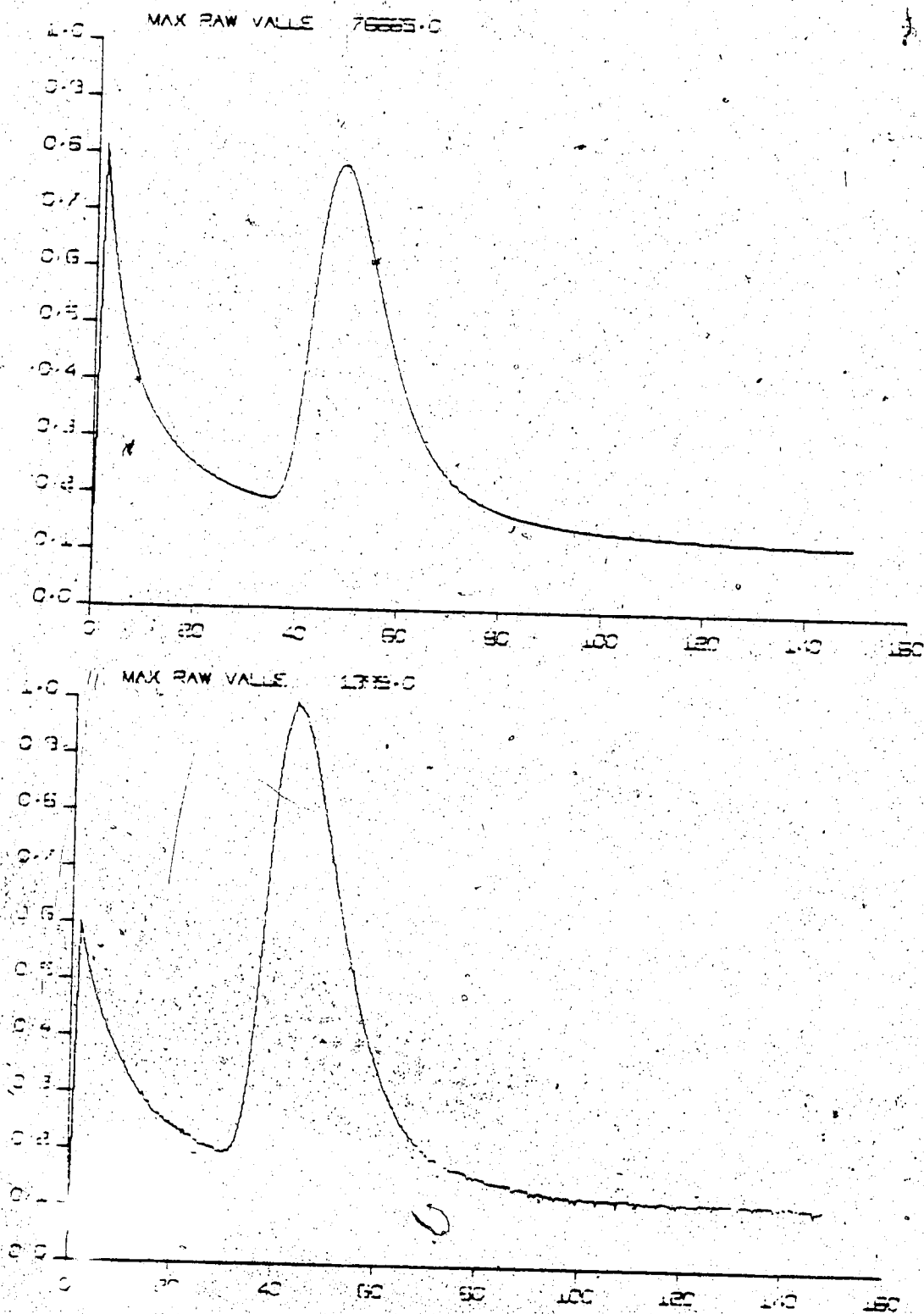


FIGURE 5.4 Typical Amplified and Unamplified Methanol Peak (~6% MeOH)

For a reliable analysis, it is necessary to have good separation of the components in the column. This necessitates careful adjustment of gas chromatograph operating conditions, column temperature and carrier gas flow rate. The injection of sample into the separation column is also very critical as the sample should come into contact with the column packing as a slug, to prevent tailing of peaks or sliding of one peak into another. Also, the sample must be in vapor form at that point. Proper sampling requires that a sample of uniform size from the circulating product stream be flashed quickly, and introduced as a slug to the separation column. Only one of three sampling valves used in the course of this study satisfied these requirements. A more thorough account of these valves is presented later in this section.

The sample volume of the first valve used was easily enlarged, so that the effect of sample size upon gas chromatograph analysis could be determined. The most important and persistent problem encountered was that of reproducibility and consistency of G.C. measurements. It is known that a smaller sample size does give a sharper and more separated chromatogram, but the voltage signal from the detector filaments are lower and, upon reaching the computer analog input point, more disturbed by noise. This was balanced by the upper limit of column loading; a larger sample gave larger peaks but poorer separation. By varying the sample volume

of the liquid sampling valve, it was established from the chromatograms that 2 - 2.5 microlitres of liquid sample was optimum for a 6 foot POROPAK Q column for the methanol-water system.

However, acceptable measurement reproducibility was not obtained despite much effort directed to defining a G.C. computer job to analyse the peaks for the particular column operating condition. The gas chromatogram was analysed using an IBM peak analysis package, which defined peak area calculations based on first and second derivatives of the analog input signal. A computer program was written to enable the user to obtain a graphical presentation of the raw input data and the first and second order derivatives. These results for a low concentration mixture are shown in Figure 5.5 and revealed the need for an amplified signal. The maximum output signal from the detector filaments was approximately 50 millivolts, and without amplification, excessive noise was introduced as the signal was transmitted from the G.C. to the computer, and in digitizing the small analog signals. The difficulty encountered in defining exactly the beginning and end of the methanol peak, from the first derivative values shown in Figure 5.5, is obvious. This uncertainty, causing a varying methanol peak elution time was taken to be the source of the poor measurement reproducibility.

To overcome the low signal readings, an amplifier with

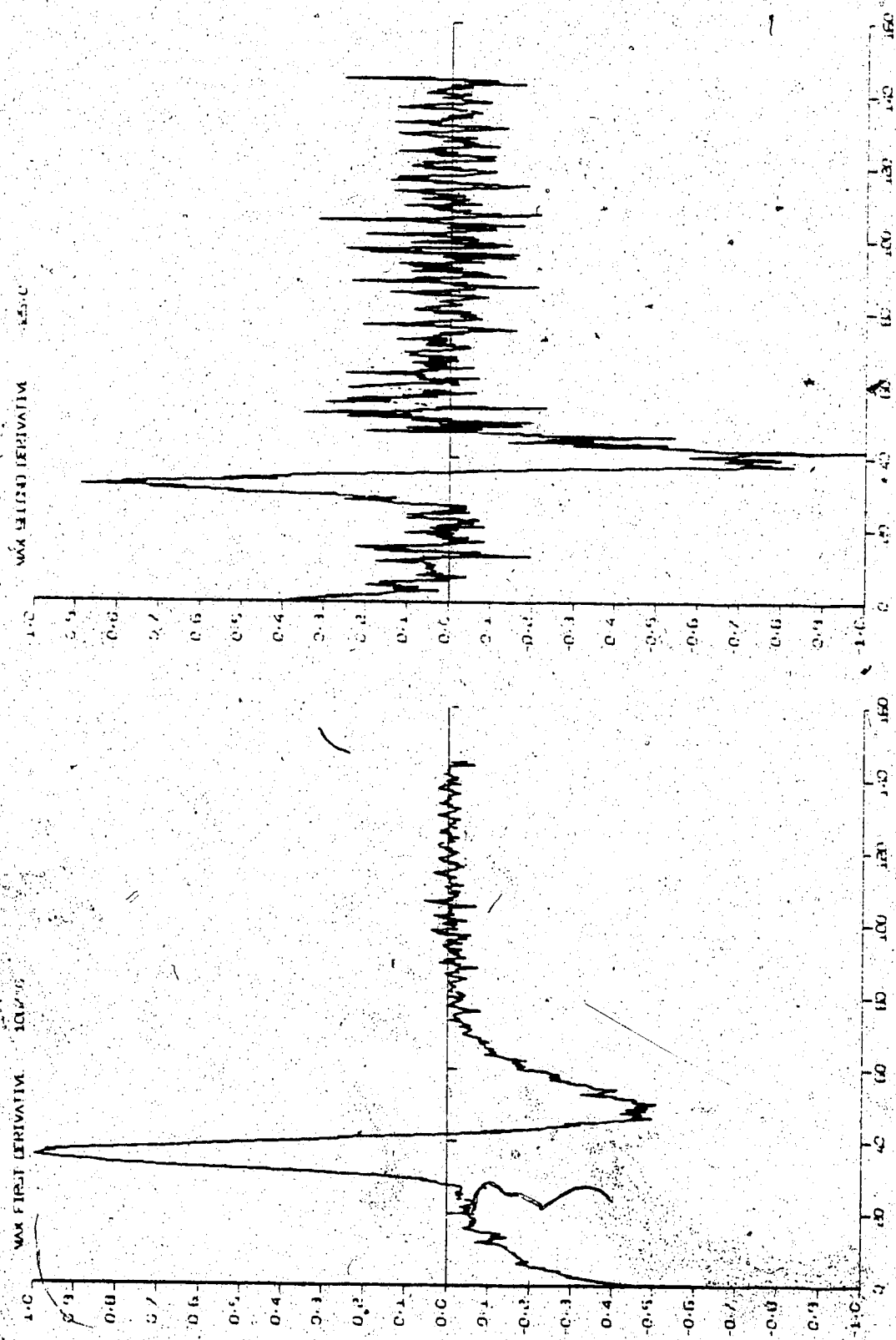


FIGURE 5.5 First and Second Derivatives, Unamplified Methanol Peak

an amplification of 30 was installed immediately after the detector filaments. The effects can be seen in Figure 5.6. For a sample similar to the one producing the results in Figure 5.5, the derivative of the amplified signal has much less noise, decreased by a factor of 30 or more, and clearly defining the beginning and end of the methanol peak. With the amplifier installed, the parameters for the G.C. job definition for the computer analysis program became less critical, and reproducibility was increased to a very satisfactory level. In essence, the amplifier reduced the effect of noise in the input signal, electrical noise due to interference between the computer and the gas chromatograph and noise due to poor digitizing at low signal levels by the analog to digital converter.

As mentioned previously, three sample valves of different design were used for taking a liquid sample and introducing it in vapor form to the separation column. These were the Beckman slider valve, a Valco Hewlett Packard Option 513 automatic sampling valve, and an Applied Automation Model IX valve.

The two main requirements of the valve were adequate flow of sample liquid through the valve and instantaneous flashing of the sample. Of course reliable, steady operation at high temperature over a period of time was desirable as well. The Beckman valve, with sample volume and a larger

MAX FIRST DERIVATIVE - 0.000 0

1.0

0.9

0.8

0.7

0.6

0.5

0.4

0.3

0.2

0.1

0.0

-0.1

-0.2

-0.3

-0.4

-0.5

-0.6

-0.7

-0.8

-0.9

-1.0

0

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140

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760

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800

820

840

860

880

900

920

940

960

980

1000

1020

1040

1060

1080

1100

1120

1140

1160

1180

1200

1220

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bypass drilled into a sliding teflon block, was originally installed in a Beckman Series 4 Industrial Chromatograph, in the same oven and thus at the same temperature as the separation column. This configuration had been used for distillation column studies at the University of Alberta until 1975. Flow and operating reliability were adequate, but flashing of the liquid sample was slow, resulting in less than optimum separation. Adjustment of column conditions was difficult, as a high flash temperature but low column temperature are needed for good analyses. Also, temperature control was a trial and error procedure, with only a crude heater to adjust. At the commencement of this work, a new chromatograph with a separate valve oven and new sample valve were installed. The sample valve, manufactured by Valco, consisted of a conical teflon block with sample volumes cut into the sides, rotating in a metal sleeve with ports. The heat transfer and flashing properties of this very compact valve were much improved, as one face of the sample volume was metal. Flow through the valve, however, was not adequate, due to the complexity of the flow circuit. Breakin procedures were difficult, and the teflon block would scorch onto the metal sleeve with continued use. Similar problems with a replacement valve of the same basic design led to the decision to "search for another type of valve.

The valve presently installed is an Applied Automation

Model IX. It is a unique design, in which pneumatically actuated small plungers move small fractions of an inch to block or open flow passages of carrier gas or liquid sample through the sample valve or bypass. The sample volume is encased in metal on 5 sides. Flow rates through the valve are higher than both other valves and it has been used successfully during most of this work. A small teflon diaphragm must be replaced periodically due to deterioration with temperature.

The sample valve was actuated under control of the IBM gas chromatograph package, which also performed the peak identification and area calculations. The composition in weight percent methanol, calculated from the areas determined by the IBM package, was then written into a DDC measurement loop by an online user written program. This program, designated as DWL44 (see Appendix B), was queued by the IBM package at the end of the job cycle. Thus, the composition was available to the bottoms composition control loop.

5.1.3 Holdup

~~An attempt was made to determine the mass holdup of liquid on each tray of the distillation column and in the reboiler and condenser for comparisons with values predicted by common correlations. Furthermore, an attempt was also made to find unique tray holdup correlation for this distillation column,~~

based on liquid and vapor flow rates predicted by the model.

Holdup could not be measured but was estimated as follows. With one tray as a standard (feed tray), estimates of the relative quantities of liquid on the trays were made by viewing the froth nature and froth heights through the clear glass walls of the column. The total weight of solution in the column was determined by operating the column at steady state and abruptly then stopping all flows, and draining the liquid from the column for weighing. The condenser and reboiler holdups were determined with the column non-operational. Since the levels of the condenser and reboiler are controlled, it was assumed that their holdups did not change so by subtracting the condenser and reboiler holdup from the total column holdup, the cumulative tray holdup could be calculated. Individual tray holdup was calculated by taking the relative volume estimates, applying a density correction, and multiplying this tray ratio by the cumulative tray holdup. This procedure was followed for the standard operating conditions, and for feed flows of ten and twenty percent above and below the normal flow rate.

The results of the estimated holdups are presented in tabular form in Appendix F (Table F.1) and in graphical form in Figure 5.7, for all operating conditions tested. Also plotted on Figure 5.7 are holdups based on published holdup

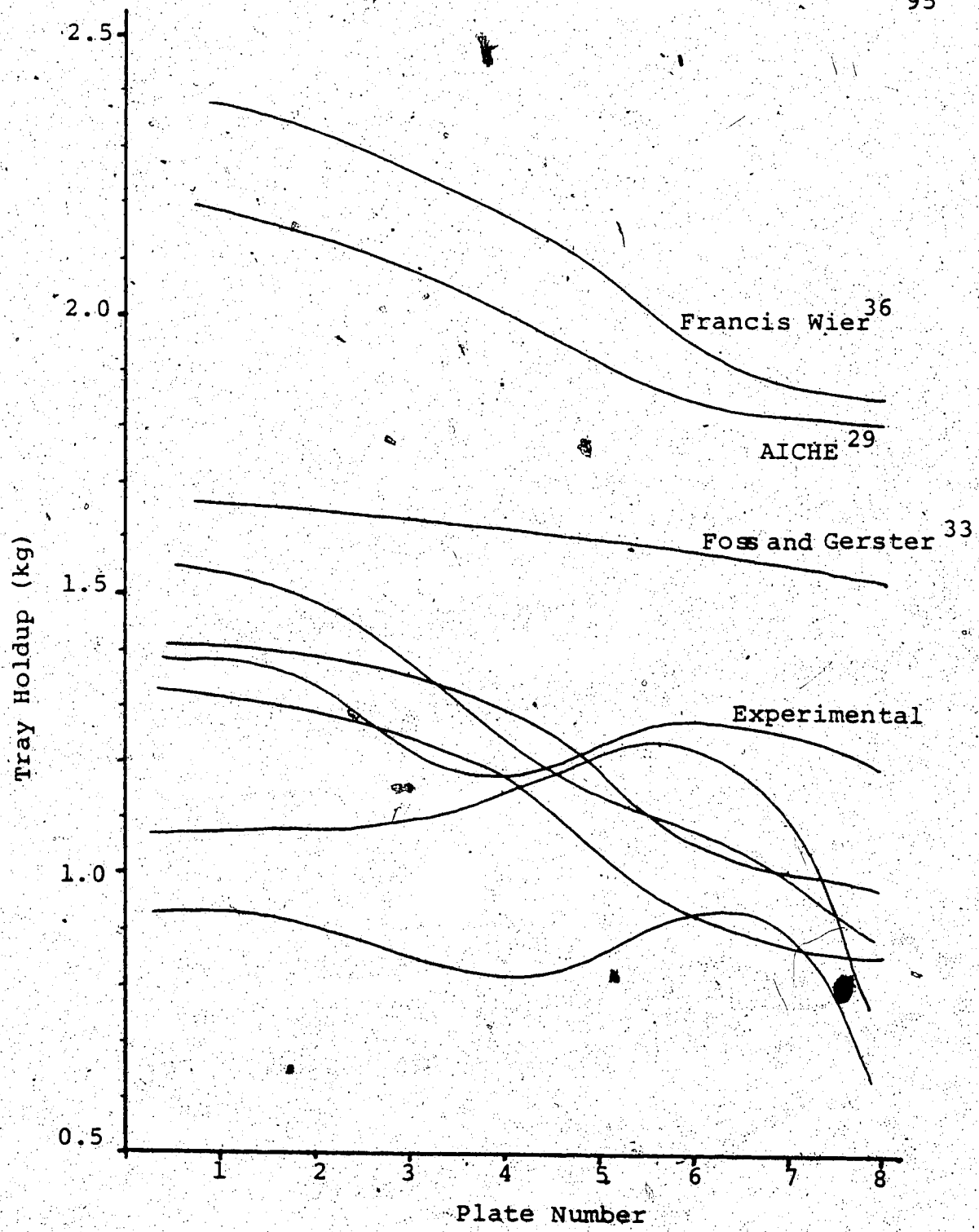


FIGURE 5.7 Liquid Holdup Trends

correlations, using liquid and vapor flow rates from individual trays, predicted by the model described in Chapter 3. Both experimental holdups (for a variety of operating conditions) and holdups derived from correlations predict a similar trend of holdup decrease with an increase in tray number. However, holdup correlations predict values that are larger than measured ones, and in the case of the Francis Wier prediction, larger by up to a factor of two. A complete summary of estimated and measured tray holdups is presented in Appendix F.

Attempts were made to correlate estimated holdup values with both tray liquid flow rate (predicted by the model described in Chapter 3) and feed flow rate, using the plots shown in Appendix F. No definite relationships were observed in either case, although generally, an increase in feed rate appears to result in an increase in holdup on all trays. The excessive scatter of data is attributed to the estimation technique, and may be observed in Figure 5.8.

The published holdup correlations used were as follows:

- i) AICHE²⁹ method of efficiency prediction for bubble cap trays:

$$z_c = 1.65 + 0.19w - 0.65F + 0.020L \quad (5.1)$$

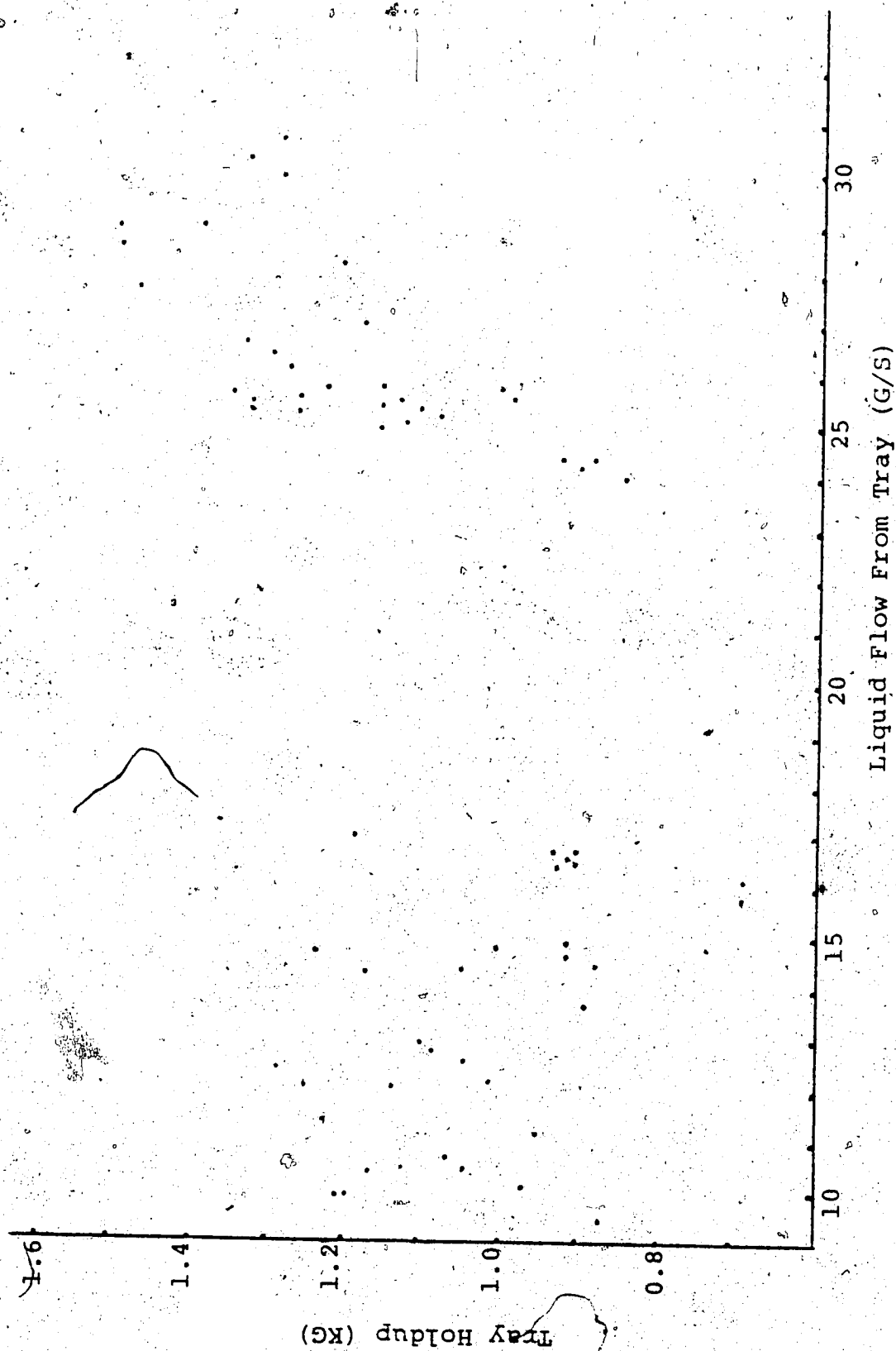


FIGURE 5.8 Measured Tray Holdups vs. Simulated Liquid Flow

where z_c = inches of liquid on the tray

w = weir height (inches)

$F = V \rho_G^{0.5}$

V = vapor flow from tray (ft/sec)

ρ_G = vapor density (lbm/ft³)

L = liquid flow (gpm/ft of flow width)

Volume holdup was converted to a mass by applying a density.

ii) The Francis Wier³⁵ formula for flow over a rectangular wier:

$$q = .415(L - 0.2h_o)h_o^{1.5} \sqrt{2g} \quad (5.2)$$

where q = volumetric flow over the weir (ft³/sec)

L = length of the wier crest (ft)

h_o = height of liquid above wier (ft)

g = local acceleration due to gravity (ft/sec²)

This equation was used to calculate a weir head for a given flow through the weir. The assumption that the term "0.2 h_o " was negligible next to the wier crest length L simplified the equation. The entire liquid volume on the tray was assumed to be clear (negligible froth volume), and volume and weight holdup was calculated on that basis.

iii) Foss and Gerster³³ holdup equation

$$h_L = 0.24 + 0.725h_w - 0.29h_w V_a G^{0.5} + 4.48 q/z \quad (5.3)$$

where h_w = outlet wier height (in)

h_L = liquid head on tray (in)

V_a = gas velocity (ft/sec)

ρ_G = gas density (lbm/ft³)

q = liquid flow rate (ft³/sec)

z = average liquid flow width (ft)

Sample calculations for all cases were made in, or the results converted to, SI units, and are also presented in Appendix C.

Holdups predicted by the published correlations are all higher in magnitude than those measured. This may be explained by the fact that bubble cap trays, designed and constructed by Svrcek²⁶ for the original distillation column control study, may not be of a standard design. Also, the column is a pilot plant column, smaller in scale than industrial columns, for which the published correlations were derived. For this reason, these published holdup correlations were not used for predicting holdups during the computer simulations of the distillation column, as presented in Chapter 4.

5.2 RESULTS

Considerable time was spent in determining an appropriate set of operating conditions. It was found that a tradeoff

between sensitivity to disturbances and bottom composition measurement accuracy was to occur. It was decided that product compositions should be between 4 and 5 percent in the bottoms and between 96 and 97 percent methanol at the top. The composition range was similar to that used by other workers, but the column had been operated at lower bottoms composition to achieve good distillate sensitivity to upsets. For this reason, certain disturbances will exhibit lower gains than open loop responses from previous tests by other workers. A list of sample initial operating conditions can be found in Figure 5.9.

As previously mentioned, a gas chromatograph was used to measure the bottom product composition. This is a discrete measurement device, and inherently has a dead time of 4 minutes (in the analysis program used in this study). As will be noted in the plots of bottom composition responses, there appears to be a time difference of from 8 to 12 minutes between the simulated and experimental trajectories. This may be explained with the aid of Figure 5.10. The G.C. program of the IBM 1800 computer was used to collect gas chromatograph analyses. Upon completing an analysis, a G.C. queued service program (DWL44, Appendix B) would write the analysis result into a measurement word in a DDC loop record. Depending upon when the measurement was taken with respect to the start of the disturbance, the delay in getting a measured

STEADY STATE DATA
 RUN NO DL1048
 01.06/76

FEED FLOW	16.97G/SEC	BOTTOM PROD	8.67G/SEC
REFLUX FLOW	13.52G/SEC	TOP PROD	8.46G/SEC
STEAM FLOW	14.93G/SEC	COOL WATER	514.05G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MECH
DIST COMP	96.57WT% MECH	BOTTOMS COMP	6.00WT% MECH
FEED INLET	74.3DEG C	REFLUX INLET	65.3DEG C
STEAM TEMP	106.7DEG C	PRESSURE	-30.3KPA

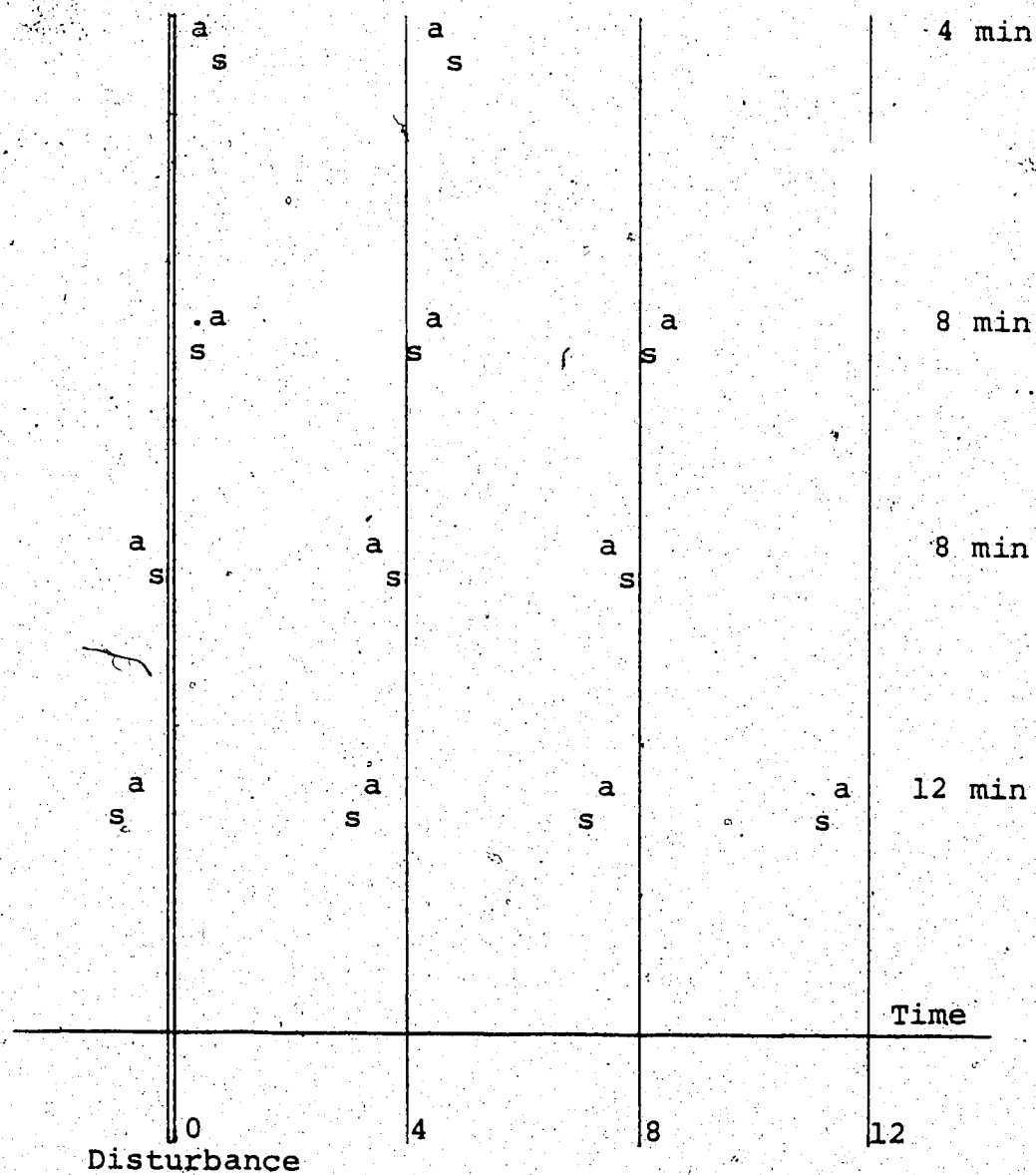
M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.97	50.00	8.48	8.48
BOTTOM PRODUCT	8.67	6.00	0.52	8.15
TOP PRODUCT	8.46	96.57	8.17	0.28
CLOSURE ERROR-PC	0.9		2.4	-0.5

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	46.19	71.95
REFLUX	2.69	2.55
TOP PRODUCT		1.59
FEED	4.61	
STEAM	40.23	6.63
BOTTOM PRODUCT		3.37
TOTAL	93.73	86.11
HEAT LOSS		7.6

Figure 5.9 Sample Standard Operating Conditions



a - G.C. analysis
s - DDC loop scan

FIGURE 5.10 Bottoms Composition Analysis Collection

composition into the DDC loop record could be from 4 to 8 minutes. Another data accumulation loop with a pole time of 256 seconds would take the measurement from the measurement loop, and write it to a measurement history file. If this loop were not in phase with the gas chromatograph analysis, an additional delay of 256 seconds could result. If the measurement were filtered, another delay could be incurred. Together, this could constitute a total of 16 minutes delay. However, all response lags fall within the 4-12 minute delay, as filtering was not used.

There also exists a slight process delay due to the G.C. cycle loop (section 5.1.3, Appendix E), but this delay was considered to be small compared to other G.C. delays.

Results are presented for positive and negative 20 percent step disturbances in reflux and feed rates, and 10 percent steps in steam rate to the reboiler. In Figures 5.11 to 5.16 the experimental response in each case is accompanied by a simulated response. The simulation results presented here are for the 04 series (Table 4.1), with the assumptions of constant mass holdup, variable heat loss and variable efficiency, with the variation according to changes in top composition. This simulation series was chosen because it was found to adequately represent the experimental response. To ascertain the agreement that would exist between predicted responses based on different assumptions and the experimental

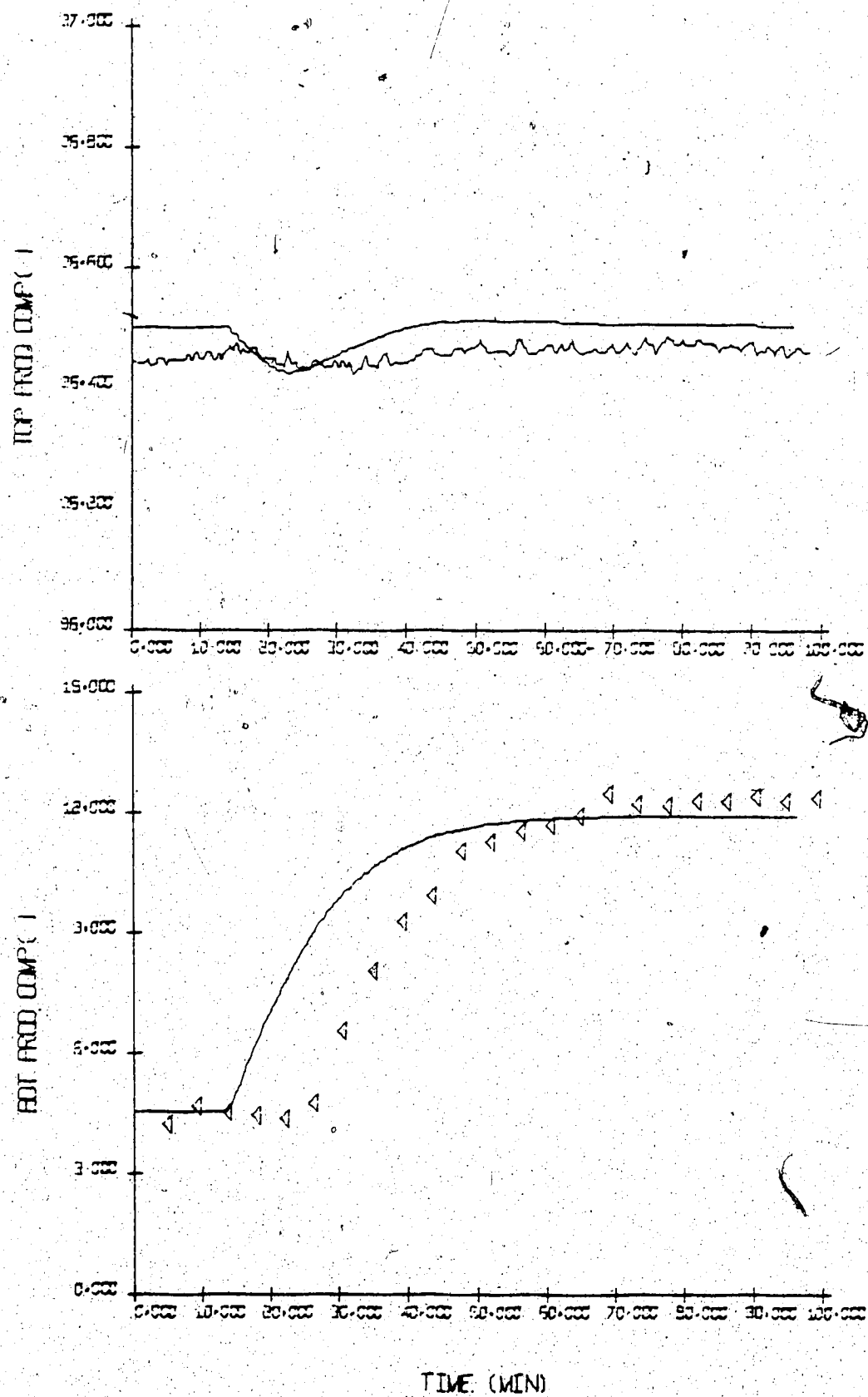


FIGURE 5.11 +20% Feed flow; Simulation vs Experimental

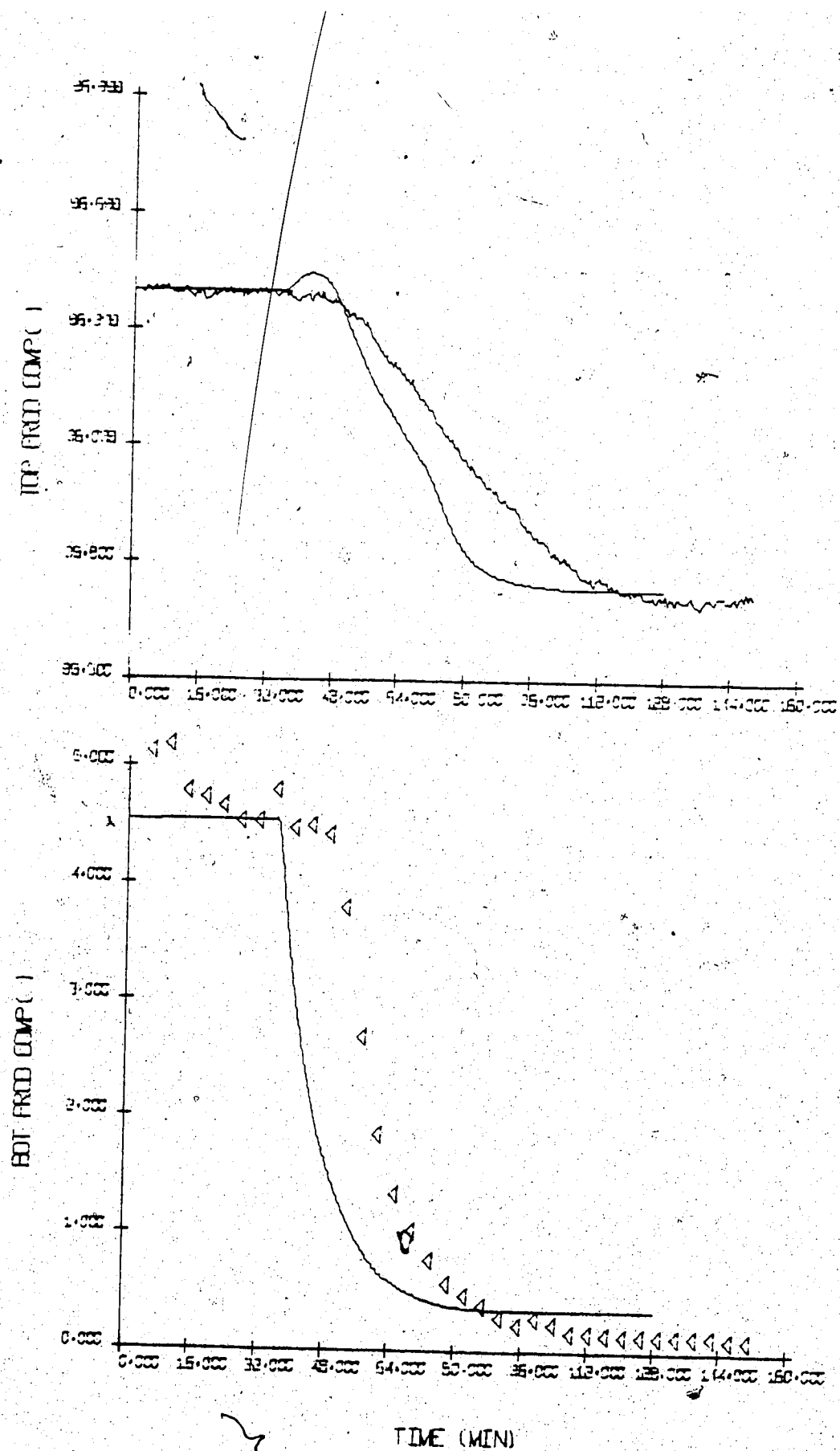


FIGURE 5.12 -20% Feed flow; Simulation vs Experimental

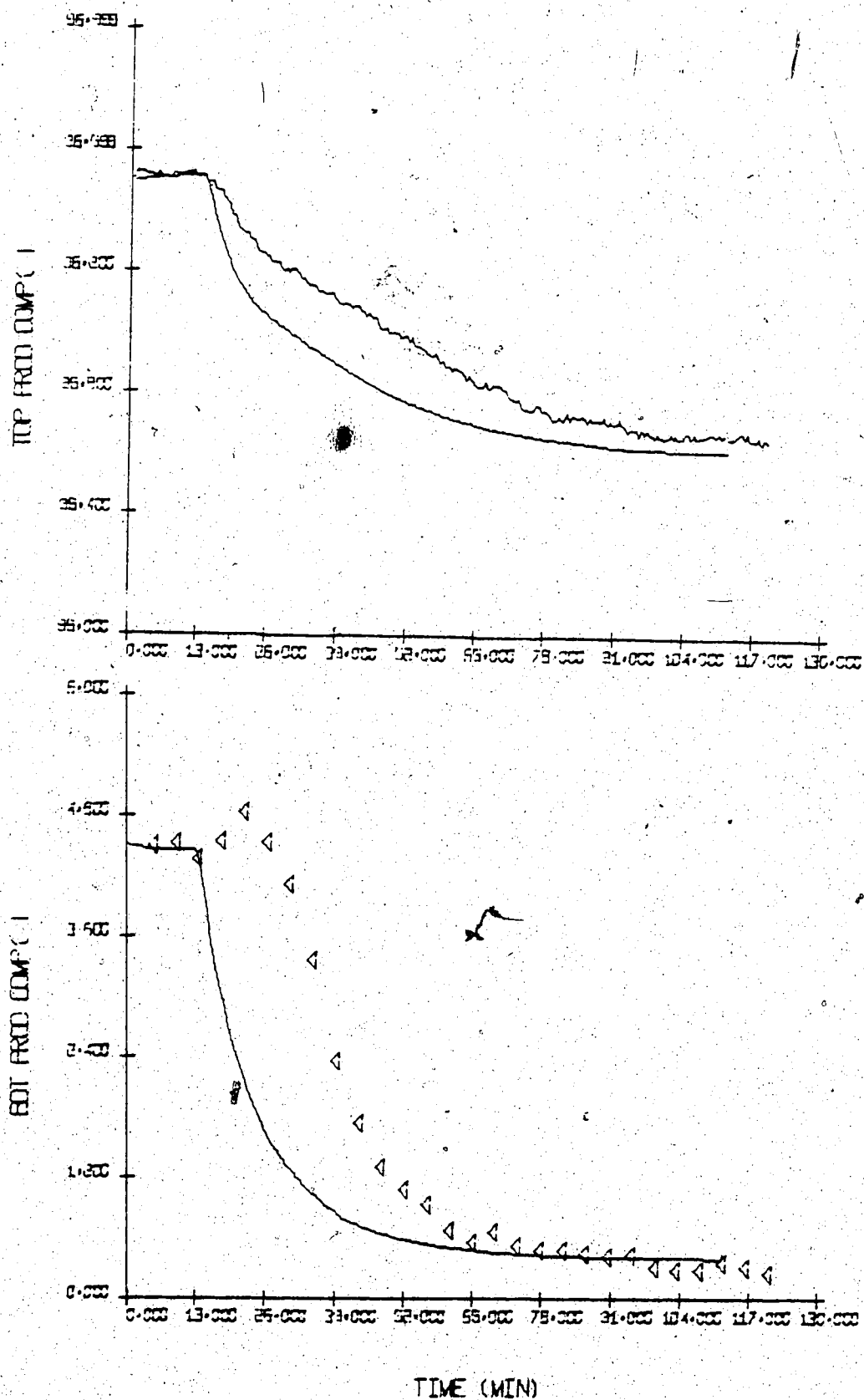


FIGURE 5.13 +10% Steam flow; Simulation vs Experimental

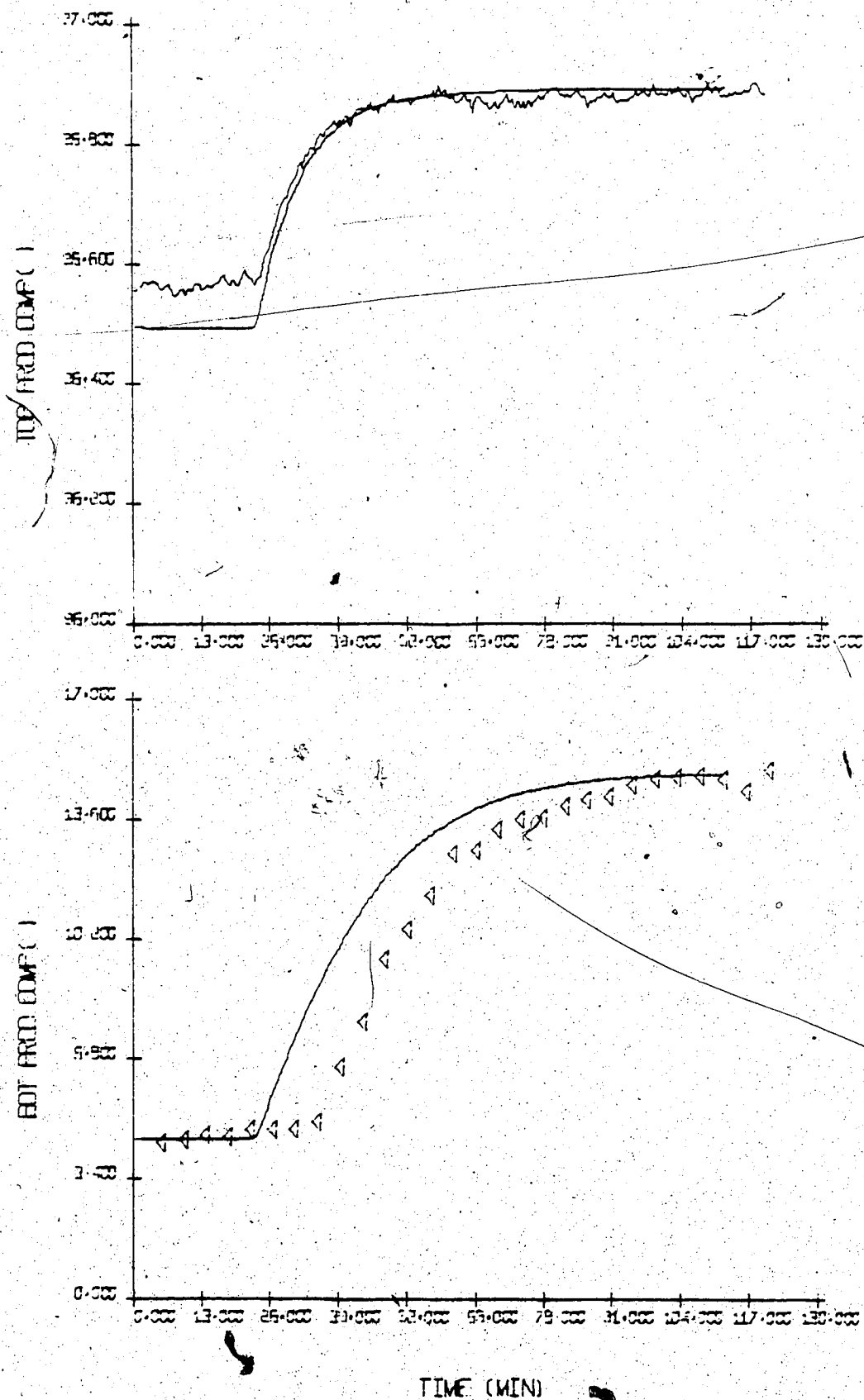


FIGURE 5.14 -10% Steam flow; Simulation vs Experimental

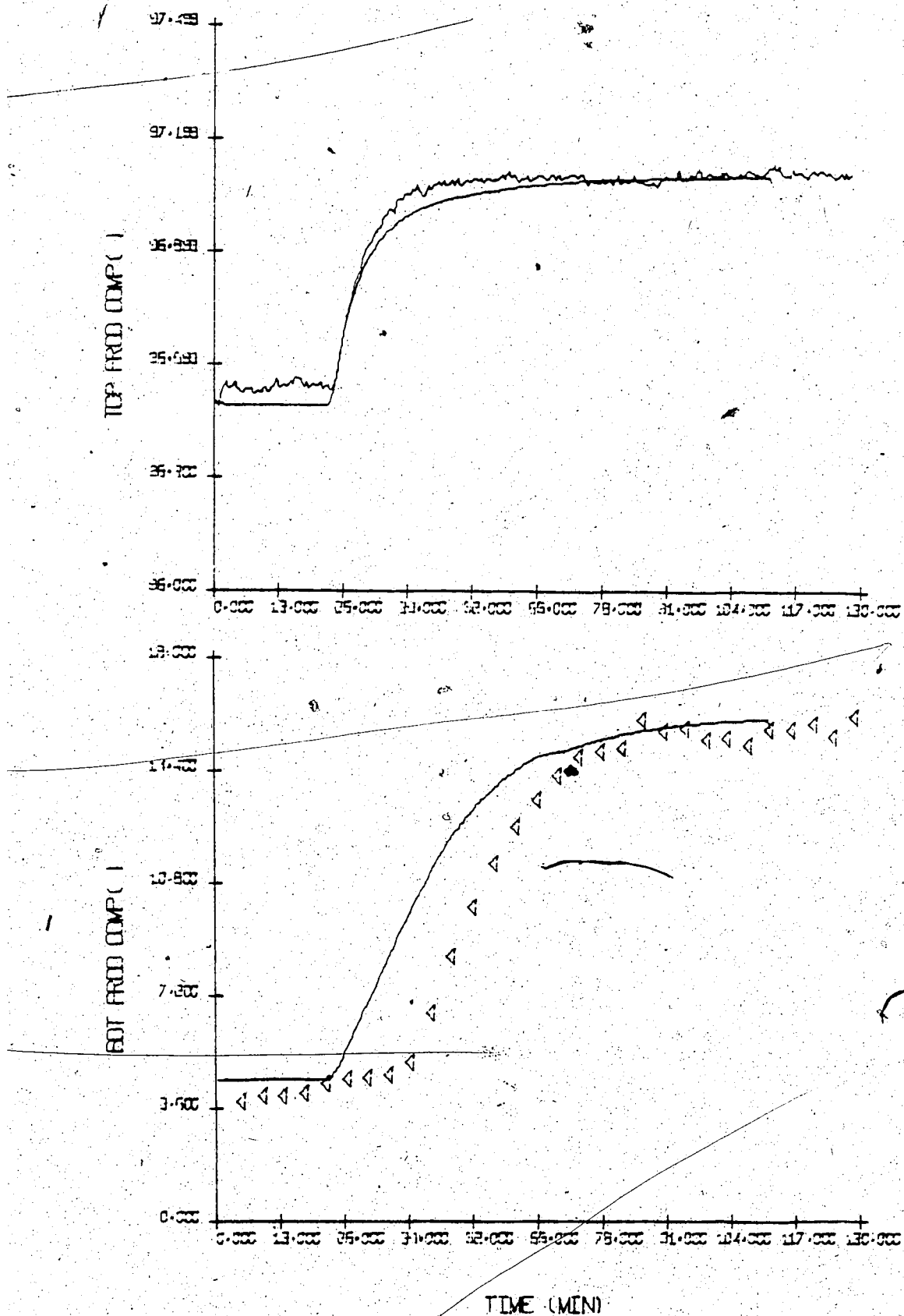


FIGURE 5.15 +20% Reflux flow; Simulation vs Experimental

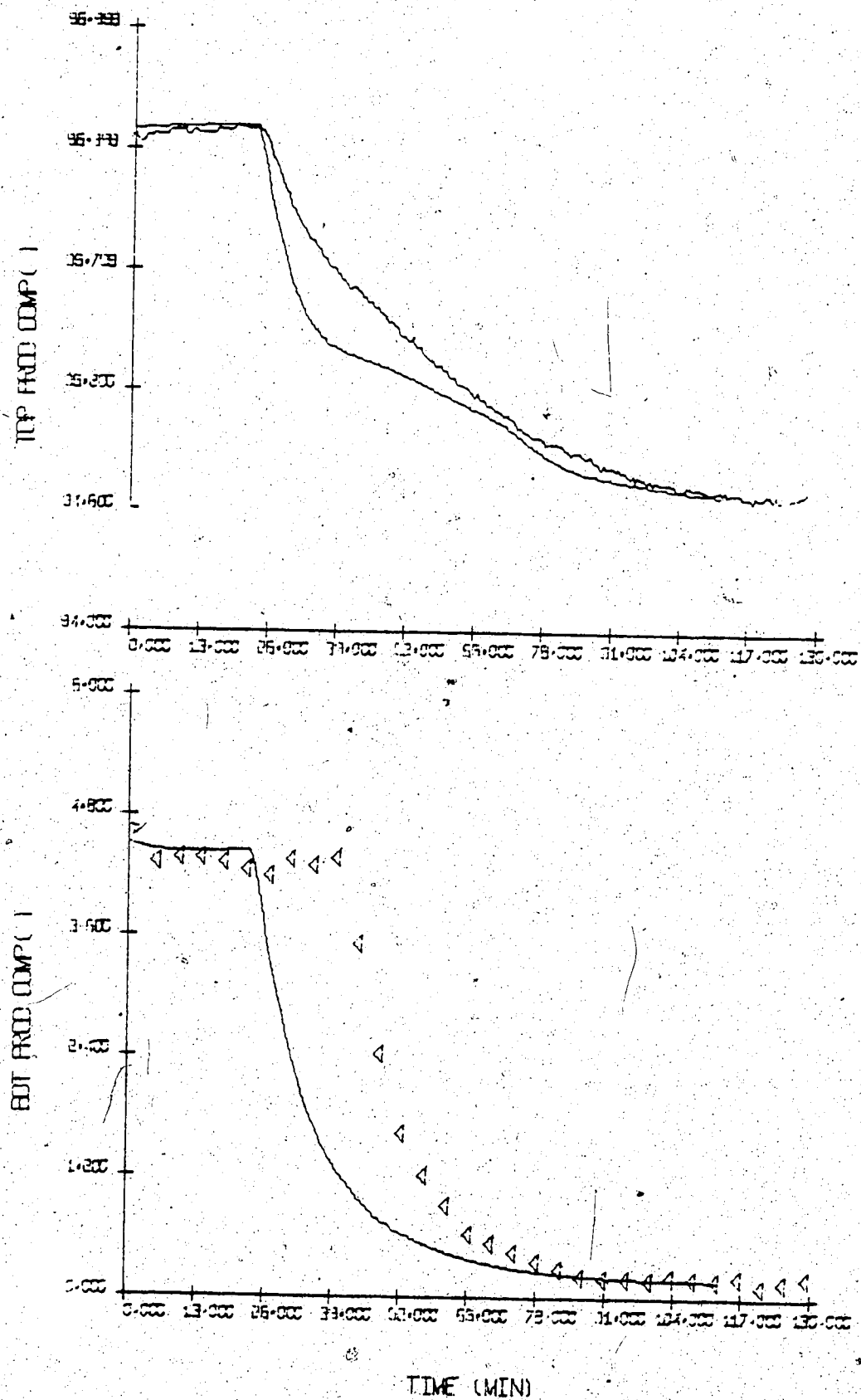


FIGURE 5.16 -20% Reflux flow; Simulation vs Experimental

results, the reader should refer to Chapter 4. Some of the initial experimental steady states vary slightly from run to run, varying by ± 0.05 percent methanol, which is close to instrument resolution. These runs were taken over a period of weeks and although every attempt was made to keep the column operation constant, such slight differences appeared and were considered normal. In simulations, no adjustments were made for this minor difference in composition.

5.3 DISCUSSION

Comparisons between experimental and simulated responses to disturbances may be made in two distinct areas to assess the validity of the model used for the predictions. The first is prediction of terminal compositions at steady state of various operating conditions. The second area for comparison is the column dynamics or rate of change of column compositions due to changes in operating state.

The experimental responses themselves are representatives of duplicate runs. Taken over a period of approximately two weeks, they are deviations from a set of reference steady state conditions decided upon with criteria of column sensitivity and composition measurement accuracy in mind. It may be noted that top composition response to a step increase in feed flow rate is almost undetectable. This was the result of a compromise between column sensitivity at different

operating conditions, and instrument sensitivity.

The steady state conditions resulting from the moderate disturbances in steam, feed and reflux rates from the reference steady state values agree well with the experimental behavior in all cases. The maximum discrepancies may be found in the feed disturbance runs, being of approximately 0.2 percent methanol (or 5% of range) in the bottom composition for both an increase and decrease in flow. The steady states here have been matched more closely by taking greater care in choosing heat loss and efficiency fitting parameters. In these cases, as in all, heat loss was taken as uniform for all trays, whereas tray efficiency was considered uniform throughout the stripping section and again throughout the rectifying section, but both heat loss and efficiency were allowed to vary with terminal composition to match the experimental steady state. Although it was not possible to determine if the efficiency was representative of the experimental value, the heat loss chosen to match the compositions at steady state compared well to the heat loss from the physical column as calculated by the energy balance program BALANC (see Appendix B). The tray efficiency, also used as a fitting parameter, was found to be less effective in this regard than heat loss and could not be determined by measurement but only calculated from column operating data. However, the fitted efficiency values used in the simulations ranged between reasonable limits as

can be seen from Table 4.3.

The transient response dynamics agree very well in all cases for the bottom product and top product composition transients involving reflux rate increases and steam rate decreases. However, the feed disturbances, and other disturbances causing a decrease in methanol concentrations in the distillate require further analysis.

Unique to the feed disturbances is an initial response in a direction opposite to that expected and to the eventual trend. This is especially prominent in the negative feed disturbances, but always only in the distillate response. Although no simulations were run testing the effect of varying efficiency on the stages to vary intermediate compositions, it is expected that the explanation may be found there.

For simplicity, efficiencies were taken as uniform from tray to tray at some value in the stripping section, and at another throughout the rectifying section. This simplification was used by Stainthorp and Searson¹ in a transfer function model study. In this work, a trial and error procedure was used to match the initial steady state. Once this steady state had been matched, these initial efficiencies were used in all simulations. As can be seen from a sample steady state presented in Figure 5.17 the resulting composition profile had a feed tray composition of 51.0 percent methanol.

TIME 5.00(MIN)

STAGE	LCOMP	VCOMP	EFFNCY	LFLOW	VFLOW	HOLDUP	LENTH	VENTH	DWT	DXT	DHL
1	0.045	0.256	1.000	8.61	14.98	6750.0	401.56	2591.22	0.0000	0.0000	0.0
2	0.179	0.510	0.780	23.58	14.64	1552.0	341.73	2489.44	-0.0003	0.0000	-0.0006
3	0.338	0.673	0.780	23.25	15.05	1511.3	262.35	2340.18	0.0000	0.0000	-0.0005
4	0.445	0.761	0.780	23.66	15.84	1475.1	223.20	2176.54	-0.0007	0.0000	-0.0001
5	0.509	0.801	0.780	24.45	16.48	1450.6	201.74	2065.20	0.0001	0.0000	0.0001
6	0.631	0.855	0.930	8.09	17.83	1393.7	179.91	1895.50	-0.0022	0.0000	-0.0001
7	0.757	0.901	0.930	9.43	19.33	1336.9	170.45	1738.71	-0.0060	-0.0000	-0.0000
8	0.851	0.938	0.930	10.93	20.57	1292.0	167.08	1623.28	0.0006	0.0000	0.0000
9	0.919	0.965	0.930	12.17	21.90	1256.0	165.17	1534.28	0.0014	0.0000	-0.0000
10	0.965	0.965	0.0	13.50	0.0	1200.0	164.10	1534.28	UUUUUUUU	-0.0000	0.0000

FEED RATE FEED COMP STEAM FLOW HT COEFF HEAT LOAD

17.00 0.5000 15.00 3597.700 35203.45

FEED ENTH 202.000 REB TEMP 96.000 STEAM TEMP 105.785

Figure 5.17 Sample Steady State Conditions

Thus, a methanol balance about the feed tray shows that the input flow with a composition of only 50.0 percent methanol tends to lower the composition on the feed tray. When this input term is reduced in magnitude, the effect would be to let the feed tray composition rise momentarily, sending a pulse up and down the column. It is postulated that this is the effect that manifests itself in the response shown by the distillate, the same effect being damped out by the large reservoir in the reboiler. This inverse response phenomenon was also observed by Luyben and Vinante¹⁵ on a pilot plant column, but was unexplained, as the study dealt with transfer function models for terminal decoupling. By the same token, a negative response could be predicted for a step increase in feed, under the same initial conditions, as is also observed. These inverse deviations are very slight, only 0.1 percent methanol at the feed tray, but it can be seen from the simulation results that this pulse magnitude is maintained through the rectifying section.

To check this explanation further, it would be necessary to use different values of efficiencies that would predict identical terminal compositions, but different intermediate compositions at steady state, more specifically, a lower composition on the feed tray.

Also showing moderate deviations in dynamics from the experimental responses, and somewhat peculiar curves in the

response, are disturbances resulting in a decrease in distillate composition. Duplication of the response with an alternate integration method and integration time step eliminated the stability of the integration as the fault. A behaviour such as exhibited by the distillate composition under a negative feed flow disturbance would indicate a series of first order systems of varying time constants between the feed tray and the distillate tray. In a matrix system, it would indicate varying eigenvalues in the "rectifying section" of the matrix. These eigenvalues are completely a function of liquid and vapor flow rates and compositions, and would change as compositions change in a nonlinear model.

The peculiarity of some of the responses in question may also be attributed to the choice of efficiency. As previously mentioned, efficiencies were arbitrarily taken as uniform throughout the stripping and rectifying sections. Certainly this forces the simulated tray compositions and flows to assume an arbitrary profile; as vapor and liquid flows, liquid heads and compositions vary from tray to tray, so will actual efficiencies. Each tray may be regarded as a buffer zone at steady state composition, with circulation between buffer zones on either side. There are composition gradients from the reboiler to the condenser and there are circulation rate gradients, with a discontinuity at the feed plate, as the feed is injected there. A buffer zone with a composition of

concentration gradient out of line from the natural gradient would retard or propagate a disturbance in an unnatural way, as its eigenvalue changed in an unnatural way. A changing efficiency or heat loss causing a variation in an eigenvalue, would cause a change in the rate of change in the column.

For example, the heat loss of tray 6 physically changes according to tray 6 temperature, but in the simulation, heat loss is varied with top composition. This may cause the tray 6 liquid flow to react more slowly than in the physical column, and cause the simulated composition to also react more slowly.

All simulations presented with experimental data here are for the case of constant mass holdup. Also, efficiencies and heat loss were taken as functions of distillate composition. For disturbances resulting in a decrease in distillate compositions, the dynamics appear to be faster than shown by the actual column. There may be several reasons for this. In the actual column, holdups vary with changing conditions. As holdups are a determining factor in tray dynamics, changing conditions affect column dynamics; the effects of varying holdups are not presented here, but in Chapter 4 of this work. Also, tray heat losses were varied as functions of distillate composition, but in actuality, are functions of tray temperature. In every type of disturbance, the heat loss on a tray would naturally rise as tray temperature rose,

reducing the magnitude of the disturbance to the next tray, and slowing it down. Again, it was only for simplification purposes that this assumption was made. The model solution scheme would readily accept tray heat loss as a function of tray temperature, and only through further testing can the effects of that simplification be established. The same holds true for tray efficiency, as efficiency was taken as a function of terminal composition also.

It is interesting to note that only in responses involving a decrease in distillate composition, are these irregularities exhibited. The large holdup of the reboiler, five times that of an average tray, effectively damps out the slight deviation for all types of disturbances in the bottom composition transient. In the distillate, disturbances causing a decrease in distillation composition, because of basic nonlinearities of the distillation column are typically more severe than other kinds. For increases in distillate composition then, stronger resistance to changes, or stronger damping, eliminates the effects of peculiar dynamics.

To test these explanations further, testing with alternate efficiency profiles and heat loss correlations must be done. The assumptions of uniform efficiency and heat loss may then be shown to be too restrictive. However, this testing without physical measurements to reference is a trial and error procedure and thus time consuming and expensive.

The simplifications chosen, however, do not limit the generality of the solution scheme of the model, as any efficiency or heat loss profile could have arbitrarily been chosen.

Despite these simplifications, the model predictions compare very well with the experimental data at every operating steady state. In all cases, the bottoms compositions trajectories simulated are very close to those obtained experimentally. Top composition responses for steam decrease and reflux increase disturbances predicted by the model match very well with the experimental responses, and for other disturbances within a reasonable error limit. The solution scheme of this model was thus considered valid, and was then used in comparison studies of models of various degrees of simplification.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

A nonlinear solution scheme to model a binary distillation column and to include the effects of variable mass holdup in the model has been developed and tested for disturbances in feed, reflux and steam flow rate. Comparisons to actual experimental responses of a pilot plant column have shown favorable agreement.

Simulations were performed with the rigorous model altered using several different common simplifying assumptions. The predicted behavior was compared to the prediction of the rigorous model and the effects of the various simplifications were made evident.

To obtain good agreement with experimental responses, it was found that fitting of simulated responses was an integral part of the modelling effort. Tray heat loss and tray efficiency were used effectively as fitting parameters, the column being more sensitive to the heat loss parameter. It was also found that overall column dynamics were strongly influenced by the dynamics of the fitting parameters. It appeared that the dynamics of the reboiler composition were largely determined by the reboiler time constant, other time constants being relatively less significant.

The model was found to be stable in the solution of a nonlinear equation set including the holdup correlation equations. The hydraulic dynamics were found to be the stability limiting element, composition dynamics being much slower. Three types of holdup correlation equations were used in variable mass holdup modelling, and it was found that overall column dynamics could be altered in both directions, depending upon the holdup correlation parameters chosen.

Finally, the solution scheme developed in Chapter 3 has been shown to be creditable, its solution satisfactorily representing actual physical data for disturbances studied, and is easily adaptable to simplification if desired.

6.2 RECOMMENDATIONS

It is recommended that effort towards further distillation column modelling be directed toward the following:

1. Elimination of the fitting parameters. It would be useful to have heat loss and efficiency data in functional form, relating these parameters to operating variables.
2. To further experiment with different holdup correlation parameters, and through simulations attempt to further define their effects upon simulated column dynamics.

In addition, a problem exists in the data gathering of the bottoms product analyses, which should be remedied for further distillation modelling studies. Gas chromatograph analyses must be properly phased with disturbance times and DDC accumulation loop pole times, to reduce the time delays observed in this work.

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NOMENCLATURE

a, b, c, d	- parameters, unique to particular equation
$\underline{\underline{A}}$	- system matrix, defined by composition balance equations
A	- intercept of enthalpy - steam temperature relationship
B	- slope of enthalpy - steam temperature relationship
D	- distillate flow (g/sec)
DT	- integration time interval (sec)
E	- Murphree Vapor Efficiency
$\underline{FT}$	- vector used in solution of vapor and liquid flows
H_n	- vapor enthalpy of vapor leaving stage n (kJ/gm)
h_n	- liquid enthalpy of liquid leaving stage n (kJ/gm)
HSI	- reboiler inlet steam enthalpy (kJ/gm)
HSO	- reboiler outlet steam enthalpy (kJ/gm)
$\underline{\underline{I}}$	- identity matrix
K_n	- ratio of vapor to liquid composition on each tray
L_n	- liquid flow rate leaving stage n (gm/sec)
P	- parameter (heat loss or efficiency)
Q	- heat input, heat loss (kJ/sec)
R	- reflux flow rate (gm/sec)
s	- Laplace domain parameter
S	- sidestream flow rate (g/sec)
S_i	- steam flow rate to the reboiler (g/sec)

S_o	- condensate flow rate from the reboiler (g/sec)
T	- temperature ($^{\circ}\text{C}$)
t	- time (sec)
$\underline{TT}$	- matrix used in solution of liquid and vapor flow rates; has enthalpy and holdup terms
UA	- heat transfer coefficient in reboiler ($\text{kJ/sec}^{\circ}\text{C}$)
V_n	- vapor flow rate leaving stage n (gm/sec)
WT_n	- liquid holdup on stage n (gms)
x	- liquid composition (% methanol)
y	- vapor composition (% methanol)

Subscripts

c	- condenser
i	- initial
f	- final
o	- reference condition
n	- stage number
r	- reboiler

Superscripts

K	- denotes variable value at the Kth time instant
$*$	- equilibrium value

Nomenclature - Distillation Model

$AHLO$	- intercept for steam enthalpy correlation (kJ/gm) $HS = (BHLO * T + AHLO)$
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BHLO	- slope for steam enthalpy correlation (kJ/gm°C)
C1	- liquid coefficient in variable holdup correlation
C2	- vapor coefficient in variable holdup correlation
C3	- intercept in variable holdup correlation
D	- (in ENTH) derivative of liquid enthalpy with respect to liquid composition - (in MAIN) disturbance vector values (gm/sec)
DHL	- derivative of liquid enthalpy with respect to time
DT	- integration time interval (sec)
DWT	- derivative of holdup with respect to time
DXT	- derivative of liquid composition with respect to time
DTL	- derivative of liquid temperature with respect to time
D2	- derivative of tray temperature with respect to liquid composition
EE	- efficiency at the initial steady state
E	- efficiency at final steady state
EF	- efficiency at current time
F	- feed flow rate vector (gm)
FL	- initial feed flow rate (gm)
FT	- (in INTGR) work vector - ratio of vapor to liquid composition, used to obtain tri diagonal matrix - (in VFLOW) work vector used in the solution of flow rates
HF	- feed enthalpy (kJ/kg)
HLT	- liquid enthalpy (kJ/kg)
HVT	- vapor enthalpy (kJ/kg)

HXS	- liquid enthalpy data vector (kJ/kg)
HYS	- vapor enthalpy data vector (kJ/kg)
HSI	- steam enthalpy (kJ/kg)
ICHNG	- vector of disturbance times (min)
I	- disturbance indicator
IN	- printing counter, PRINT index
II	- printing counter, PRINT2 index
ISTRN	- vector of possible disturbances
ITEST	- ASCII characters indicating disturbance type
JM,JN	- variables specifying print times (sec)
KK	- cycle indicator
KT	- indicator for operations occurring on the first program cycle (eg. read data)
LT	- liquid flow rates (gm)
LF	- initial feed flow (gm/sec)
MTT	- number of trays plus reboiler plus condenser
NTT	- number of trays plus reboiler
NL	- number of trays
NI	- simulated run time (min)
QLP	- heat loss at initial steady state (j/sec)
QP	- reboiler heat input (j/sec)
QRP	- heat loss at final steady state (j/sec)
QSP	- heat loss at current state (j/sec)
RE	- initial reflux flow (g/sec)
RR	- ratio of feed to feed tray composition (to obtain a tri diagonal matrix representation)
ROG	- vapor density (g/l)

ROL	- liquid density (g/l)
SF	- steam flow rate (g/sec)
SFF	- steam flow rate at initial steady state
TS	- steam temperature ($^{\circ}\text{C}$)
T	- time (min)
TL	- tray temperature vector ($^{\circ}\text{C}$)
TT	- base matrix in integration scheme
TA, TSU	- matrix in integration scheme
TLS	- temperature-composition data vector ($^{\circ}\text{C}$)
TR	- reboiler liquid temperature ($^{\circ}\text{C}$)
UA	- heat transfer coefficient (j/sec $^{\circ}\text{F}$)
VT	- vapor flow vector (g/sec)
WT	- holdup vector (gm)
XF	- feed composition vector
XFT	- initial feed composition
XIF	- final composition interpolation value (for varying heat loss, efficiency)
XIN	- initial composition interpolation value
XT	- liquid composition vector
XST	- liquid composition data vector
XXT	- printing buffer for top composition
XBT	- printing buffer for bottom composition
YT	- vapor composition vector
YST	- vapor composition - enthalpy data vector
YS	- vapor composition data vector

APPENDIX A

THE METHANOL WATER SYSTEM

TABLE A.1 EQUILIBRIUM, TEMPERATURE DATA

X	Y	$T_L (^{\circ}\text{C})$	$T_V (^{\circ}\text{C})$
0.0000	0.0000	100.0	100.0
0.0350	0.2158	93.3	99.6
0.0689	0.3469	93.6	99.1
0.1020	0.4372	91.4	98.6
0.1339	0.5055	89.1	98.2
0.1650	0.5609	87.5	97.8
0.2026	0.6143	-	-
0.2389	0.6556	84.3	96.4
0.2739	0.6870	-	-
0.3078	0.7100	81.7	94.9
0.3405	0.7331	-	-
0.3772	0.7500	79.6	93.2
0.4028	0.7652	-	-
0.4325	0.7793	77.8	91.6
0.4612	0.7927	-	-
0.4892	0.8043	-	-
0.5162	0.8159	-	-
0.5246	0.8271	75.3	88.8
0.5926	0.8449	-	-
0.6400	0.8624	73.2	84.7
0.7273	0.8934	71.3	80.8
0.8508	0.9224	69.4	76.6
0.8767	0.9504	67.9	72.4
0.9412	0.9759	66.1	68.3
0.9713	0.9881	65.4	66.4
1.0000	1.0000	64.7	64.7

TABLE A.2 EQUILIBRIUM, ENTHALPY DATA

X (wt%)	Y (wt%)	h (kJ/kg)	H (kJ/kg)
0.00	0.00	418.68	2658.6
0.05	0.2373	399.83	2597.0
0.10	0.4370	378.90	2535.3
0.15	0.5342	355.88	2473.9
0.20	0.6140	331.69	2412.5
0.25	0.6656	305.64	2350.9
0.30	0.7110	280.52	2289.2
0.35	0.7382	256.56	2227.8
0.40	0.7652	235.62	2166.2
0.45	0.7874	221.90	2104.8
0.50	0.8087	203.99	2043.2
0.55	0.8337	191.66	1981.5
0.60	0.8476	183.05	1920.1
0.65	0.8659	177.94	1858.5
0.70	0.8837	173.75	1797.1
0.75	0.9018	170.73	1735.4
0.80	0.9224	168.64	1673.8
0.90	0.9596	165.61	1550.7
1.00	1.0000	163.28	1427.7

X - liquid phase methanol composition
 Y - vapour phase methanol composition
 h - liquid phase enthalpy (kJ/kg)
 H - vapour phase enthalpy (kJ/kg)

PHYSICAL PROPERTY CORRELATIONS

The following correlations were converted for use with variables in the SI unit system, from correlations for use with the engineering system of units.

- a) density of liquid water

$$\rho(\text{gm/ml}) = 1.005 - 2.14 \times 10^{-4}T - 2.51 \times 10^{-6}T^2 \quad (\text{A.1})$$

$$40^\circ\text{C} \leq T \leq 120^\circ\text{C}$$

- b) density of liquid methanol

$$\rho(\text{gm/ml}) = 0.808 - 7.74 \times 10^{-4}T - 1.98 \times 10^{-6}T^2 \quad (\text{A.2})$$

$$0^\circ\text{C} \leq T \leq 120^\circ\text{C}$$

- c) density of methanol-water mixture

$$\rho(\text{gm/ml}) = 1.020 - 5.12 \times 10^{-4}T - 1.51 \times 10^{-6}T^2 - 1.52 \times 10^{-3}X - 8.11 \times 10^{-6}X^2$$

$$40^\circ\text{C} \leq T \leq 120^\circ\text{C} \quad (\text{A.3})$$

$$0 \text{ wt\% MeOH} \leq X \leq 100 \text{ wt\% MeOH}$$

- d) density of saturated methanol-water solutions

$$\rho(\text{gm/ml}) = 0.960 - 1.36 \times 10^{-3}X - 7.46 \times 10^{-6}X^2 \quad (\text{A.4})$$

$$0 \text{ wt\% MeOH} \leq X \leq 100 \text{ wt\% MeOH}$$

- e) density of steam

$$\rho(\text{gm/cm}^3) = -1.018 \times 10^{-3} + 3.610 \times 10^{-5}T - 4.292 \times 10^{-7}T^2 + 2.33 \times 10^{-10}T^3$$

$$77^\circ\text{C} \leq T \leq 160^\circ\text{C} \quad (\text{A.5})$$

f) heat of vaporization of water

$$H(\text{kJ/kg}) = 2487.4 - 1.826 \times 10^{-1}T - 4.24 \times 10^{-4}T^2 \quad (\text{A.6})$$

$$66^\circ\text{C} \leq T \leq 150^\circ\text{C}$$

g) heat of vaporization of methanol

$$H(\text{kJ/kg}) = 651 - 1.80T - 1.156 \times 10^{-2}T \quad (\text{A.7})$$

h) heat capacity of steam

$$c_p \text{ cal/gm}^\circ\text{C} = .433 + 1.78 \times 10^{-4}T - 2.68 \times 10^{-6}T^2$$

$$c_p \text{ kJ/kg } ^\circ\text{C} = 1.811 + 7.448 \times 10^{-4}T - 1.12 \times 10^{-5}T^2 \quad (\text{A.8})$$

i) heat capacity of liquid methanol

$$c_p \text{ cal/gm}^\circ\text{C} = .557 + 1.87 \times 10^{-3}T + 9.01 \times 10^{-6}T^2$$

$$c_p \text{ kJ/kg } ^\circ\text{C} = 2.33 + 7.82 \times 10^{-3}T + 3.77 \times 10^{-5}T^2 \quad (\text{A.9})$$

j) heat capacity of vapour methanol

$$c_p \text{ cal/gm}^\circ\text{C} = .3206 + 6.19 \times 10^{-4}T - 4.5 \times 10^{-8}T^2$$

$$- 6.0 \times 10^{-11}T^3$$

$$c_p \text{ kJ/kg } ^\circ\text{C} = 1.34 + 2.59 \times 10^{-3}T - 1.88 \times 10^{-7}T^2$$

$$- 2.5 \times 10^{-10}T^3 \quad (\text{A.10})$$

k) saturated liquid temperatures of methanol-water mixtures

$$T(^{\circ}\text{C}) = 99.61 - .861X + 1.02 \times 10^{-2}X^2$$

$$- 5.18 \times 10^{-5}X^3 \quad (\text{A.11})$$

$$0 \text{ wt\% MeOH} \leq X \leq 100 \text{ wt\% MeOH}$$

- l) saturated vapor temperature of methanol-water mixtures

$$T(^{\circ}\text{C}) = 99.91 - 1.25 \times 10^{-1}X - .994 \times 10^{-3}X^2 - 1.294 \times 10^{-5}X^3 \quad (\text{A.12})$$

$$0 \text{ wt\% MeOH} \leq X \leq 100 \text{ wt\% MeOH}$$

- m) enthalpy of saturated methanol-water vapor

$$H(\text{kJ/kg}) = 2655.3 - 12.29X \quad (\text{A.13})$$

$$0 \text{ wt\% MeOH} \leq X \leq 100 \text{ wt\% MeOH}$$

- n) enthalpy of saturated methanol-water liquid

$$H(\text{kJ/kg}) = 418.7 - 5.75X + 3.15 \times 10^{-2}X^2 \quad (\text{A.14})$$

$$0 \text{ wt\%} \leq X \leq 100 \text{ wt\%}$$

- o) vapor pressure of liquid methanol

$$\log_{10} V_p (\text{mm Hg}) = 7.8786 - 1473.11/(T(^{\circ}\text{C}) + 230) \quad (\text{A.15})$$

$$0^{\circ}\text{C} \leq T \leq 120^{\circ}\text{C}$$

- p) vapor pressure of liquid water

$$\log_{10} V_p (\text{mm Hg}) = 7.9668 - 1668.2/(T + 220) \quad (\text{A.16})$$

$$60^{\circ}\text{C} \leq T \leq 100^{\circ}\text{C}$$

APPENDIX B

ON-LINE AND SIMULATION SOFTWARE

Computer programs used in this work were either used in gathering transient data for open loop runs, steady state information gathering and reporting at the beginning and end of the experimental runs, or were used in the distillation simulations.

Data accumulation and control of all pertinent variables on the distillation column was handled by an on-line IBM 1800 computer with a DDC package. All control or accumulation loops were accessible through a teletype near the pilot plant column. Other loops provided the capability of storing historical information. A program RETRV could access the historical data, type it on a terminal or punch it onto cards for later plotting.

The programs DASS, DATAC and BALNC were used to measure and report column conditions at steady state. These programs were run at the beginning and end of experimental runs to check calibrations of flow and composition measurements by mass, composition and energy balances. These programs were modified from the originals used by other workers at the University of Alberta in that a conversion was made from Engineering units to the new SI system of units in all calculations and report formats.

The gas chromatograph system involved the use of several on-line computer programs. G.C. monitoring and peak analyses were implemented by the IBM G.C. program package available on the IBM 1800 computer used to control the pilot plant column. The package enabled automation of the bottoms product sampling system, and in conjunction with a program DWL44, used peak areas to calculate methanol composition, and provided output on a teletype with each analysis. A special DDC data accumulation loop was updated with each analysis. The GCRAW group of programs, specially supplied by the DACS centre staff, aided in G.C. job definition; the programs were used to scan G.C. output of a typical methanol-water chromatogram and plot the raw data along with calculated first and second derivatives. The plots were then used to choose realistic G.C. job parameters. Documentation of the GCRAW programs is available from DACS centre staff.

All distillation column simulations were run on an IBM 360 and later an AMDAHL 470/V6 operated by the Computing Services department at the University of Alberta. Simulation programs were kept on disk files and were run from a remote terminal.

The source module for the main modelling program was kept under US010.S, and a listing is provided in this Appendix. Modification to implement simplifying assumptions were made to this program, but copies of modified programs were not kept.

Data required by the simulation were kept in three files, one for each type of disturbance examined. The names were US010.d, US020.d, US030.d for reflux flow, steam flow and feed flow disturbances respectively.

Calculated output of the simulation program was kept in several different scratch files with varying formats. When desired, the files were output to cards, for later plotting.

The program EPLLOT was used to plot transient column responses, both experimental and simulated. Several variables could be plotted on one set of coordinates, for comparison purposes.

C THIS PROGRAM USES MASS ENERGY AND COMPONENT BALANCES TO
 C SOLVE FOR COMPOSITION TRANSIENT RESPONSES FOR A 2C STAGE
 C DISTILLATION COLUMN. THE ASSUMPTIONS ARE:
 C 1. NEGLIGIBLE VAPOR HOLDUP
 C 2. PERFECT MIXING
 C 3. CONSTANT EFFICIENCIES

```

1.000      REAL LT(20)
2.000      DIMENSION ISTRN(3)
3.000      DIMENSION D(3), ICHNG(3), RE(3)
4.000      COMMON WT(20), DNL(20), LT(20), VT(20), HVT(20), E(20), XF(3), XIF
5.000      1, EF(10), XIN, QRP(10), DXT(20), TS, DWT(20), KK, TPROD, T, MTT, KT, I, XT(20)
6.000      COMMON VT(20), SF(3), HSI, AHLO, BHLQ, UA, TR, F(3), QLP(20), DT, LF, HE, OP
7.000      COMMON QSP(10), TL(10), DTI(10)
8.000      DATA ISTRN, RE, EF, ST, /
9.000      900 FORMAT(10F8.2)
10.000     901 FORMAT(F8.2, 2I5)
11.000     902 FORMAT(10I5)
12.000     903 FORMAT(10F8.4)
13.000     904 FORMAT(A2)
14.000     HEAD(5, 904) ITESI
15.000     READ(5, 902) ICHNG
16.000     READ(5, 903) D
17.000     READ(5, 902) LF
18.000     READ(5, 901) DT, NJ, NIT
19.000     MTT=NTT+1
20.000     READ(5, 900) LT(NTT)
21.000     READ(5, 903) XIF, XIN
22.000     READ(5, 900) FL, XFT, HF
23.000     READ(5, 900) SFF, HSI, BHLQ, AHLO, UA, TR
24.000     KT=0
25.000     JME=JM/DT
26.000     JN=JN/DT
27.000     IN=0
28.000     II=0
29.000     NI=60*NI/DT
30.000     DO 5, I=1, 3
31.000     IF (ISTRN(I)-ITEST) 5, 7, 5
32.000     5 CONTINUE
33.000     7 GO TO (6, 8, 9), I
34.000     6 DO 61 I=1, 3
35.000     ICHNG(I)=60*ICHNG(I)/DT
36.000     XF(I)=XF
37.000     F(I)=F
38.000     SF(I)=SFF
39.000     61 RE(I)=D(I)
40.000     GO TO 11
41.000     8 DO 81 I=1, 3
42.000     ICHNG(I)=60*ICHNG(I)/DT
43.000     XF(I)=XF
44.000     SF(I)=SFF
45.000     81 F(I)=D(I)
46.000
47.000
48.000
49.000
50.000
51.000
52.000
53.000
54.000
55.000
56.000
57.000
58.000

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```

00000000 C047      GO TO 11
00000000 C048      9 DO 91 I=1,3
00000000 C049      ICING(I)=ICING(I)*0/DT
00000000 C050      RE(I)=LT(MIT)
00000000 C051      F(I)=FL
00000000 C052      XF(I)=XFT
00000000 C053      91 SF(I)=0(I)
00000000 C054      11 CONTINUE
00000000 C055      I=1
00000000 C056      CALL INEL
00000000 C057      KT=2
00000000 C058      CALL PRINT
00000000 C059      DO 100 KK=1,N1
00000000 C060      T=KK*DT/60.
00000000 C061      DO 23 JJ=1,3
00000000 C062      IF (KK=ICING(JJ))23,22,23
00000000 C063      22 I=JJ
00000000 C064      23 CONTINUE
00000000 C065      LT(MIT)=RE(I)
00000000 C066      CALL COMP
00000000 C067      CALL ENTH
00000000 C068      CALL YFLCW
00000000 C069      CALL INTGR
00000000 C070      KT=2
00000000 C071      11=11+1
00000000 C072      IN=IN+1
00000000 C073      IF (11-JM)20,21,20
00000000 C074      21 11=0
00000000 C075      CALL PRIN2(JM)
00000000 C076      20 IF (IN-JN)30,31,30
00000000 C077      31 IN=0
00000000 C078      CALL PRINT
00000000 C079      30 CONTINUE
00000000 C080      100 CONTINUE
00000000 C081      CALL EXIT
00000000 C082      END

```

```

0001 95.000 SURROUTINE INTGR
0002 96.000 REAL LT(2)
0003 97.000 DIMENSION FY(10),TSU(10,10),TA(10,10)
0004 98.000 DIMENSION TT(10,10),FX(10),FT(10)
0005 99.000 COMMON WT(20),DHL(20),LT(20),VT(20),HVT(20),E(20),XF(3),XIF
0006 100.000 1,EF(10),XIN,ORP(10),DAT(20),TS,DWT(20),KK,TPROD,T,MTT,KT,I,XI(20)
0007 101.000 COMMON XI(20),SF(3),HSI,AHLO,BILO,UA,TR,F(3),OLP(20),DT,LF,IF,QP
0008 102.000 COMMON QSP(10),IL(10),DTL(10)
0009 103.000 100 FORMAT(2X,'EIGENVALUES:')
0010 104.000 101 FORMAT(2X,'REAL PART:',10F6.2)
0011 105.000 102 FORMAT(2X,'IMAG PART:',10F6.2)
0012 106.000 MTT=MTT-1
0013 107.000 NL=MTT-2
0014 108.000 11 DO 20 N=1,MTT
0015 109.000 DO 20 M=1,MTT
0016 110.000 TT(N,M)=0.
0017 111.000 TSU(N,M)=0.
0018 112.000 20 TA(N,M)=0.
0019 113.000 DO 10 N=1,MTT
0020 114.000 10 FT(N)=VT(N)/XT(N)
0021 115.000 RR=XF(1)/XT(LF)
0022 116.000 DO 1 N=2,MTT
0023 117.000 M=N-1
0024 118.000 TT(N,M)=(VT(N)*(1-FI(N))-VT(N-1)-LT(N+1))*DT/(WT(N)*2)
0025 119.000 TT(M,M)=LT(M)*DT/(WT(M)*2)
0026 120.000 1 TT(N,M)=VT(M)*FT(M)*DT/(WT(N)*2)
0027 121.000 TT(1,1)=(VT(1)*(1-FI(1))-LT(2))*DT/(WT(1)*2)
0028 122.000 TT(LF,LF)=TT(LF,LF)+BB*F(1)-F(1))*DT/(WT(LF)*2)
0029 123.000 TT(MTT,MTT)=VT(MTT)*DT/(WT(MTT)*2)
0030 124.000 CALL GMPROD(TT,XI,DAT,MTT,MTT,1)
0031 125.000 DO 2 N=1,MTT
0032 126.000 TA(N,N)=1+TT(N,N)
0033 127.000 2 TSU(N,N)=1+TT(N,N)
0034 128.000 DO 3 N=1,MTT
0035 129.000 TA(N,N+1)=TT(N,N+1)
0036 130.000 TA(N+1,N)=TT(N+1,N)
0037 131.000 TSU(N,N+1)=TT(N,N+1)
0038 132.000 TSU(N+1,N)=TT(N+1,N)
0039 133.000 3 CONTINUE
0040 134.000 C INTEGRATION
0041 135.000 CALL MINV(TSU,MTT,D,FX,FT)
0042 136.000 CALL GMPROD(TSU,TA,TT,MTT,MTT,MTT)
0043 137.000 CALL GMPROD(TT,XI,FT,MTT,MTT,1)
0044 138.000 DO 4 N=1,MTT
0045 139.000 4 XT(N)=FI(A)
0046 140.000 DO 7 N=2,MTT
0047 141.000 DWT(N)=LT(N+1)-LT(N)-VT(N)-VT(N+1)
0048 142.000 WT(N)=DT*DWT(N)+WT(N)
0049 143.000 7 CONTINUE
0050 144.000 WT(LF)=WT(LF)+DT*F(1)
0051 145.000 DWT(LF)=DWT(LF)+F(1)
0052 146.000 RETURN
0053 147.000 END
0054 148.000
0055 149.000
0056 150.000
0057 151.000

```

```

152.000 0001 SUBROUTINE INIT
153.000 0002 REAL LT(20)
154.000 0003 DIMENSION ROL(10)
155.000 0004 COMMON WT(20),DHL(20),LT(20),HVT(20),E(20),XF(3),XIF
156.000 0005 1,EF(10),XIN,ORP(10),DXT(20),IS,DWT(20),KK,IPROD,I,MT,KT,I,XI(20)
157.000 0006 COMMON YT(20),SF(3),HSL,AHLO,BHLO,UA,TR,F(3),OLP(20),DT,LF,HF,OP
158.000 0007 COMMON OSP(10),TL(10),DTL(10)
159.000 0008 NIT=MTT-1
160.000 0009 E(MIT)=0.
161.000 0010 VT(MIT)=0.
162.000 0011 READ(5,100)(XT(N),N=1,MTT)
163.000 0012 READ(5,101)(LT(N),N=1,MTT)
164.000 0013 READ(5,101)(VT(N),N=1,MTT)
165.000 0014 READ(5,101)(WT(N),N=1,MTT)
166.000 0015 READ(5,101)(DLP(N),N=1,MTT)
167.000 0016 READ(5,101)(ORP(N),N=1,MTT)
168.000 0017 DO 1 N=1,MTT
169.000 0018 DXT(N)=0.
170.000 0019 DO 1 N=1,MTT
171.000 0020 1 DHL(N)=0.
172.000 0021 1 DHL(N)=0.
173.000 0022 CALL COMP
174.000 0023 DO 10 N=2,MTT
175.000 0024 ROL(N)=(1000-136*XT(N)-74.6*XT(N)*#2)
176.000 0025 WT(N)=(LT(N)/1.206)*.667*5.0)*.316*ROL(N)
177.000 0026 DWT(N)=LT(N+1)+VT(N-1)-LT(N)-VT(N)
178.000 0027 10 CONTINUE
179.000 0028 DWT(1)=LT(2)-LT(1)-VT(1)
180.000 0029 DWT(LF)=DWT(LF)+F(1)
181.000 0030 CALL ENTH
182.000 0031 KT=2
183.000 0032 CALL PRINT
184.000 0033 CALL VFLOW
185.000 0034 100 FORMAT(10F8.4)
186.000 0035 101 FORMAT(10F8.3)
187.000 0036 RETURN
188.000 0037 END

```

```

193.000 0001      SURROUTINE ENTN
194.000 0002      REAL LT(20)
195.000 0003      DIMENSION D(10),XST(19),YST(19),HXS(19),HYS(19)
196.000 0004      COMMON MT(20),DHL(20),LI,VT(20),HVT(20),E(20),XF(3),XIF
197.000 0005      1,EF(10),XIN,ORP(10),DXT(20),TS,DWT(20),KK,IPROD,I,MT,KI,1,XI(20)
198.000 0006      COMMON YTI(20),SF(3),HSI,AHLO,BHLO,UA,TR,F(3),QLP(20),OT,LF,MF,OP
199.000 0007      COMMON QSP(10),TL(10),DTL(10)
200.000 0008      100 FORMAT(10F8.4)
201.000 0009      101 FORMAT(10F8.3)
202.000 0010      102 FORMAT(10F8.2)
203.000 0011      105 FORMAT(2X,F6.4,4X,F7.2,3X,F6.4,4X,F8.2)
204.000 0012      IF(KI-2)1,4,4
205.000 0013      1 READ(5,100)XST
206.000 0014      READ(5,100)YST
207.000 0015      READ(5,101)HXS
208.000 0016      READ(5,102)HYS
209.000 0017      4 K=1
210.000 0018      DO 10 N=1,MTT
211.000 0019      DO 20 J=K,19
212.000 0020      IF(XST(J)-XT(N)20,20,2
213.000 0021      20 CONTINUE
214.000 0022      2 K=J-1
215.000 0023      L=J
216.000 0024      D(N)=(HXS(L)-HXS(K))/(XST(L)-XST(K))
217.000 0025      HLT(N)=D(N)*(XT(N)-XST(K))+HXS(K)
218.000 0026      10 CONTINUE
219.000 0027      K=1
220.000 0028      DO 30 N=1,MTT
221.000 0029      DO 40 J=K,19
222.000 0030      IF(YST(J)-YT(N)40,40,3
223.000 0031      40 CONTINUE
224.000 0032      3 K=J-1
225.000 0033      L=J
226.000 0034      HVT(N)=(YT(N)-YST(K))/(HYS(L)-HYS(K))/(YST(L)-YST(K))+HYS(K)
227.000 0035      30 CONTINUE
228.000 0036      7 DO 9 N=1,MTT
229.000 0037      9 DHL(N)=D(N)+DXT(N)
230.000 0038      RETURN
231.000 0039      END

```

```

232.000 C001      SUBROUTINE COMP
233.000 C002      REAL LT(20)
234.000 C003      DIMENSION YST(20),DHL(20),LT,VLT(20),HVT(20),E(20),XF(3),XIF
235.000 C004      COMMON WT(20),XIN,ORP(10),DXT(20),TS,DWT(20),KK,IPROD,T,MTT,KT,I,XT(20)
236.000 C005      COMMON YT(20),SF(3),HST,AHLO,UHLO,UA,TR,F(3),QLP(20),DT,LF,HF,OP
237.000 C006      COMMON QSPI(10),IL(10),DTL(10)
238.000 C007      NIT=MTT-1
239.000 C008      101 FORMAT(10F8.3)
240.000 C009      103 FORMAT(/,'*****EQUILIBRIUM DATA*****')
241.000 C010      104 FORMAT(10X,'LCOMP,10X,'VCOMP,1
242.000 C011      107 FORMAT(10X,F8.5,F8.5)
243.000 C012      IF(KT-211.3,3
244.000 C013      1 READ(5,101)XST
245.000 C014      READ(5,101)YS
246.000 C015      READ(5,101)TLS
247.000 C016      READ(5,101)EE
248.000 C017      WRITE(6,103)
249.000 C018      WRITE(6,104)
250.000 C019      DO 98 N=1,26
251.000 C020      WRITE(6,107)XST(N),YS(N)
252.000 C021      98 CONTINUE
253.000 C022      3 J=1
254.000 C023      DO 20 K=1,MTT
255.000 C024      DO 10 N=J,26
256.000 C025      IF(XST(N)-XT(K))10,10,2
257.000 C026      10 CONTINUE
258.000 C027      2 J=N+1
259.000 C028      L=N
260.000 C029      D2(K)=(TLS(L)-TLS(J))/(XST(L)-XST(J))
261.000 C030      TL(K)=TLS(J)+XT(K)-XST(J))*D2(K)
262.000 C031      YST(K)=(((XT(K)-XST(J))*YS(L)-YS(J))/(XST(L)-XST(J)))+YS(J)
263.000 C032      909 FORMAT(10X,'XT',F12.4,'YST',F12.4)
264.000 C033      20 CONTINUE
265.000 C034      YT(1)=E(1)*YST(1)
266.000 C035      DO 21 N=1,NIT
267.000 C036      J=N+1
268.000 C037      DTL(N)=D2(N)*XT(N)
269.000 C038      EF(J)=E(J)*(EF(J)-E(J))*((XT(MTT)-XIN)/(XIF-XIN)
270.000 C039      21 YT(J)=EF(J)*(YST(J)-YT(N))+YT(N)
271.000 C040      EF(1)=E(1)
272.000 C041      RETURN
273.000 C042      END
274.000 C042

```

```

275.000 C001
276.000 C002
277.000 C003
278.000 C004
279.000
280.000 C005
281.000
282.000 C006
283.000 C007
284.000 C008
285.000 C009
286.000 C010
287.000 C011
288.000 C012
289.000 C013
290.000 C014
291.000 C015
292.000 C016
293.000 C017
294.000 C018
295.000 C019
296.000 C020
297.000 C021
298.000 C022
299.000 C023
300.000 C024
301.000 C025
302.000 C026

SUBROUTINE PRIN2(JM)
REAL LT(20)
DIMENSION XAT(20),XBT(20)
COMMON WT(20),DIL(20),LT,VT(20),HLI(20),HVT(20),E(20),XF(3),XIF
COMMON YI(20),XIN,ORP(10),DXT(20),TS,DWT(20),KK,TPROD,I,MTI,KI,I,XI(20)
COMMON QSP(10),TL(10),DIL(10)
100 FORMAT(10F8.6)
IF(KK-JM)1,1,4
1 NN=0
4 CONTINUE
WRITE(7,100)(XT(N),N=1,MTT)
DO 20 N=1,9
XAT(N)=XBT(N+1)
20 XAT(N)=XAT(N+1)
XAT(10)=XAT(11)+100
XAT(10)=XT(MTT)*100
NN=NN+1
102 FORMAT(10I8)
IF (NN-10)30,40,40
40 WRITE(8,101)(XAT(N),N=1,10)
WRITE(9,101)(XBT(N),N=1,10)
NN=0
30 CONTINUE
101 FORMAT(10F8.3)
RETURN
END

```

```
302.000 0001 SUBROUTINE PRINT
303.000 0002 REAL LT(20)
304.000 0003 COMMON WT(20),DHL(20),LT,VT(20),MLT(20),HVT(20),E(20),XF(3),XIF
305.000 1,EF(10),XIN,QRP(10),DXT(20),TS,DWT(20),KK,IPROD,T,MTI,KI,I,XI(20)
306.000 0004 COMMON YT(20),SF(3),RSI,AHLQ,BHLO,UA,TR,F(3),QLP(20),DT,LF,HF,OP
307.000 0005 COMMON QSP(10),TL(10),DTL(10)
308.000 0006 101 FORMAT(' ',20X,'TIME',F8.2,'(MIN)')
309.000 0007 102 FORMAT(' ',2X,'STAGE',2X,'LCOMP',2X,'VCOMP',2X,'EFFNCY',2X,'LFLOW',2
310.000 1X,'VFLOW',2X,'HOLDUP',2X,'LENTH',2X,'VENTH',4X,'DWT',5X,'TL',
311.000 25X,'QSP:')
312.000 0008 103 FORMAT(' ',16,F7.3,F7.3,F8.3,F8.2,F7.2,F8.1,F9.2,F8.2,F8.4,F8.1
313.000 1,F8.2)
314.000 0009 104 FORMAT(' ',5X,'FEED RATE',2X,'FEED COMP',2X,'STEAM FLOW',2X,'HT COEF
315.000 IF',2X,'HEAT LOAD')
316.000 0010 105 FORMAT(' ',F11.2,F11.4,F12.2,F12.3,F15.2)
317.000 0011 106 FORMAT(' ',2X,'FEED ENTH',F9.3,3X,'RED TEMP',F9.3,5X,'STEAM TEMP',F9
318.000 1,3)
319.000 0012 WRITE(6,101)T
320.000 0013 WRITE(6,102)
321.000 0014 DO I=N-1,MTI
322.000 0015 I WRITE(6,103)N,XI(N),YT(N),EF(N),LT(N),VT(N),MLT(N),HVT(N),
323.000 1,DWT(N),TL(N),QSP(N)
324.000 0016 WRITE(6,104)
325.000 0017 WRITE(6,105) F(1),XF(1),SF(1),UA,OP
326.000 0018 WRITE(6,106)HF,TR,TS
327.000 0019 RETURN
328.000 0020 END
```

```

329.000 0001 SUBROUTINE VFLOW
330.000 0002 READING(20)
331.000 0003 DIMENSION TT(18,18),FT(18),C1(9),C2(9),C3(9)
332.000 0004 DIMENSION FX(18),FY(18),ROG(9),ROL(9)
333.000 0005 COMMON WT(20),OHL(20),LT,VT(20),HVT(20),E(20),XF(3),XIF
334.000 0006 1,EF(10),XIN,CRP(10),OXT(20),TS,DWT(20),KK,TPROD,Y.MTT,KT,I,XI(20)
335.000 0007 COMMON YT(20),SF(3),HSI,AILO,BILO,UA,TR,F(3),OLP(20),DT,LF,HF,OP
336.000 0008 COMMON QSP(10),TL(10),DIL(10)
337.000 0009 NNT=MIT-1
338.000 0010 NL=MIT-2
339.000 0011 NNT=NTT*2
340.000 0012 KS=0
341.000 0013 HW=5.0
342.000 0014 Z=15.0
343.000 0015 DO 11 KL=1,18
344.000 0016 DO 11 J=1,18
345.000 0017 11 TT(KL,J)=0.
346.000 0018 C STEAM TEMPERATURE
347.000 0019 TS=(SF(1)*(HSI-AILO)+UA*TR)/(UA+B*HLO+SF(1))
348.000 0020 C HEAT INPUT
349.000 0021 OP=UA*(TS-TR)
350.000 0022 DO 6 N=1,NTT
351.000 0023 QSP(N)=OLP(N)+(QRP(N)-OLP(N))*(XT(MTT)-XIN)/(XIF-XIN)
352.000 0024 C HOLDUP CORRELATION PARAMETERS
353.000 0025 ROL(N)=(1000-136*XT(N)-74.6*XT(N)*T2)
354.000 0026 C1(N)=(-.316+6*ROL(N)*(LT(N)/1.206)**.667)/LT(N)
355.000 0027 C2(N)=0.00
356.000 0028 C3(N)=58*ROL(N)*.317
357.000 0029 6 CONTINUE
358.000 0030 TT(1,1)=-1
359.000 0031 TT(1,2)=1.
360.000 0032 TT(1,10)=-1.
361.000 0033 DO 3 N=2,NTT
362.000 0034 TT(N,N+NTT)=C2(N)
363.000 0035 TT(N,N)=C1(N)
364.000 0036 3 CONTINUE
365.000 0037 DO 4 N=2,NTT
366.000 0038 M=N+NL
367.000 0039 TT(M,M)=HLT(N)-HVT(N)
368.000 0040 4 TT(M,M-1)=HVT(N-1)-HLT(N)
369.000 0041 TT(MT,MT)=HLT(1)-HVT(1)
370.000 0042 101 FORMAT(RF10.2)
371.000 0043 C CALCULATE THE VAPOUR FLOWS
372.000 0044 FT(1)=0.
373.000 0045 FT(MT)=-LT(MTT)
374.000 0046 DO 2 N=1,NL
375.000 0047 M=N+1
376.000 0048 FT(M)=WT(M)-C3(M)
377.000 0049 NN=N+NTT
378.000 0050 2 FT(NN)=WT(N)+OHL(N)+QSP(N)+5.42*DIL(N)
379.000 0051 FT(MT)=FT(MT)-OP
380.000 0052 N=NTT+LF
381.000 0053
382.000 0054
383.000 0055
384.000 0056
385.000 0057
386.000 0058

```

```
387.000 0051 FT(N)=FT(N)-F(I)*(HF-HLT(LF))
388.000 0052 NN=2*NTT
389.000 0053 FT(NN)=WT(NTT)+DHL(NTT)-LT(MTT)*(HLT(MTT)-HLT(NTT))
390.000 0054 CALL SIMQ(IT,FT,NT,KS)
391.000 0055 DO 30 N=1,NTT
392.000 0056 M=NT+N
393.000 0057 30 VT(N)=FT(M)
394.000 C CALCULATE THE LIQUID FLOWS
395.000 DO 40 N=1,NTT
396.000 40 LT(N)=FT(N)
397.000 RETURN
398.000 0061 END
```

[illegible]

7woodius020.n

STEAM DISTURBANCE

00 93 105
15.0 16.5 16.5

6.00 110 9

13.5

480.00 200.00

17.0 0.50 202.00

13.00 2787.08 4.18

32 300

0.0450680, 1783480, 17.0640, 4449050, 5089480, 6303990, 7585420, 8513220, 9187240, 964965

8.60 23.58 23.24 23.65 24.44 8.08

14.98 14.64 15.05 15.84 16.48 17.83

6750.000

1000.0 000.0 000.0 000.0 000.0 000.0

1000.0 700.0 700.0 700.0 700.0 700.0

0.9999 0.7800 0.7800 0.7800 0.7800 0.7800

0.0000 0.0350 0.0687 0.1020 0.1339 0.1650

0.3405 0.3722 0.4028 0.4325 0.4612 0.4892

0.7273 0.8058 0.8767 0.9412 0.9743 1.0000

0.0000 0.2158 0.3469 0.4372 0.5055 0.5609

0.7331 0.7500 0.7652 0.7793 0.7927 0.8043

0.8934 0.9224 0.9504 0.9759 0.9881 1.0000

100.0 93.33 93.61 91.39 89.05 87.50

80.67 79.61 78.78 77.78 77.17 76.53

71.33 69.44 67.9 66.11 65.44 64.70

0.9999 0.9200 0.9200 0.9200 0.9200 0.9200

0.00 0.05 0.10 0.15 0.20 0.25

0.50 0.55 0.60 0.65 0.70 0.75

0.00 0.2373 0.4370 0.5342 0.6140 0.6656

0.8087 0.8337 0.8476 0.8659 0.8837 0.9018

418.68 397.83 378.90 355.88 331.69 305.64

203.99 171.66 183.05 177.94 173.73 170.73

2658.6 2577.0 2535.3 2473.9 2412.5 2350.9

2043.2 1981.5 1920.1 1858.5 1797.1 1735.4

0.00 0.00 0.00 0.00 0.00 0.00

0.00 0.00 0.00 0.00 0.00 0.00

0.00 0.00 0.00 0.00 0.00 0.00

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APPENDIX C

DERIVATIONS

The solution schemes described in Chapter 3 had the objective of solving the system of 40 equations, or 4 systems of 10 equations, shown below, where "n" denotes the stage.

a) composition balances

$$\frac{dx_n}{dt} = \frac{1}{WT_n} [L_{n+1}x_{n+1} - L_nx_n - V_ny_n + V_{n-1}y_{n-1} - x_n \frac{dWT_n}{dt}] \quad (C.1)$$

b) total balances

$$\frac{dWT_n}{dt} = L_{n+1} - L_n - V_n + V_{n-1} \quad (C.2)$$

c) enthalpy balances

$$\frac{dh_n}{dt} = \frac{1}{WT_n} [L_{n+1}h_{n+1} - L_nh_n - V_nH_n - h_n \frac{dWT_n}{dt} - QLP] \quad (C.3)$$

d) holdup correlations

$$WT_n = a_nL_n + b_nV_n + c_n \quad (C.4)$$

These equations are not rigorous, but specific to this work; the limiting assumptions used in this model have already been applied. These equations apply to the general equilibrium stage. Terms must be added or deleted for special

stages such as feed tray, reboiler and condenser. The column parameters x, y, L, V and WT are all functions of time t . The following solution schemes attempt to solve this system of 40 equations, although by two different methods.

C.1 SUBSTITUTION METHOD

In both the substitution method and matrix inversion methods, the coefficients of the composition balances were determined from the remaining three systems of equations. It was the composition balances that were directly integrated to obtain the trajectories of terminal and intermediate column compositions.

The energy and total balances were solved together to obtain preliminary liquid and vapor flow rates. The coefficients of these two sets of equations are enthalpies (directly obtainable from current compositions), holdups and holdup derivatives obtained from the previous time step. Holdups and holdup derivatives were recalculated in a correction step, where the preliminary flow rates were substituted into the holdup correlation equations (C.4). The new holdups, accumulations and flow rates were then used as coefficients in the next integration step.

In solving for the 20 liquid and vapor flow rates, 20 equations were used. Specified variables were reflux flow,

feed flow, reflux and feed enthalpies and heat input to the reboiler. The method for accomplishing this is outlined below, and mainly involves consecutive substitutions of expressions of variables in terms of each other, until an expression for one variable (V_9) in terms of known quantities is obtained.

The systems of equations used to solve for liquid and vapor flow rates are Equations (C.2) and (C.3). The derivative terms are directly obtainable from information from the previous time step, where K denotes the time step.

$$\frac{dWT_n^K}{dt} = \frac{1}{DT} (WT_n^K - WT_n^{K-1})$$

$$\frac{dh_n^K}{dt} = C_n \frac{dx_n^K}{dt}$$

The coefficient C_n is the slope of the enthalpy-composition relationship, and continually varies with composition.

The liquid flows are eliminated from Equations (C.3) by substituting equations from (C.2).

stage 9

$$V_8 H_8 + L_{10} h_{10} - L_9 h_9 - V_9 H_9 - QLP_9 = WT_9 Dh_9 + h_9 DWT_9$$

substituting for L_9

$$\begin{aligned} V_8 H_8 + L_{10} h_{10} - h_9 (V_8 + L_{10} - V_9 - DWT_9) - V_9 H_9 - QLP_9 \\ = WT_9 Dh_9 + h_9 DWT_9 \end{aligned}$$

or

$$V_8(H_8 - h_9) + L_{10}(h_{10} - h_9) - V_9(H_9 - h_9) - QLP \\ = WT_9 Dh_9$$

Following the same procedure for stage 8

$$V_7(H_7 - h_8) + L_9(h_9 - h_8) - V_8(H_8 - h_8) - QLP_8 \\ = WT_8 Dh_8$$

but

$$L_9 = V_8 + L_{10} - V_9 - DWT_9$$

substituting

$$V_7(H_7 - h_8) + V_8(h_9 - H_8) + L_{10}(h_9 - h_8) - V_9(h_9 - h_8) \\ - QLP = WT_8 Dh_8 + (h_9 - h_8) DWT_9$$

One can continue this procedure for all stages, including terms for the feed flow rate and the reboiler heat duty in the equations for stages below the feed stage and in the reboiler equation respectively.

If all non-vapor flow terms are collected on the right hand side of the equations obtained, and all terms in vapor flow on the left hand side, a matrix representation can be formulated. Let $\underline{V}$ be a vector of vapor flows and let $\underline{F}$ be a vector of terms on the right hand sides of the resultant equations.

$$\underline{A} \underline{V} = \underline{F}$$

(C.5)

where

$$f_n = QLP_n + (h_{n+1} - h_n) \sum_{i=n}^9 DWT_i + L_{10}(h_{n+1} - h_n) + WT_n Dh_n - F(h_{n+1} - h_n) \quad n = 1, 5 \quad (C.6)$$

and

$$f_n = QLP_n + (h_{n+1} - h_n) \sum_{i=n}^9 DWT_i + L_{10}(h_{n+1} - h_n) + WT_n Dh_n \quad n = 6, 9 \quad (C.7)$$

and where the matrix A has the form

$$\underline{A} = \begin{bmatrix} a_1 & & & & & & & & c_1 \\ b_1 a_2 & & & & & & & & c_2 \\ & b_2 a_3 & & & & & & & c_3 \\ & & b_3 & & & & & & . \\ & & & . & . & & & & . \\ & & & & . & . & & & . \\ & & & & & . & . & & . \\ & & & & & & a_7 & & . \\ & & & & & & & b_7 a_8 c_8 & \\ & & & & & & & & b_8 a_9 \end{bmatrix} \quad (C.8)$$

The elements in matrix $\underline{A}$ are:

$$a_n = h_{n+1} - h_n$$

$$b_n = -a_n$$

$$c_n = h_n - h_{n+1}$$

All terms in the $\underline{F}$ vector defined above are known, and the $\underline{F}$ vector may be fully calculated. The $\underline{A}$ matrix is also fully defined as all enthalpies are known. All liquid flow terms (except reflux flow L_{10}) have been eliminated from the system of equations represented by (C.5). With further substitution, an expression for the vapor flow from stage 9 may be found, in terms of known quantities.

From Equations (C.5) and (C.8)

stage 1

$$f_1 = a_1 V_1 + b_1 V_9$$

or

$$V_1 = \frac{f_1}{a_1} - \frac{b_1 V_9}{a_1}$$

stage 2

$$f_2 = -a_1 V_1 + a_2 V_2 + b_2 V_9$$

$$V_2 = \frac{f_2}{a_2} + \frac{a_1 V_1}{a_2} - \frac{b_2 V_9}{a_2}$$

substituting for V_1

$$V_2 = \frac{f_2}{a_2} + \frac{f_1}{a_2} - \frac{b_1 V_9}{a_2} - \frac{b_2 V_9}{a_2}$$

similarly for stage n

$$V_n = \frac{1}{a_n} \left(\sum_{i=1}^n f_i - \sum_{i=1}^n b_i V_9 \right)$$

$$n = 1, 8$$

for stage 9 however,

$$f_9 = a_9 V_9 - a_8 V_8$$

$$V_9 = \frac{f_9}{a_9} + \frac{a_8 V_8}{a_9}$$

$$= \frac{f_9}{a_9} + \frac{1}{a_9} \left(\sum_{i=1}^8 f_i - \sum_{i=1}^8 b_i V_9 \right)$$

solving for V_9

$$V_9 = \left(\sum_{i=1}^9 f_i \right) / \left(a_9 + \sum_{i=1}^8 b_i \right)$$

This expression for V_9 may be solved, as all terms on the right hand side are in known enthalpies and derivatives. By back substitution, all other liquid and vapor flows may be calculated.

The change of liquid and vapor flow rates is accompanied by a change in tray liquid holdups, and the holdup correlations (C.4) were used to calculate these new values. Rates of change of holdup were then calculated by Equation (C.9)

$$DWT_n^* = (WT_n^* - WT_n) / DT \quad (C.9)$$

where the superscript * denotes the updated holdup and derivatives. Before completing the cycle by taking the updated coefficients to the integration step, final corrected flow rates were calculated via the substitution method just described. It was found that one such iteration was sufficient to accurately correct flows for changes in holdup, and vice versa. These updated values of holdup, rate of holdup change, and liquid and vapor flow were used in further calculations in the next time step.

With new holdups, flow rates and derivatives, the coefficients in the composition balance equations had been determined. These equations were then integrated via the modified Euler or other integration scheme.

In summary, with the substitution method, the energy balances were presolved together with the total balances for the general case, yielding expressions which were solved for liquid and vapor flows. Holdups were calculated from liquid and vapor flows, and the latter were again corrected for holdup changes. The new holdups, flow rates and derivatives were used in the integration step as coefficients, to complete the integration cycle.

C.2 THE MATRIX INVERSION METHOD

Unlike the substitution method, the systems of equations were formulated into a matrix format, and were solved by computer library routines, by matrix inversion and vector operations. Other differences are evident in the choice of the systems of equations that are solved together. In the matrix inversion method, the energy balances (C.3) and holdup correlations (C.4) were solved together to calculate vapor and liquid flows, and the component balances (C.1) and total balances were solved by integration. The tray holdup derivative term was eliminated from the energy balance by substituting in the total balance (C.2). Equations (C.10) and

(C.11) were the result. Arranging the equations as

$$WT_n - c_n = a_n L_n + b_n V_n \quad (C.10)$$

$$WT_n \frac{dh_n}{dt} + QLP_n = L_{n+1}(h_{n+1} - h_n) + V_n(h_n - H_n) + V_{n-1}(H_{n-1} - h_n) \quad (C.11)$$

allows a matrix equation to be formulated. The equations to be solved (analogous to C.11) for the special stages were as follows:

feed tray

$$WT_5 \frac{dh_5}{dt} + QLP_5 = L_6(h_6 - h_5) + V_5(h_5 - H_5) + V_{n-1}(H_{n-1} - h_n) + F(h_F - h_n)$$

reboiler

$$WT_1 \frac{dh_1}{dt} + QLP_1 - QP = L_2(h_2 - h_1) + V_1(h_1 - H_1)$$

No enthalpy balance was required for the condenser.

The matrix equation formed was

$$\begin{bmatrix} \underline{f}_1 \\ \underline{f}_2 \end{bmatrix} = \begin{bmatrix} \underline{A}_1 \\ \underline{A}_2 \end{bmatrix} \begin{bmatrix} \underline{L} \\ \underline{V} \end{bmatrix} \quad (C.12)$$

As can be seen, terms in liquid and vapor flows were gathered on the right hand side, others on the left hand side.

To solve for the liquid and vapor rates, the matrix "A" is inverted.

$$\begin{bmatrix} \underline{L} \\ \underline{V} \end{bmatrix} = \begin{bmatrix} \underline{A}_1 \\ \underline{A}_2 \end{bmatrix}^{-1} \begin{bmatrix} \underline{f}_1 \\ \underline{f}_2 \end{bmatrix} \quad (\text{C.13})$$

Both the "A" matrix inversion and the transformation of the "F" vector by the inverted A matrix were accomplished by computer library subroutines (IBM routine SIMQ). The result of these operations is the flow vector, which consists of the liquid and vapor flows for each stage.

The second step in the calculation cycle is the integration step. The composition balances were worked into the matrix form (C.14)

$$\dot{\underline{x}} = \underline{B} \underline{x} \quad (\text{C.14})$$

To obtain this form, the following expression was substituted for the vapor compositions.

$$y_n = K_n x_n \quad (\text{C.15})$$

The ratio K_n was calculated for each stage just prior to the integration step, solely for the purpose of converting to the matrix form (C.14). As in the energy balances, the total balances were used to substitute flow rates for the holdup derivative term. The resulting set of equations have the general form (C.16)

$$a_n = \frac{1}{WT_n} (V_n(1-K_n) - L_{n+1})$$

$$n = 1$$

$$a_n = \frac{1}{WT_n} V_n(1-K_n)$$

$$n = 10$$

$$b_n = \frac{1}{WT_n} (V_{n-1}K_{n-1})$$

$$n=2,10 \quad (C.19)$$

$$c_n = \frac{1}{WT_n} (L_{n+1})$$

$$n=1,9 \quad (C.20)$$

Upon inspection, this matrix can be seen to be diagonally dominant, as K_n , the ratio of vapor composition to liquid composition on any tray, is always larger than 1, and thus all a_n terms are negative, and relatively large.

To integrate Equation (C.14), the following sequence was used. An approximation to the derivative at time step $K+1$ may be written

$$\underline{x}^{K+1} = \underline{x}^K + DT \dot{\underline{x}}^K$$

$$\text{or } \dot{\underline{x}}^K = (\underline{x}^{K+1} - \underline{x}^K) / DT \quad (C.21)$$

where DT is the width of the time step. Substituting Equation (C.14)

$$\underline{B}^K \underline{x}^K = (\underline{x}^{K+1} - \underline{x}^K) / DT$$

solving for $\underline{x}^{K+1}$

$$\underline{x}^{K+1} = (\underline{I} + DT \underline{B}^K)^{-1} \underline{x}^K \quad (C.22)$$

A better approximation for the derivative vector during the time step may be expressed as an average of the derivative at the beginning and end of the time step.

$$\dot{\underline{x}}_{av} = \frac{1}{2} \dot{\underline{x}}^{K+1} + \frac{1}{2} \dot{\underline{x}}^K \quad (C.23)$$

substituting Equations (C.14) and (C.21)

$$(\underline{x}^{K+1} - \underline{x}^K)/DT = \frac{1}{2} \underline{B}^{K+1} \underline{x}^{K+1} + \frac{1}{2} \underline{B}^K \underline{x}^K$$

solving for $\underline{x}^{K+1}$

$$\underline{x}^{K+1} = [\underline{I} - DT/2 \underline{B}^{K+1}]^{-1} [\underline{I} + DT/2 \underline{B}^K] \underline{x}^K$$

In this expression at time K, the matrix B at time K+1 is unknown. If one assumes that the matrix does not change quickly during the time step (i.e. if the system is not very non-linear), it may be taken to be constant. This results in Equation (C.24), from which $\underline{x}^{K+1}$ can be calculated

$$\underline{x}^{K+1} = [\underline{I} - DT/2 \underline{B}^K]^{-1} [\underline{I} + DT/2 \underline{B}^K] \underline{x}^K \quad (C.24)$$

Again, as in the coefficient determination step, library computer routines were used to do all inversions and matrix-vector operations.

A simple Euler method was used to integrate the total mass balances, to obtain holdups. This set (3.30) of equations was not able to be formulated into a matrix form similar to Equation (C.14), necessitating the use of the Euler or other such method.

C.3 HOLDUP CORRELATIONS

a) Rademaker et al.²⁸

According to Rademaker at least two ways of expressing a holdup relationship can be found

$$M = M \{L_1 V / \sqrt{\rho_v}\}$$

or

$$L = L \{M_1 V / \sqrt{\rho_v}\}$$

Rademaker presents parameters for these formulations.

In this work, the latter formulation was chosen, and a conversion was necessary. According to Rademaker:

$$\Delta L = \left(\frac{\partial L}{\partial M}\right)_{V, \rho_v} \Delta M + \left(\frac{\partial L}{\partial V}\right)_{M, \rho_v} \left[\Delta V - \frac{\bar{V}}{2\rho_v} (\Delta \rho_v)\right]$$

or for the i th stage in perturbation quantities,

$$L_i = \frac{M_i}{\tau_1} + \lambda_1 (V_{i-1} - \lambda_2 \rho_{v_{i-1}})$$

as constant pressure was assumed, the pressure correction was dropped. Rearranging,

$$\begin{aligned} \Delta M_i &= \left(\frac{\partial M}{\partial L}\right)_V \Delta L_i - \left(\frac{\partial M}{\partial V}\right)_L \Delta V_{i-1} \\ &= \left(\frac{\partial M}{\partial L}\right)_V \Delta L_i - \left(\frac{\partial M}{\partial L}\right)_V \left(\frac{\partial L}{\partial V}\right)_M \Delta V_{i-1} \end{aligned}$$

In the Rademaker formulation,

$$\tau_1 = \left(\frac{\delta M}{\delta L} \right)_V = 2.2 \text{ sec}$$

$$\lambda_1 = \left(\frac{\delta L}{\delta V} \right)_M = 0.7$$

substituting these values

$$\Delta M_i = 2.2 \Delta L_i - 1.54 \Delta V_{i-1}$$

Since this is the perturbed equation, modifications must be applied for absolute values:

$$M = \bar{M} + \Delta M, \quad L = \bar{L} + \Delta L, \quad V = \bar{V} + \Delta V$$

$$M - \bar{M} = 2.2 (L - \bar{L}) + 1.54 (V - \bar{V})$$

$$M = (\bar{M} - 2.2\bar{L} + 1.54\bar{V}) + 2.2L - 1.54V$$

$$= a + bL + cV$$

b) AICHE Bubble Tray Design Manual²⁹

To predict holdups for use in the AICHE efficiency prediction procedure, the following equation has been determined empirically:

$$z_c = 1.65 + 0.19w - 0.65F + 0.020L$$

where

w = weir height (in)

L = liquid flow (gpm/ft of flow width)

$F = V_G \rho_G^{0.5}$

V_G = vapor rate (ft/sec)

ρ_G = gas density (lbm/ft³)

z_c = height of clear liquid on the tray (in)

c) Francis Weir Formula

The expression given by Luyben³⁶ is

$$F_L = 3.33 \ell (h)^{3/2}$$

where F_L = liquid rate (ft³/sec)

ℓ = length of weir (ft)

h = height of liquid over weir (ft)

while Perry³⁵ gives the following expression

$$q = 0.415(L - 0.2 h_o) h_o^{1.5} \sqrt{2g}$$

where L = crest length (ft)

h_o = head above weir (ft)

q = flow over weir (ft³/sec)

g = force of acceleration due to gravity (ft/sec²)

If it is assumed that $0.2h_o \ll L$ i.e. small head on weir, then the expression simplifies to

$$q = 0.415L h_o^{1.5} \sqrt{2g}$$

and introducing that $g = 32.2$ ft/sec², reduces the equation to the one cited by Luyben. Expressed in SI units, the equation becomes

$$q(\text{cm}^3/\text{sec}) = .415L(\text{cm}) h_o^{1.5}(\text{cm}) (2g(\text{cm}/\text{sec}^2))^{0.5}$$

Holdup values calculated at the standard operating conditions are given in Table C.1 for the Francis Weir and AIChE correlations.

TABLE C.1

CALCULATED TRAY LIQUID HOLDUPS

Tray	Francis Weir Holdup (kg)	AIChE Holdup (kg)
1	2.18	1.90
2	2.13	1.85
3	2.07	1.77
4	1.99	1.73
5	1.92	1.65
6	1.82	1.53
7	1.82	1.51
8	1.79	1.46

These holdups were calculated for the base operating state as follows:

feed flow	18 g/s
steam flow	14 g/s
reflux flow	14 g/s

Simulations using these operating conditions gave liquid and vapor flows for each tray, which were then used along with tray dimensions to calculate the entries in Table C.1.

It may be noted that holdups predicted by correlations are much larger in magnitude than those measured. The AIChE correlation was developed for use with industrial scale columns so the pilot plant tower may not fit perfectly in the range in which the correlation applies. However, an indication of holdup dependence upon column parameters was obtained, showing weak dependence upon vapor flow in one case and a weak dependence upon liquid flow in the other.

C.4 EXPERIMENTAL PARAMETER SAMPLE CALCULATIONS

C.4.1 ²⁹ AIChE Holdup Correlation

The relationship for liquid tray holdup given by the AIChE Bubble Tray Design manual is:

$$z_c = 1.65 + .019w - .65F + .020L$$

where $F = V \rho_G^{0.5}$

z_c = height of liquid on the tray (in)

V = vapor flow (ft/sec)

ρ_G = vapor density (lbm/ft³)

L = liquid flow (gpm/ft of flow width)

w = weir height

assumption - feet of flow width = 8 in $\approx$.7 ft

example stage 3 (tray 2)

$x = .335$ (liquid composition)

$y = .680$ (vapor composition)

$$v(\text{ft}^3/\text{lbm}) = \frac{zRT}{p}, \quad z \approx 1$$

for MeOH

$$v = \frac{(354.2^\circ\text{K})}{(1 \text{ atm})} \left(\frac{.0821 \text{ atm l}}{\text{gmole } ^\circ\text{K}} \right) \left(\frac{\text{ft}^3}{28.32 \text{ l}} \right) \left(\frac{454 \text{ gmole}}{\text{lbmole}} \right) \left(\frac{\text{lbmole}}{32.04 \text{ lbm}} \right)$$

$$v = 14.55 \text{ ft}^3/\text{lbm}$$

similarly for water

$$v = 26.83 \text{ ft}^3/\text{lbm}$$

at $y = .680$

$$v = .680(14.55) + .32(26.83)$$

$$= 18.48 \text{ ft}^3/\text{lbm}$$

gas volumetric flow is:

$$V = 15.1 \text{ g/sec} \left(\frac{\text{lbm}}{454 \text{ gm}} \right) \left(\frac{18.48 \text{ ft}^3}{\text{lbm}} \right)$$

$$= .615 \text{ ft}^3/\text{sec}$$

area for gas flow:

$$A = \frac{\pi D^2}{4} = \frac{\pi 9^2}{4} = 63.6 \text{ in}^2$$

$$= .442 \text{ ft}^2$$

$$\therefore v = \frac{.615 \text{ ft}^3/\text{sec}}{.442 \text{ ft}^2}$$

$$= 1.39 \text{ ft/sec}$$

gas density:

$$\rho = \frac{1}{v}$$

$$= \frac{1}{18.48} \text{ lbm/ft}^3 = .0541 \text{ lbm/ft}^3$$

$$\rho = (.0541)$$

vapor term in correlation:

$$F = v\rho^{0.5} = (1.39)(.0541)^{0.5} = .324$$

The liquid flow term:

$$L = 23.22 \text{ g/sec} \left(\frac{\text{cm}^3}{.93 \text{ gms}} \right) \left(\frac{1 \text{ ft}}{30.5 \text{ cm}} \right) \left(\frac{3600 \text{ sec}}{\text{hr}} \right) \left(\frac{.1247 \text{ gpm}}{\text{ft}^3/\text{hr}} \right)$$

$$= .3938/.7 \text{ ft}$$

$$= .5625 \text{ gpm/ft of flow width}$$

$$z_c = 1.65 + .19w - .65F + .020L$$

$$= 1.65 + .19(2) - .65(.324) + 0.20(.5625)$$

$$= 1.83 \text{ cu in/in}^2$$

but the column has 64 in² of area

$$w = z_c (64)$$

$$= 116.4 \text{ in}^3 \text{ or } 1910 \text{ cm}^3$$

liquid density at $x = .30$ is $.93 \text{ gm/cm}^3$

Total weight of tray holdup = 1774 gms

C.4.2 Francis Weir Correlation³⁵

The Francis Weir correlation in SI units is:

$$q(\text{cm}^3/\text{sec}) = .415 L(\text{cm}) h_o^{1.5}(\text{cm}) \sqrt{2g} (\text{cm}/\text{sec}^2)$$

assume the weir is 3 sections of 2.5 cm widths

example stage 3 (tray 2)

liquid flow - 23.2 g/sec or 7.73 g/sec through one section of the weir.

liquid density at $x = .335$:

$$\rho_L = .93 \text{ g/cm}^3$$

volumetric liquid flow:

$$q = (7.73 \text{ g/sec}) / (.93 \text{ g/cm}^3)$$

$$= 8.3 \text{ cm}^3/\text{sec}$$

substituting

$$8.3 = .415 (2.5) h_o^{1.5} \sqrt{981}$$

$$h_o^{1.5} = .255$$

$$h_o = .402$$

$$\text{Total head on tray} = 5 \text{ cm} + .402 \text{ cm}$$

$$= 5.402 \text{ cm}$$

total tray area 64 in^2 or 413 cm^2

total holdup = $5.4 \text{ cm} (413 \text{ cm}) (.93 \text{ gm/cm}^3)$

= 2074 gms

C.4.3 Foss and Geister Holdup Correlation³³

The Foss and Geister correlation is:

$$h_L = 0.24 + 0.725 h_w - .29 h_w V_a^{0.5} \rho_G + 4.48 \frac{q}{z}$$

where h_w = outlet weir height (in)

V_a = gas velocity (ft/sec)

ρ_G = gas density (lbm/ft^3)

q = liquid flow rate (cu ft/sec)

z = average liquid flow width (ft)

h_L = liquid head on tray (in)

assume average liquid flow width = 8 in or = .7 ft.

example stage 3 (tray 2)

$V = 15.1 \text{ g/s}$

$L = 23.2 \text{ g/sec}$

$x = .335$

$y = .680$

from the sample calculation of the AIChE holdup prediction:

$$V_a \rho_G^{0.5} = .324$$

volumetric liquid flow:

$$q = 23.2 \text{ g/sec} \left(\frac{1 \text{ cm}^3}{.93 \text{ gms}} \right) \left(\frac{1 \text{ ft}}{30.5 \text{ cm}} \right)$$

$$= 8.79 \times 10^{-4} \text{ ft}^3/\text{sec}$$

$$h_L = 0.24 + 0.725(2) - .29(2)(.324) + 4.48(.000879)/(.7)$$

$$= 1.507 \text{ in or } 3.83 \text{ cm}$$

total mass holdup:

$$3.83 \text{ cm } (413) \text{ cm}^2 (.93 \text{ gm/cm}^3)$$

$$= 1470 \text{ gms}$$

C.4.4 ²⁹ AICHE Tray Efficiency Calculation

The correlation to determine efficiency given by the AICHE Bubble Tray Design manual is:

$$N_G = \frac{.776 + .116 z_w - .290 V_G \rho_G^{0.5} + 0.0217 L_w}{N_{sc}^{0.5}}$$

where

$$N_{sc} = \frac{\mu_G}{\rho_G D_G} \text{ (dimensionless)}$$

z_w = height of outlet weir (in)

μ_G = gas viscosity

ρ_G = gas density (lbm/ft³)

L_w = liquid rate (gallon/min)/ft of average liquid flow width

D_G = diffusivity of the gas

assume average liquid flow width ≈ 8 in or .7 ft

Limitations

This correlation was developed from empirical data in the following ranges:

$$N_{sc} \approx .61$$

$$V_G \rho_G^{0.5} \approx 1.0 - 2.3$$

$$L \approx 5 - 25 \text{ gpm/ft of flow width}$$

$$z_w \approx 1 - 5 \text{ in}$$

example stage 3 tray 2

$$L = 23.2 \text{ g/s}$$

$$V = 15.1 \text{ g/s}$$

$$x = .335$$

$$y = .680$$

Calculate the Schmidt number:

Estimate the diffusivity using correlation from Perry:³⁵

$$D = \frac{BT^{3/2} \sqrt{\frac{1}{M_1} + \frac{1}{M_2}}}{p r_{12}^2 I_D}$$

Going through the steps of estimating atomic radii etc.; a diffusivity was calculated.

$$D \approx .0417 \text{ in}^2/\text{sec} \approx .269 \text{ cm}^2/\text{sec}$$

vapor density:

using ideal gas law,

$$V_{\text{MeOH}} = 14.55 \text{ ft}^3/\text{lbm}$$

from steam tables,

$$V_{\text{water}} = 26.83 \text{ ft}^3/\text{lbm}$$

at $Y = .680$, $V = 18.48 \text{ ft}^3/\text{lbm}$
 $= 1150 \text{ cm}^3/\text{gm}$

vapor viscosities:

from Perry, viscosities are

water - $.0114 \text{ cp}$ ($\text{gm}/\text{cm sec} \times 10^{-2}$).

MeOH - $.0116 \text{ cp}$

∴ assume an average of $.0115 \text{ cp}$ over all compositions.

$$N_{sc} = \frac{\mu_G}{\rho_G D_G} = \frac{.000115 \text{ gm}/\text{cm sec} \cdot 1150 \text{ cm}^3/\text{gm}}{.269 \text{ cm}^2/\text{sec}}$$

$$= .487$$

$$N_{sc}^{0.5} = .70$$

Liquid flow rate:

$$L_w = 23.2 \text{ g/s} \left(\frac{1 \text{ cm}^3}{.93 \text{ gm}} \right) \left(\frac{\text{ft}}{30.5 \text{ cm}} \right)^3 \left(\frac{3600 \text{ sec}}{\text{hr}} \right) \left(\frac{.1247 \text{ gpm}}{\text{ft}^3/\text{hr}} \right)$$

$$= .395 \text{ gpm}$$

or $.564 \text{ gpm}/\text{ft}$ of flow width

Vapor flow rate:

from AIChE holdup prediction calculation,

$$V_G \rho_G^{0.5} = .324$$

To calculate vapor transfer units:

$$N_G = \frac{.776 + .116 z_w - 0.290 V_G \rho_G^{0.5} + 0.0217 L_w}{N_{sc}^{0.5}}$$

$$= (.776 + .116(2) - 0.290(.324) + .0217(.564)) / .70$$

$$= 1.323$$

Calculate number of liquid transfer units:

$$N_L = [(1.065 \times 10^4) D_L]^{0.5} (.26 V_G \rho_G^{0.5} + 1.5) t_L$$

where D_L = liquid diffusivity (ft²/hr)

$$t_L = \frac{37.4 z_C z_L}{L_w}$$

z_C = holdup, inches of clear liquid on tray

z_L = liquid flow distance (ft)

From AIChE holdup sample calculation,

$$z_C = 1.83 \text{ in}^3/\text{in}^2$$

$$L_w = .563 \text{ gpm/ft of flow width}$$

$$V_G \rho_G^{0.5} = .324$$

$$z_L \approx 8 \text{ in or } .7 \text{ ft}$$

$$t_L = \frac{37.4(1.83)(.7)}{.563}$$

$$= 85.1 \text{ sec}$$

Liquid phase diffusivity $D_L = 1.6 \times 10^{-5} \text{ cm}^2/\text{sec}$ (Perry)

$$= 6.2 \times 10^{-5} \text{ ft}^2/\text{hr}$$

Calculating the liquid transfer unit,

$$N_L = [(1.065 \times 10^4) (6.2 \times 10^{-5})]^{0.5} (.26(.324) + 1.5) 85.1$$

$$= 109.5$$

Combine the liquid and vapor phase

$$\frac{1}{N_{OG}} = \frac{1}{N_G} + \frac{\lambda}{N_L}$$

where $\lambda = \frac{mG}{L}$

m = slope of equilibrium line

G = vapor rate lbmoles/ft²hr

L = liquid rate lbmoles/ft²hr

From equilibrium data, at stage 3 composition

$$m = .7064$$

$$L = 23.2 \text{ g/sec}$$

$$V = 15.1 \text{ g/sec}$$

Assume approximately similar molecular weights in both

liquid and vapor phase,

$$\text{then, } \lambda = \frac{.7064(15.1)}{23.2}$$

$$= .454$$

$$\frac{1}{N_{OG}} = \frac{1}{N_G} + \frac{\lambda}{N_L}$$

$$= \frac{1}{1.323} + \frac{.454}{109.5}$$

$$N_{OG} = 1.315$$

$$E_{OG} = 1 - e^{-N_{OG}}$$

$$= 1 - e^{-1.315}$$

$$= .732$$

To convert to Murphree tray efficiency:

calculate eddy diffusivity

$$D_E = (0.0124 + .0171 V_G + .00250L + .0150w)^2$$

where V_G = vapor velocity (ft/sec)

L = liquid flow (gpm/ft of flow width)

w = weir height (in)

substituting in precalculated values,

$$\begin{aligned} D_e &= (0.0124 + 0.171(1.39) + .00250(.564) + .0150(2))^2 \\ &= .00456 \end{aligned}$$

Calculate the Peclet number

$$\begin{aligned} P_e &= \frac{z_L^2}{D_E t_L} \\ &= \frac{.7^2}{(.00456)(85.1)} \\ &= 1.26 \end{aligned}$$

From Figure 8a, AIChE Bubble Tray Design Manual, a ratio is obtained from the Peclet number:

$$\frac{E_{MV}}{E_{OG}} = 1.02$$

for $P_e = 1.26$

$$\lambda E_{OG} = (.454)(.732)$$

$$= .332$$

$$\begin{aligned} \therefore E_{MV} &= (1.02)(.732) \\ &= .746 \end{aligned}$$

or Murphree vapor efficiency = .746 or 74.6%

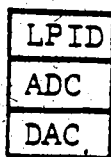
APPENDIX D

THE PILOT PLANT DISTILLATION COLUMN

The following is a detailed schematic of the pilot plant distillation column, with all pertinent control and data accumulation loops marked.

Detailed information about all data accumulation or control loops on the column can be found in the DDC loop records in this Appendix. DDC loops not marked on Figure D.1, but included in the DDC loop list are history data accumulation loops.

Engineering drawings and dimensions of the pilot plant column and bubble cap trays are obtainable from the thesis of Svrcek²⁶.

LEGEND

- indicates measurements available to IBM 1800
- LPID - DDC loop identification code in hexadecimal
 - crosshatching indicates no loop
- ADC - multiplexer input address (in hexadecimal)
- DAC - multiplexer output address (in hexadecimal)
 - crosshatching indicated no output
- loop is for data acquisition only



- indicates local analog equipment
- TYPE - gives function

TYPE CODES

- T - transmitter
- R - recorder
- C - controller
- I - indicator
- F - flow
- A - concentration (analyzer)
- T - temperature
- P - pressure
- DP - differential pressure

n - identification number



- heater exchanger
- n - identification number



- gas chromatograph



- thermocouple



indicates utilities

- UT - ST - 60 psig steam
- CW - cooling water



- solenoid valve
- n - identification number

--- - control lines

— - process lines

LOOP RECORD 0601

0601	E21B	005C	1006	3800	9E40	1FAB	0000	0000	3286
7FFF	7FFF	200C	7FFF	0000	2000	7FFF	0000	2000	106A
7FFF	0080	0000	6F17	E5C0	00CD	FFC1			

LOOP RECORD 0602

0602	E21B	005C	1005	1800	9E40	1719	FE28	0000	4467
7FFF	7FFF	000C	7FFF	0000	0029	7FFF	0000	2110	116A
A358	0380	0000	A35C	C000	0400	FFF0			

LOOP RECORD 0603

0603	E21B	005C	1004	3800	9E40	1050	FDF3	0000	4515
7FFF	7FFF	200C	7FFF	0FA4	2000	7FFF	0000	2000	126A
A391	0080	0000	A384	70C0	0066	FFDB			

LOOP RECORD 0604

0604	E21B	204F	100A	0000	9100	0850	2422	E021	22E3
7FFF	7FFF	255C	35E8	0000	0000	7FFF	0000	0000	0602
EC4F	2020	0000	E28F	6700	001F	FFDB			

LOOP RECORD 0605

0605	E21B	005E	1007	3340	9E40	0005	FF67	0000	3C7F
7FFF	7FFF	200C	7FFF	0000	2000	7FFF	0000	0000	0000
0000	0000	0000	0000	0000	0000	0000			

LOOP RECORD 0606

0606	E010	205F	1008	1000	9EE0	0C39	FFB0	0000	6690
7FFF	7FFF	211C	7FFF	0000	A11C				

LOOP RECORD 0607

0607	A010	407F	0608	3A00	9180	4E20	0000	0104	01EB
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0608

0608	8010	C00F	0000	0000	9180	4E20	0000	0104	01EB
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0609

0609	A010	707F	0629	3000	91A0	4E20	0000	FEBA	4666
7FFF	0000	8000	0000	0000	800C				

LOOP RECORD 0610

0610	F010	218F	E091	3800	A0C0	2B68	FF4E	0990	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0611

0611	F010	21BF	E083	3800	A0C0	2868	FF4E	09C7	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0612

0612	F010	21BF	E084	3800	A0C0	2868	FF4E	09C4	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0613

0613	F010	21BF	E085	3800	A0C0	2868	FF4E	2C7B	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0614

0614	F010	21BF	E086	3800	A0C0	2868	FF4E	09DD	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0615

0615	F010	21BF	E087	3800	A0C0	2868	FF4E	09FA	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0616

0616	F010	21BF	E088	3800	A0C0	2868	FF4E	09DA	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0617

0617	F010	21BF	E089	3800	A0C0	2868	FF4E	09B4	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0618

0618	F010	21BF	E08A	3800	A0C0	2868	FF4E	098F	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0619

0619	F010	21BF	E08B	3800	A0C0	2868	FF4E	0906	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0620

0620	F010	21BF	E093	3800	A0C0	2868	FF4E	098F	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0621

0621	F010	21BF	E092	3800	A0C0	2868	FF4E	09AB	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0622

0622	F010	21BF	E095	3800	A0C0	2868	0000	098D	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0623

0623	F010	218F	E096	3800	A0C0	2868	FF4E	10987	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0624

0624	E010	200F	2097	0000	A100	F2F0	02DD	000A	0000
0000	7FFF	3000	7FFF	0000	3000				

LOOP RECORD 0625

0625	E010	200F	100E	0000	8440	03E8	FF9C	FF8E	0000
7FFF	7FFF	3000	7FFF	0000	3000				

LOOP RECORD 0626

0626	E010	200F	100C	0000	9940	BF28	1F40	0016	0000
7FFF	7FFF	3000	7FFF	0000	3000				

LOOP RECORD 0627

0627	E010	205F	1009	3340	AEE0	3520	FA56	0000	0000
7FFF	7FFF	211C	7FFF	0000	A11C				

LOOP RECORD 0628

0628	F010	218F	E082	3800	A0C0	2868	FF76	0931	0000
7FFF	7FFF	2110	7FFF	0000	2110				

LOOP RECORD 0629

0629	8010	800F	0000	0000	91A0	4E20	0000	FEBA	0000
0000	0000	8000	0000	0000	800C				

LOOP RECORD 0630

0630	0620	5000	0601	0008	0335	FF0D	38CA	38DF	3806
38E8	380A	38DB	3880	38C8	3889	3887	388F	38E9	380A
38EA	38CC	38D7	38D6	38D0	38E9	38E2	38D4	38CD	38CC
38C4	38B9								

LOOP RECORD 0631

0631	0620	6000	0602	101E	03FF	E115	2EEE	2E86	36BD
2F0E	3FE3	2F62	3FFE	2F4D	3FF3	2F52	3FAE	406C	40E9
40EC	40B5	2F97	3FD1	2F3A	2F65	406C	301D	4002	2F4D
39F8	3A29								

LOOP RECORD 0632

0632	0620	5000	0603	0010	0335	E10D	4480	44FC	44DE
457C	4581	4496	4409	4508	4598	4557	4493	449D	450D
458B	4587	456C	44F4	4485	4480	44AB	456B	4595	457C
44E4	44CD								

LOOP RECORD 0633

0633	0620	6000	0604	0017	060A	E134	E6F2	E70D	E708
E6FC	E703	E6E8	E724	E706	E706	E701	E708	F1CE	FC14
0599	0CDD	10E3	E726	E6FC	E6F7	E724	E701	E6F2	E701
E6F7	E708								

LOOP RECORD 0634

0634	0620	8000	0608	0019	0608	FF2C	0104	0104	0104
0104	0104	0104	0104	0104	0104	0104	0104	0104	0104
0104	0104	0104	0104	0104	0104	0104	0104	0104	0104
0104	0104								

LOOP RECORD 0635

0635	0620	8000	0607	0014	0334	FF29	0159	0199	01A8
017E	0174	0178	0196	01E9	01EB	01F5	0241	0258	022E
0104	011F	011F	0110	010E	0114	0126	0130	0166	0171
0159	0141								

LOOP RECORD 0636

0636	0620	5000	0628	000E	0335	E10D	1A9B	1AA3	1AA5
1AA3	1AA7	1AAC	1A83	1A86	1A87	1A88	1A89	1A89	1A86
1A87	1A89	1A89	1A8E	1A93	1A96	1A96	1A93	1A91	1A9E
1A9B	1A9A								

LOOP RECORD 0637

0637	0620	6003	0602	1014	0334	E129	3927	3927	3927
3927	3927	3927	3927	3927	3927	3927	3927	3927	3927
3927	3927	3927	3927	3927	3927	3927	3927	3927	3927
3927	3927								

LOOP RECORD 0638

0638	0620	5007	0603	1016	0335	E10D	4515	4515	4515
4515	4515	4515	4515	4515	4515	4515	4515	4515	4515
4515	4515	4515	4515	4515	4515	4515	4515	4515	4515
4515	4515								

LOOP RECORD 0639

0639	0620	8000	0608	001C	0334	E129	011E	011C	0138
014D	017D	0178	0152	013D	0169	0183	0178	0173	0182
017A	01F2	021E	01C1	0239	0272	0263	0206	0131	0129
0124	0102								

APPENDIX E

THE GAS CHROMATOGRAPH ANALYSIS SYSTEM

In this work, the bottoms composition analysis apparatus was changed from a Beckman series 4 industrial chromatograph, in which a common oven volume was shared by the separation column and automatic slider sampling valve, to a Hewlett Packard gas chromatograph (G.C.) Model No. 5225A, with a separate oven for the sampling valve. Several problems were encountered during this transition. The approach to solving these difficulties is described herein.

A short description of the steps involved in an analysis is in order before discussing some of the problems encountered. A representative sample of the liquid to be analyzed must be collected and injected into the column. Since the bottoms product is a liquid, much smaller volumetric samples need be drawn from the process stream, in the order of 1 microlitre, which is about one tenth of the volume that would be used for a vapor sample. This creates two problems. It is more difficult to consistently provide a sample of the same size and furthermore a consistent analysis for small sample volumes becomes much more difficult. As a consequence of the small volume, the valve is prone to plugging by any particulate matter, and thorough filtering is necessary.

A liquid sample must be quickly flashed before being introduced into the separation column. This required the sampling valve to be operated at higher temperatures than vapor sampling valves, and resulted in dissolved solids being deposited in the sample volume, again plugging it up. All these problems were encountered at some time during this work and three different sampling valves were tried and tested before the problems were sufficiently resolved.

By far the most difficult of the above problems to overcome was that of high temperature and proper flashing. Schematic diagrams of the valves used are shown in Figures E.1 to E.4. With the arrival of the new gas chromatograph, the Valco valve shown in Figure E.2 was installed. Flow through the valve was poor, introducing a further time delay in the process measurement, which was undesirable. In addition, the oven in which the valve was operated initially had the Hewlett Packard insulation only, which was relatively poor, and slow flashing was the result. High temperature operation caused the valve internals to scorch, making actuation difficult. The internals were once changed, with little improvement. Other problems also surfaced but plugging, low flow and high temperature scorching were the most serious. A 10 micron filter installed in the sample line eliminated much of the plugging, but no amount of G.C. and valve oven temperature adjustment could avoid the scorching problem, and maintain

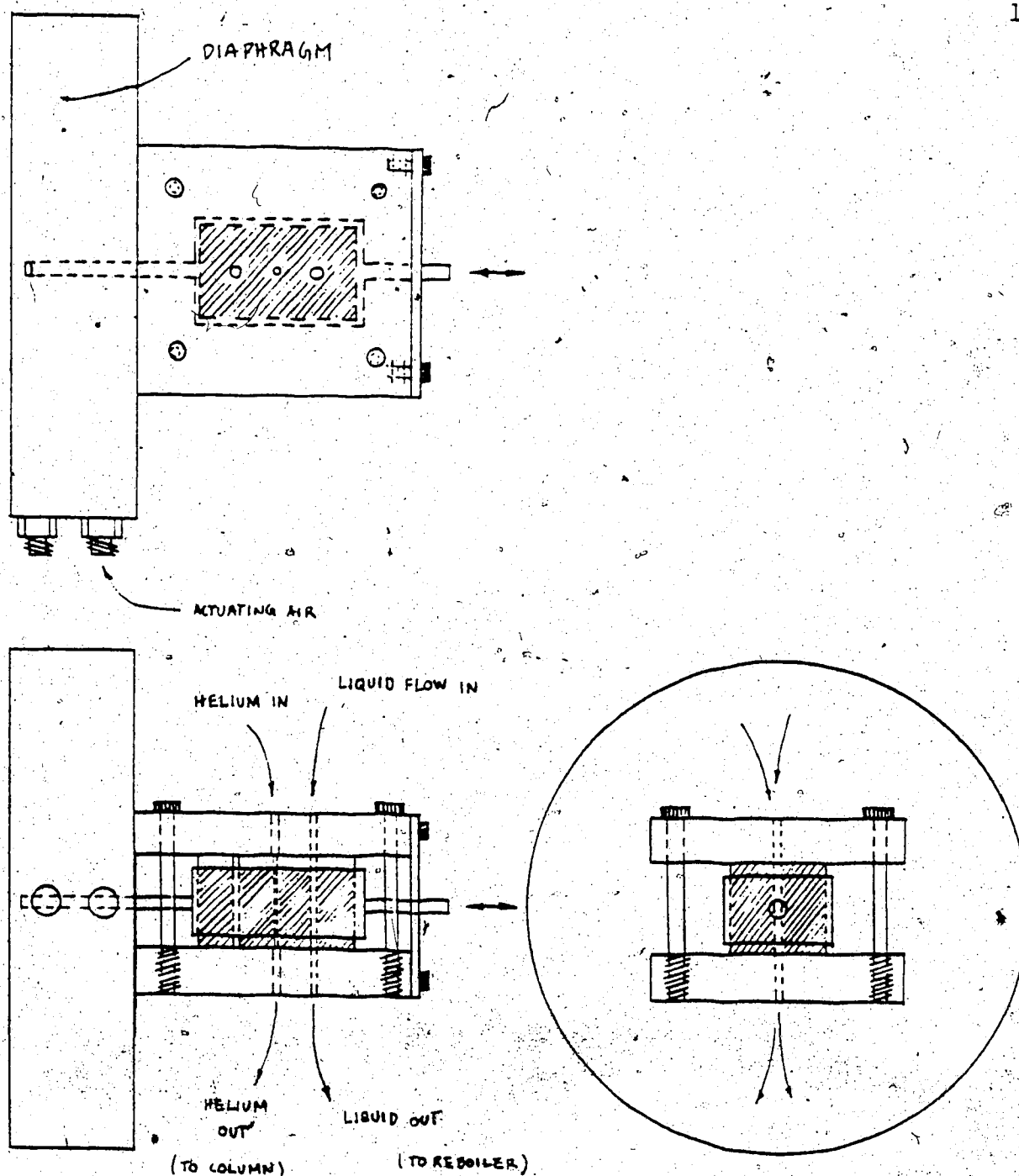


FIGURE E.1 Beckman Liquid Sampling Slider Valve

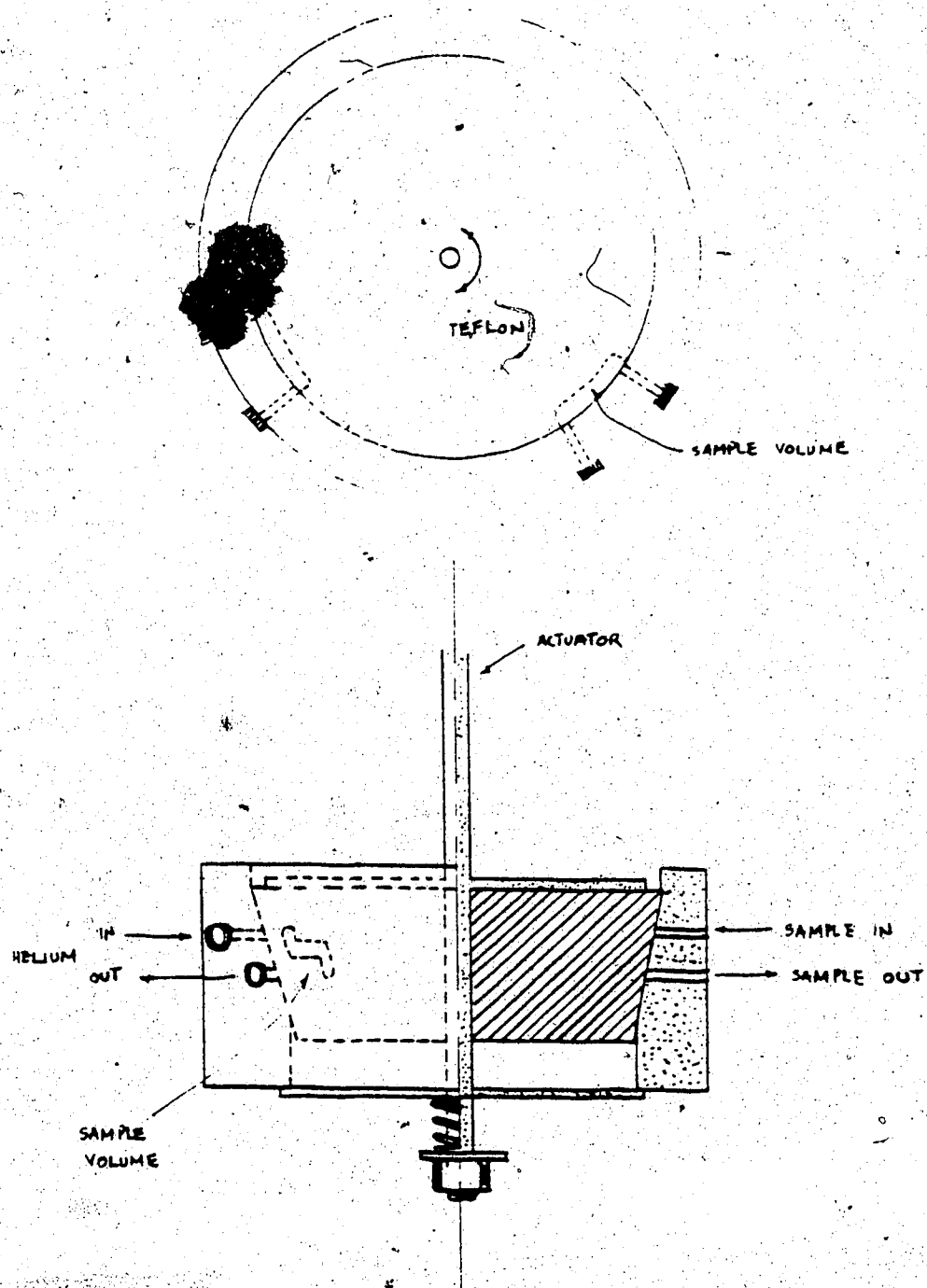


FIGURE E.2 Valco Liquid Sampling Valve

good analyses.

Another valve installed as shown in Figure E.3 and E.4, manufactured by Applied Automation and known as Mark IX, contains very few moving parts and has a high metal content. This resulted in good flashing and negligible mechanical failure. The only problem encountered was a deterioration of a teflon membrane with continued use. Adequate documentation was not supplied with the valve, so after a trial and error procedure, proper operating conditions were found. A high temperature membrane was required, as the valve was run at 150°C, a temperature that was a compromise between deterioration and quick flashing. Valve actuation pressure was an important variable, minimal pressure being optimum. The internal arrangement of the valve, containing springs to load the flow paths was found to be important. A spring arrangement resulting in minimal pressure on the diaphragm is necessary for diaphragm longevity. Other spring settings may be used for higher pressure, lower temperature operations.

The internal flow path is also critical to good flashing, and good separation. The valve allows for a choice between two possibilities of the flow path. The one which results in better flashing was established by trial and error. A scheme was found to pass the liquid sample from the process stream to the separation column as a vapor, with consistency, reliability and reproducibility necessary for continuous

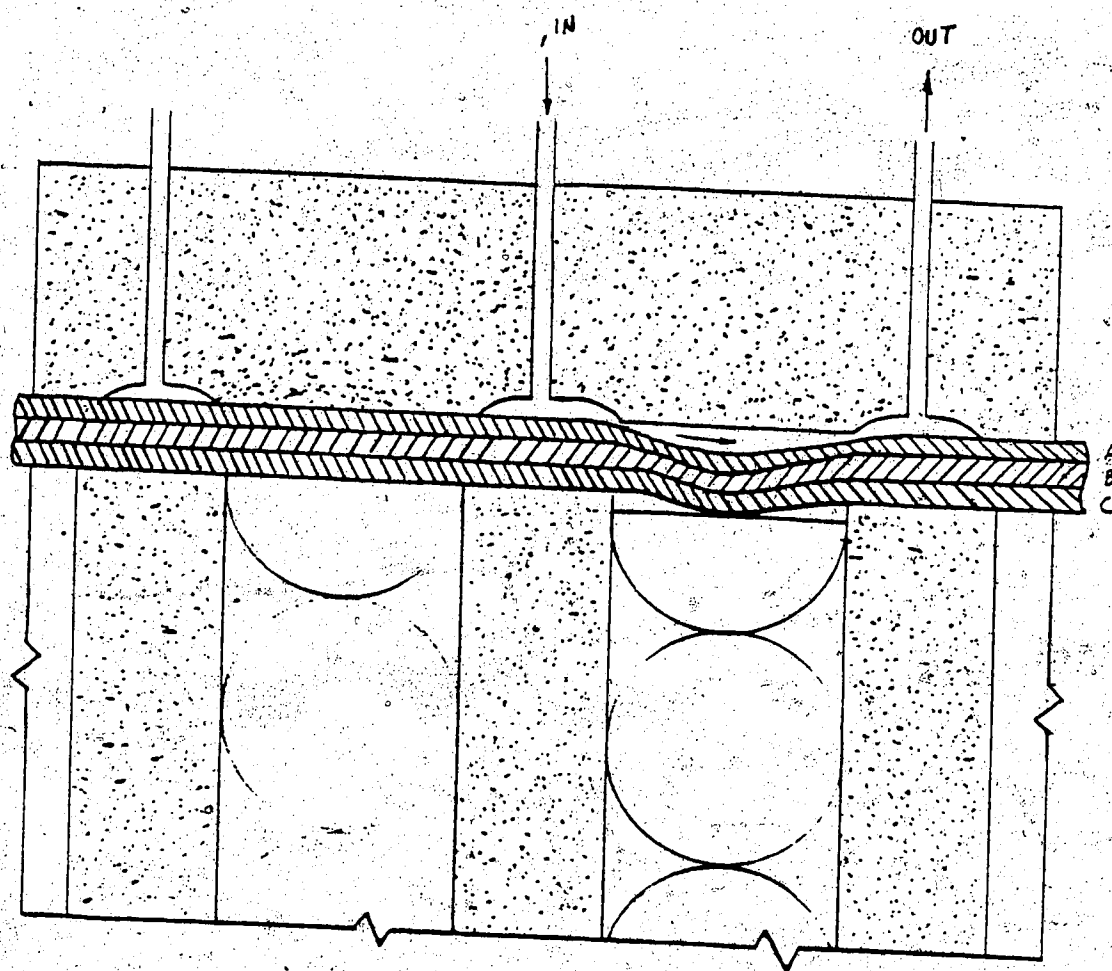


FIGURE E.3 Applied Automation Mark IX Liquid Sampling Valve - Cross Sectional View

VALVE CAP - FLOW PATHS

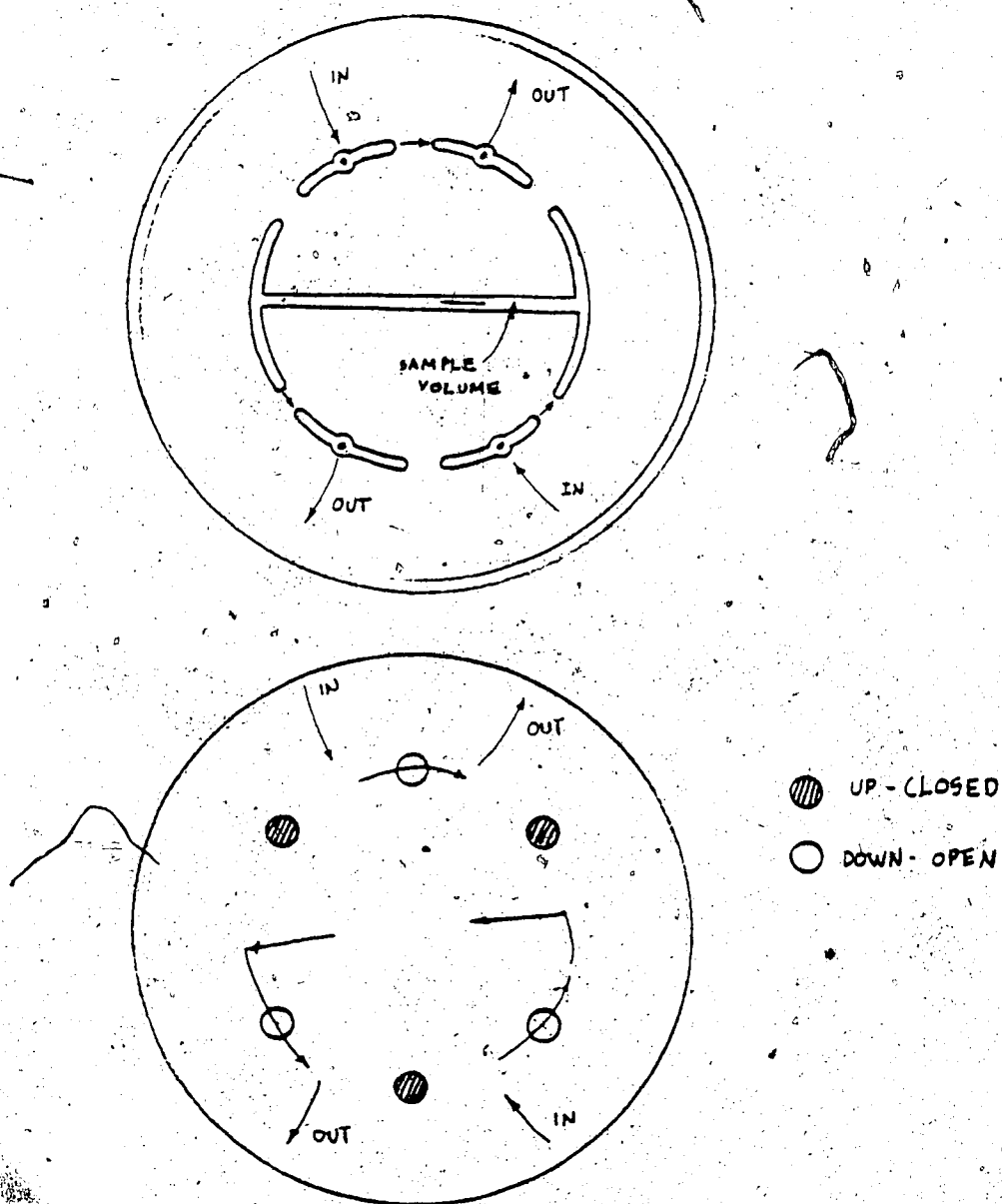


FIGURE E.4 Applied Automation Mark IX Liquid Sampling Valve - Top View

accurate analyses. It was then the task of the gas chromatograph to separate and analyse the sample, and the task of the computer to interpret the G.C. output and calculate a composition in weight percent methanol.

Problems encountered in the separation were typical of any gas chromatography analyses: the specification of operating conditions for optimum separation and minimum elution times. Poropak Q packing had been used to separate water and methanol, but at small concentrations, the methanol peak was always found high on a long tail from the water peak, and made analyses difficult. Experiments were done with Poropak QS, a new packing, but it was found to degrade very rapidly with time. Finally, the operating conditions listed in Table E.1 were established, based on a compromise between elution time and separation.

TABLE E.1

G.C. OPERATING CONDITIONS

Separation Oven Temperature	90°C
Valve Oven Temperature	150°C
Carrier Gas (Helium) Flow	0.9 ml/sec
Detector Temperature	200°C
Detector Current	150 mA
Sample Size	2 μ l

A sample size of 2 microlitres was chosen, since for small concentrations of methanol, use of 1 microlitre sample volume gave rise to a methanol peak so small that it was lost in the noise encountered in transmitting the G.C. output signal to the IBM 1800 while use of larger samples tended to flood the separation column and degrade the separation. It should be noted also that subsequently an amplifier was installed on the G.C. output, thus reducing the significance of noise.

The methanol content in the samples was determined by the G.C. analysis package on the IBM 1800. This program integrates the area under component peaks, and by comparing relative areas, relative quantities in the sample are determined. Operation of the program is by specification of a "GC job" for each particular type of separation or chromatogram. Peaks beginnings and ends are identified by observing first and second derivative changes in the G.C. output. Defining an appropriate G.C. job for typical chromatogram is accomplished with the help of a new GCRAW group of programs which collect and plot peak first and second derivative data.

The addition of the amplifier was the single most important modification that reduced the difficulty in obtaining consistent measurements, once the sample had been injected into the separation column. The significant changes in the signals have already been discussed in Chapter 5.

Degradation of analyses can be detected by a change in the elution times of the peaks. Once the G.C. has warmed up, elution times should not vary by ± 2 seconds. Flow through the sampling valve is a reliable indicator of valve failure. Inconsistent analyses may be traced to valve failure in most cases. The line filter must be cleaned periodically and care must be taken to keep the valve oven temperature as close to the set operating temperature as possible. Also, since the column is operated as a closed system, any solids accumulate in the methanol-water solution; the solution must be purged from time to time, or plugging of the valve by dissolved solids will become a problem.

APPENDIX F

EXPERIMENTAL PARAMETER PREDICTION

The parameters of holdup and efficiency are important in determining the agreement between experimental and simulated distillation column responses. Experimental measurements were performed to estimate parameter values, or to define a parameter range. The results of these measurements are documented in this appendix. In addition, parameter values calculated from several common correlations are also presented.

F.1 Sample Calculations for Experimental Holdup Measurement

First estimates were made of relative volumes of liquid on all trays, using tray 5 as the reference tray. Five independent estimates were made, with the column operating at steady state and the results of the different observers averaged.

Sample (for illustration only, not actual data):

TRAY	1	2	3	4	5	6	7	8
RATIO	1.2	1.2	1.1	1.1	1.0	0.9	0.95	0.85

The column was shut down abruptly; all input and output streams were stopped. All liquid was drained into a container and weighed. A density was determined and a volume calculated.

total weight of solution in column	20 kg
average composition (measured)	25% MeOH
density	.952 kg/l
volume	$20 \text{ kg} / .952 \text{ kg/l}$
	$= 21.0 \text{ l}$

The measured volumes of the reboiler and condenser, being constant were subtracted:

measured reboiler volume	7.3 l
measured condenser volume	1.7 l

Then the total tray volume was calculated

total tray volume $21.0 - 7.3 - 1.7 = 12.0 \text{ l}$

The estimated ratios were converted into fractions and the total tray volume multiplied by the fraction to obtain individual tray volumes. An approximate density was applied to each tray, yielding the tray mass holdup, as given below in a sample set of data (not representing any actual measurements):

Tray	Ratio	Fraction	Volume (ℓ)	Density (kg/ℓ)	Mass (kg)
1	1.2	.145	1.73	1.0	1.73
2	1.2	.145	1.73	1.0	1.73
3	1.1	.133	1.59	.95	1.51
4	1.1	.133	1.59	.95	1.51
5	1.0	.120	1.45	.90	1.31
6	0.9	.108	1.30	.90	1.17
7	0.95	.114	1.37	.85	1.16
8	0.85	.102	1.23	.85	1.04
Total	8.3				

Fraction $1.2/8.3 = .145$ for first tray

A summary of all estimated ratios and the corresponding tray holdups for a variety of operating conditions is given in Table F.1

F.2 Attempts At Correlation of Measured Holdup

With the nonlinear model, the steady state terminal compositions were matched with their experimental counterparts, by fitting efficiency and heat loss parameters in the model. The model yielded also intermediate compositions and flow-rates, which were used as independent variables in a correlation technique.

Simulated steady state values of liquid flow rates, vapor flow rates and corresponding measured tray mass holdups were used as input data to a correlation program on an IBM 370 at the University of Alberta. The name of the

TABLE F.1

EXPERIMENTAL HOLDUP DATA

Operating Conditions	test no.	1	2	3	4	5	6
feed flow (g/s)		18.0	18.0	19.8	16.3	18.0	21.6
reflux flow (g/s)		18.0	18.0	18.0	18.0	18.3	18.0
steam flow (g/s)		18.5	15.0	15.0	15.0	14.5	14.0
total tray holdup (kg)		18.2	18.4	18.5	15.4	18.6	18.7
average ratios (basis tray 5 = 1.0) estimates							
tray 1		0.81	1.03	1.04	0.97	1.15	1.27
tray 2		0.85	1.05	1.02	0.96	1.30	1.27
tray 3		0.91	1.06	0.86	0.98	1.18	1.19
tray 4		0.95	1.08	0.98	0.94	1.20	1.14
tray 5		1.0	1.0	1.00	1.0	1.0	1.0
tray 6		1.12	0.99	1.05	1.13	1.0	1.0
tray 7		0.96	1.08	1.21	1.10	0.98	1.0
tray 8		0.84	0.86	1.06	0.91	0.98	0.90
tray holdups (kg)							
tray 1		1.88	1.36	1.34	0.93	1.12	1.51
tray 2		1.09	1.33	1.31	0.89	1.27	1.51
tray 3		1.17	1.33	1.07	0.91	1.14	1.33
tray 4		1.14	1.28	1.21	0.82	1.17	1.28
tray 5		1.20	1.20	1.17	0.87	0.97	1.07
tray 6		1.24	1.08	1.27	0.90	0.97	1.07
tray 7		1.07	1.18	1.22	0.88	0.87	1.01
tray 8		0.90	0.91	1.18	0.70	0.87	0.90

correlation (regression) program was MLREGR and the desired format of the correlation is given below, where P denotes fitting parameter.

$$WT = P_1 L^{P_2} + P_3 V^{P_4} + P_5$$

The regression gave no meaningful results, due to the excessive scatter of the data. This data is presented in Figure 5.10, where holdup is plotted against liquid flow rate, and where it may be observed that there is no discernible pattern.

It would have been expected that even using only liquid and vapor flow as independent variables that some valid correlation would have resulted. Perhaps the visual observation technique led to such variation that any trends were obscured. Even when tray holdups are plotted against feed flow rate such as in Figures F.1 to F.4, considerable scatter is observed, although a general trend is discernible. An increase in feed rate appears to bring about an increase in holdup on all trays. The holdup values in these figures represent a graphical summary of all values that could be calculated from the different independent observations.

F.3 Efficiencies Prediction

An experimental and a mathematical technique was used to establish a range of efficiency that would be used in the

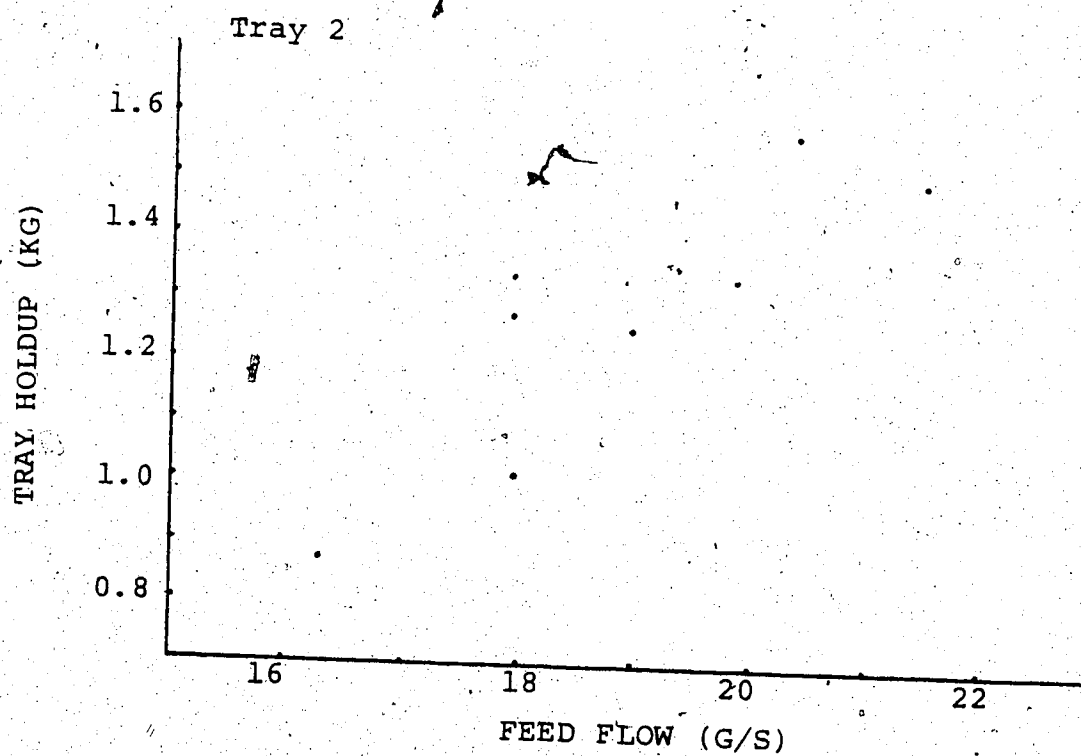
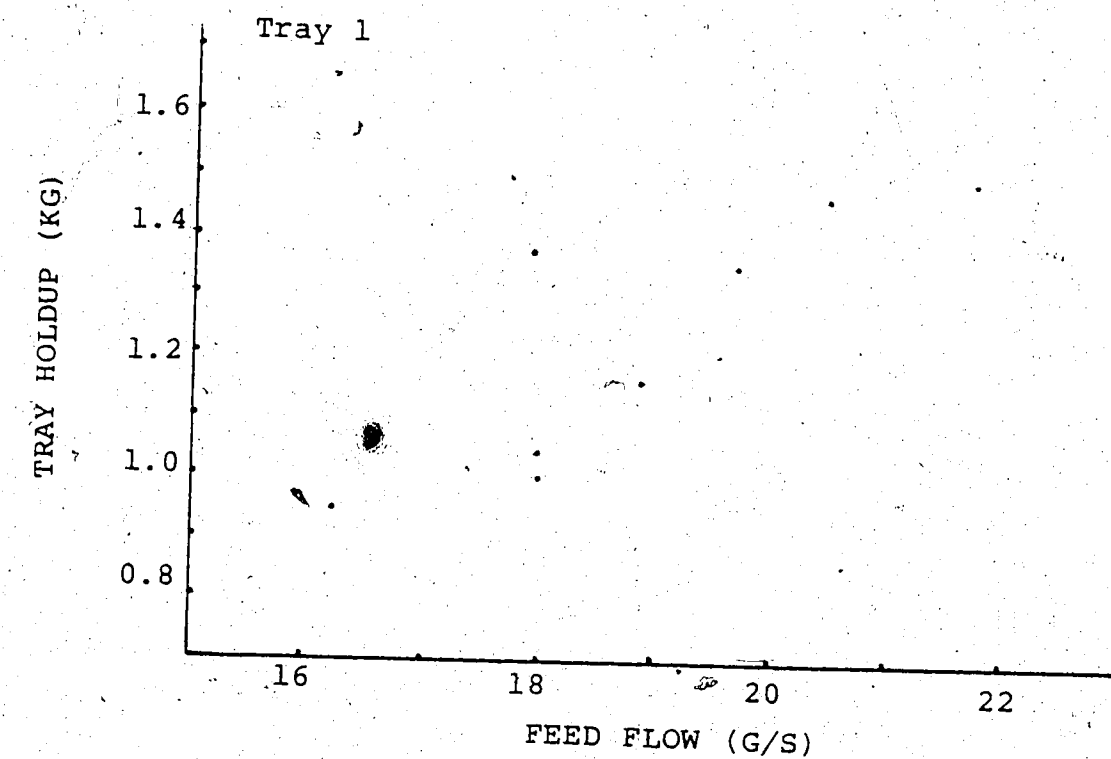


FIGURE F.1 Experimental Holdup Estimates,
Trays 1 & 2

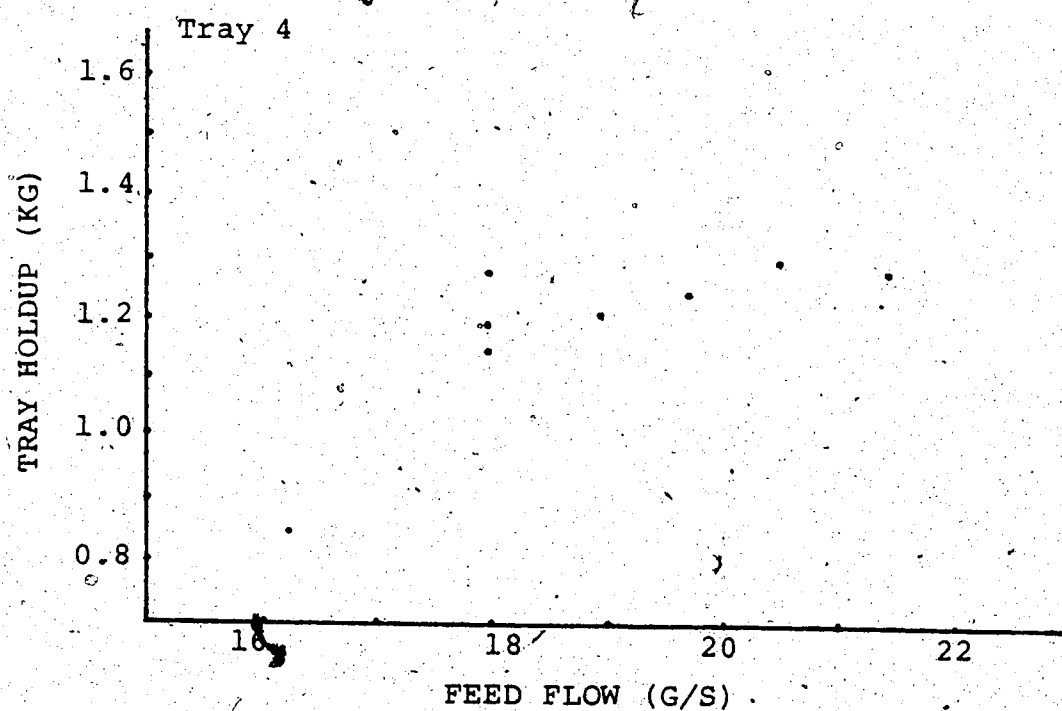
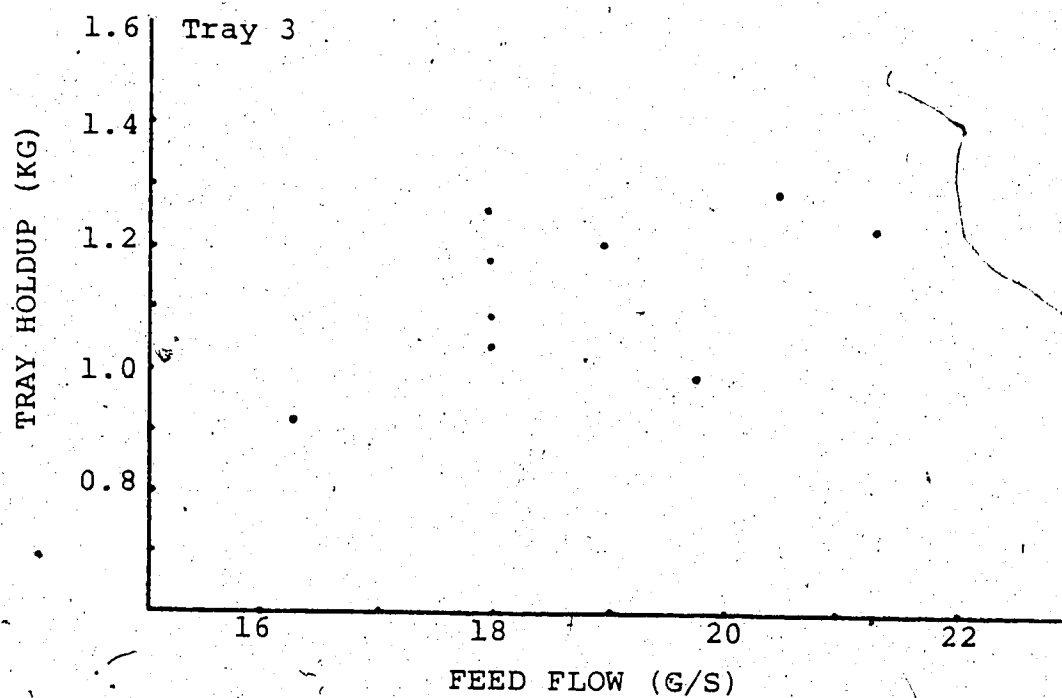


FIGURE F.2 Experimental Holdup Estimates,
Trays 3 & 4

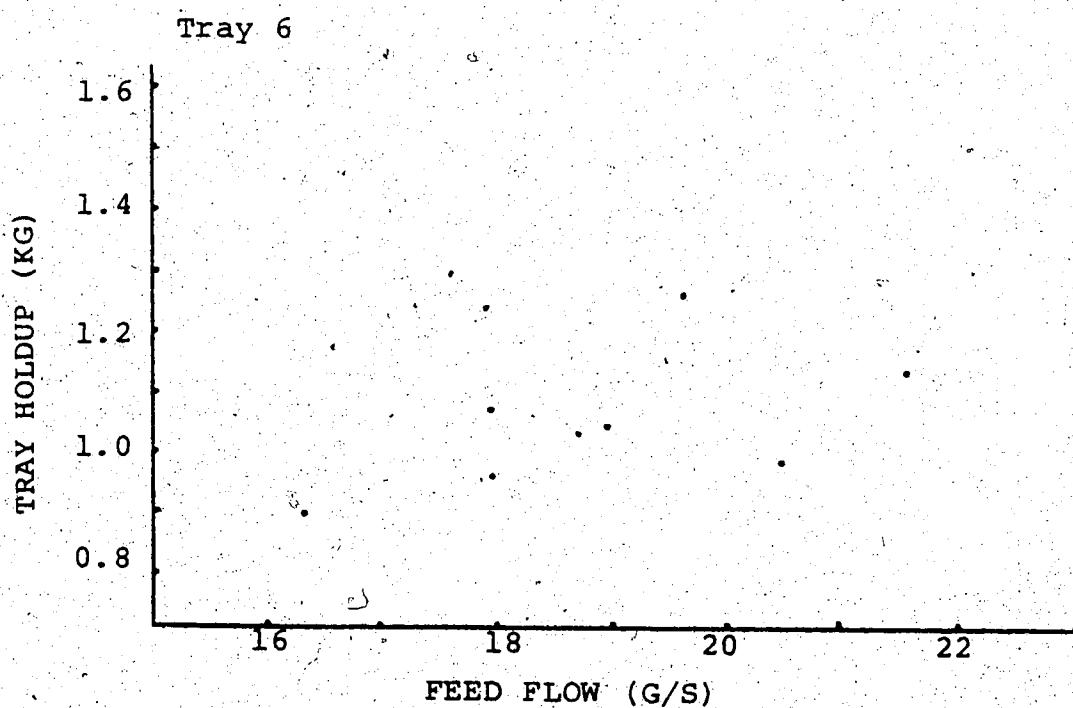
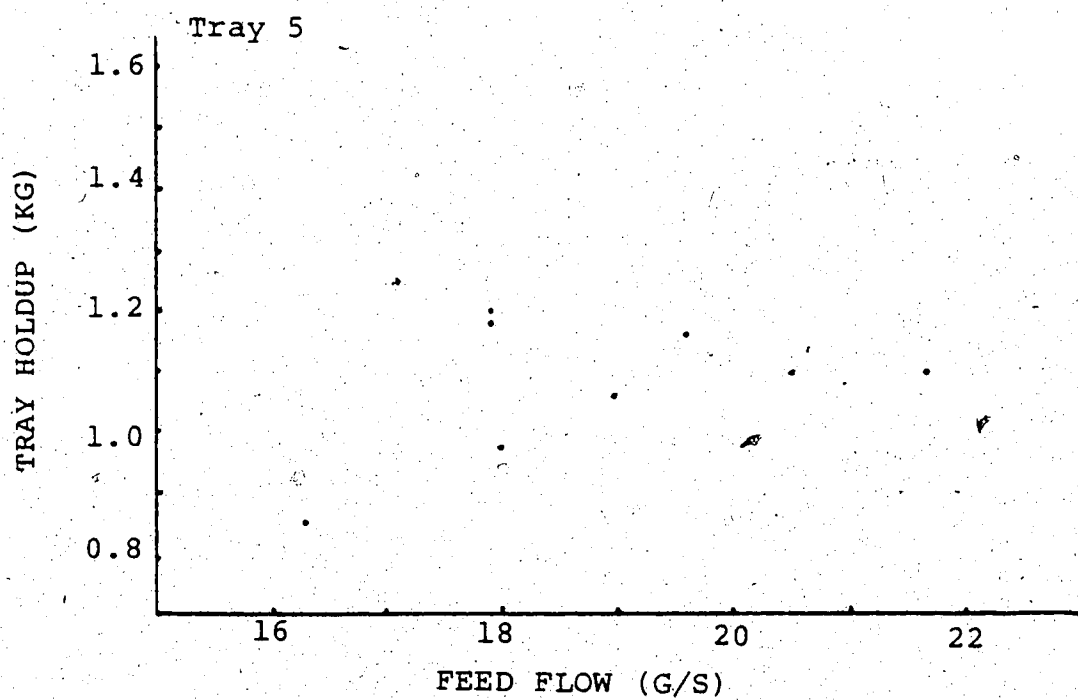


FIGURE F.3 Experimental Holdup Estimates,
Trays 5 & 6

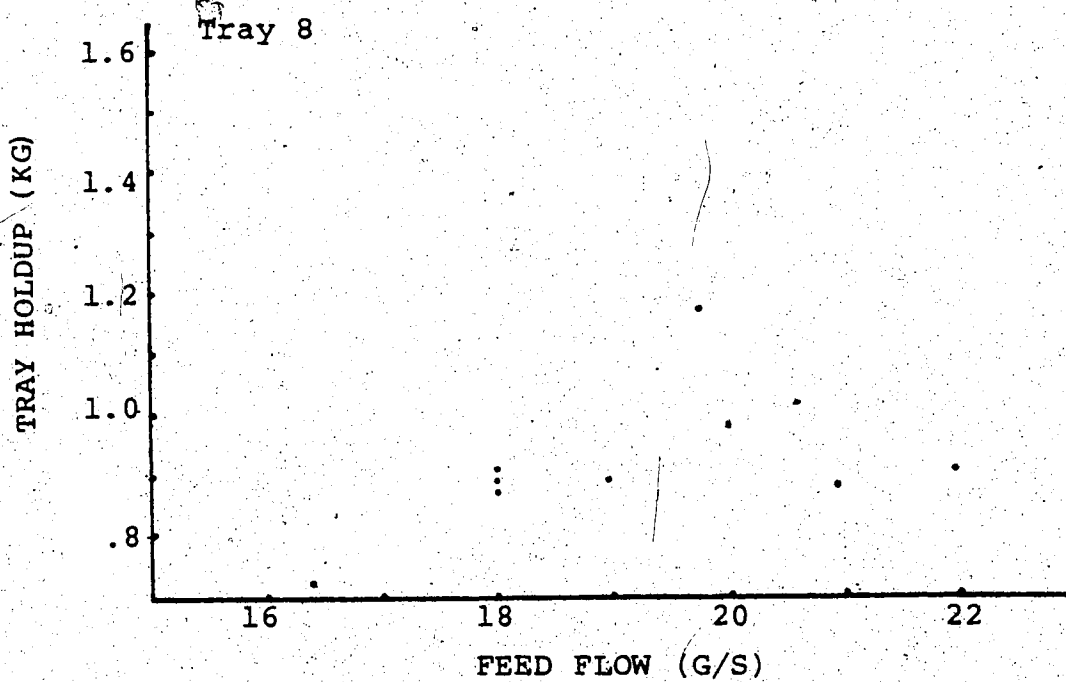
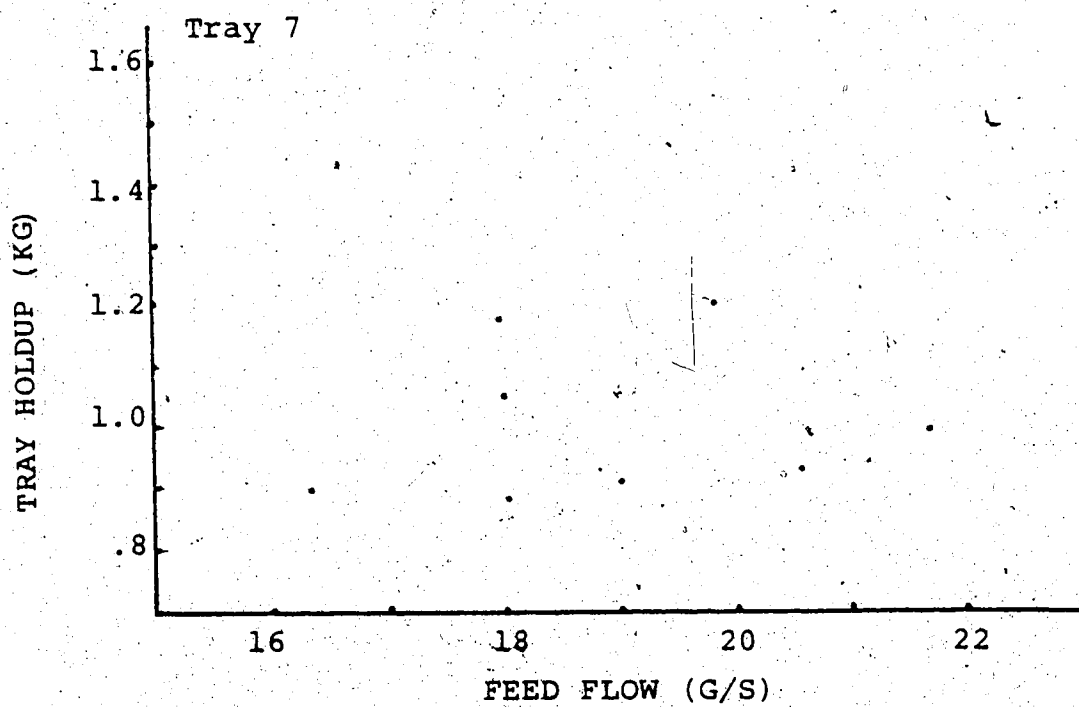


FIGURE F.4 Experimental Holdup Estimates,
Trays 7 & 8

simulation.

At steady state, liquid samples were taken from each stage of the distillation column. The samples were subsequently analyzed by a gas chromatograph and a column composition profile was determined. This profile was matched by simulation with the mathematical model. With the same operating conditions, efficiencies were changed by trial and error, to give new steady states. The efficiencies that gave an initial steady state composition profile within a few percent of that measured are shown in Table F.2.

This method has two disadvantages. First, the accuracy of the mathematical model must be accepted, before actual comparison to experimental results are carried out. Secondly, it is a time consuming and expensive procedure, in terms of computation, since the values must be established by trial and error. If vapor compositions could be sampled and measured, efficiencies could be calculated directly.

The only prediction attempted was done using the recommended AIChE efficiency prediction for bubble cap trays. This method has limitations (see sample calculations, Appendix C) for pilot plant scale operations, and the calculated estimates are included in Table F.2. The Peclet numbers offer an indication of the extent of mixing that takes place; a Pe of near 1 indicates good mixing, while a higher Pe of greater

TABLE F.2
EFFICIENCIES PREDICTION

Tray	Composition Measured	Composition Simulated	Efficiency Simulated	Efficiency Calculated	Peclet Number
1	3.126	3.09	96.4	70.5	1.42
2	11.971	12.29	92.43	66.8	1.31
3	32.043	31.78	89.74	74.6	1.26
4	38.339	38.20	74.35	78.9	1.11
5	56.234	56.82	89.90	78.5	.416
6	73.504	73.05	87.00	77.5	.471
7	80.702	83.68	82.10	76.2	.526
8	89.759	90.77	80.10	75.3	.573
R	.758	.81	100.00	-	-
C	95.63	95.59	-	-	-

than 5 indicates an approach to plug flow. The bubble cap trays here all appear to be well mixed.

F.4 Estimation of Column Heat Loss

As presented in Appendix B, the programs DASS, DATAL and BALNC calculate an energy balance on the distillation column, with the difference between energy input and removal taken as heat loss. This heat loss was found to be due to losses from the column walls and losses from the gas chromatograph recycle system. The combined heat loss as calculated by the DASS program was of the order of 8-9 kJ/s. To verify these calculations to some degree, the following heat loss calculation was performed.

a) column

7 trays - 23 cm diam x 30 cm height

1 tray - 20 cm x 60 cm + 9 cm x 120 cm

1 reboiler + glass below first tray $\approx$ area of

1 1/2 regular trays.

heat loss due to convection (45)

$q = h A(T - T_o)$ where T_o is the ambient temperature assuming natural convection, laminar flow around column, for vertical cylinders⁴⁵

$$h = 0.29 \left(\frac{\Delta T}{L} \right)^{1/4} \quad [\text{BTU/hr ft}^2 \text{ } ^\circ\text{F}]$$

where ΔT [$^{\circ}\text{F}$]

L cylinder height [ft]

Using the temperatures predicted by the computer simulations, heat transfer coefficients are calculated. Due to the nature of the correlations, calculations were done in Engineering units, and results were converted into SI units.

TABLE F.3
CONVECTIVE HEAT LOSS

Stage	T ($^{\circ}\text{F}$)	$\Delta T/L$ ($^{\circ}\text{F}/\text{ft}$)	$(\Delta T/L)^{0.25}$	h (BTU/hr $\text{ft}^2\text{ }^{\circ}\text{F}$)	A ₂ (ft^2)	Q (Kj/sec)
1	212	142	3.45	1.00	2.65	.110
2	205	135	3.41	0.99	1.77	.068
3	196	126	3.35	0.97	1.77	.063
4	187	117	3.29	0.95	1.77	.057
5	178	108	3.22	0.93	1.77	.052
6	171	101	3.17	0.92	1.77	.048
7	163	93	3.11	0.90	1.77	.043
8	156	86	3.05	0.89	1.77	.040
9	149	79	2.98	0.86	4.90	.097

heat loss due to radiation (45)

$$q = A F \sigma e (T_g^4 - T_r^4) \text{ BTU/hr}$$

A = surface area (ft^2)

F = shape factor (1)

$$\sigma = 0.1714 \times 10^{-8} \text{ BTU/hr ft}^2\text{ }^{\circ}\text{R}^4$$

e = emissivity (.90 for pyrex glass)

T_g = temperature of glass column

TABLE F.4

HEAT LOSS DUE TO RADIATION

Stage	T_g (°R)	T_g^4 (°R ⁴) (x10 ¹¹)	$T_g^4 - T_o^4$ (°R ⁴) (x10 ¹¹)	A (ft ²)	Q (joules/sec) (x 10 ⁻²)	Q _{TOTAL} (Kj/s)
1	672	2.04	1.25	2.65	0.15	.26
2	665	1.96	1.17	1.77	0.093	.17
3	656	1.85	1.06	1.77	0.085	.16
4	647	1.75	0.96	1.77	0.077	.14
5	638	1.66	0.87	1.77	0.069	.12
6	631	1.59	0.80	1.77	0.064	.11
7	623	1.51	0.72	1.77	0.058	.10
8	616	1.44	0.65	1.77	0.053	.09
9	609	1.38	0.59	4.90	0.130	.23

Note that the total of convective and radiation heat loss (the sum of the last column in Table F.4) explains one quarter of the steady state heat loss.

b) G.C. recycle system

Heat loss was not rigorously calculated here due to the complex configuration of the recycle system; the circuit included a small heat exchanger, a pump, lengths of tubing of different sizes and materials and various other points of contact with metal surfaces in the system. Instead, the material and energy balance programs DASS, DATAC and BALNC were run with and without the recycle system operative. From

these runs, it was found that half of the total heat loss could be attributed to the G.C. sampling system.

F.5 Energy Storage in Column Material

As pointed out by Rademaker et al.³², the storage of energy in the metal and glass of the column may be an important enthalpy consideration, more important perhaps than the energy storage in the liquid holdup on the tray. This effect was introduced into the model in the enthalpy balance.

Volume of metal in a tray:

plate itself	3/8" x 14" diam (less cut-out area)	55.60	(55.60)
downcomer	1" OD x 11 7/8" x .062"	2.34	
	1 1/2" OD x 1 1/4" x .062"	.37	(2.71)
downcomer	1 1/2" OD x 2" x .062"	.58	
outlet wier	1 7/8" OD plate x .062"	.17	(0.75)
bubble cap (4)	1 7/8" OD x 2 1/4" x .062"	.82	
	1 1/4" OD x 1 7/8" x .062"	.46	
	1 7/8" OD plate x .062"	.17	
	1 1/4" OD plate x 1/8"	.153	(6.412)
TOTAL		65.5 in ³	

Total volume	.0378 ft ³
Stainless steel density	488 lbm/ft ³
Total weight	18.45 lbm
Heat capacity	0.11 BTU/lbm°F

To raise tray metal one degree Celsius, the amount of heat

required is:

$$\begin{aligned}
 Q &= mC_p \\
 &= 18.4 \text{ lbm} (0.011 \text{ BTU/lbm}^\circ\text{F}) (1.8^\circ\text{F}/^\circ\text{C}) (1.054 \text{ Kj/BTU}) \\
 &= 3.83 \text{ Kj}
 \end{aligned}$$

Similarly, for the tray glass,

8" diam x 12" x 1/8"	37.7 in ³
total volume	.0218 ft ³
glass density	170 lbm/ft ³
heat capacity	0.2 BTU/lbm ^o F

Energy storage per °C is:

$$\begin{aligned}
 Q &= mC_p \\
 &= 4.17 \text{ lbm} (0.2 \text{ BTU/lbm}^\circ\text{F}) (1.054 \text{ Kj/BTU}) (1.8 \text{ }^\circ\text{C}/^\circ\text{F}) \\
 &= 1.58 \text{ Kj}
 \end{aligned}$$

Energy storage per tray and glass section per °C is thus:

$$1.58 + 3.83 \text{ Kj}$$

or

$$5.41 \text{ Kj}$$

APPENDIX G

EXPERIMENTAL STEADY STATES

The pages following present a summary of the terminal operating states of the distillation column open loop responses presented in this thesis; the run number codes are explained below:

OL104	-20% Feed Flow
OL105	+20% Feed Flow
OL106	+20% Reflux Flow
OL107	-20% Reflux Flow
OL108	+10% Steam Flow
OL109	-10% Steam Flow

B Beginning

E End

STEADY STATE DATA
 RUN NO. PL1048
 01.06/76

FEED FLOW	16.97G/SEC	BOTTOM PROD	8.47G/SEC
REFLUX FLOW	13.52G/SEC	TOP PROD	8.46G/SEC
STEAM FLOW	14.93G/SEC	COOL WATER	514.95G/SEC
FEED PLATE	14	FEED COMP	50.00%T _W F0H
DIST COMP	96.57%T _W F0H	BOTTOMS COMP	6.00%T _W F0H
FEED INLET	74.30DEG C	REFLUX INLET	65.30DEG C
STEAM TEMP	103.70DEG C	PRESSURE	-30.3KPA

MATERIAL BALANCE

	FLOW (G/SEC)	COMP (WT PCT)	ETHANOL (G/SEC)	WATER (G/SEC)
FEED	16.97	50.00	8.48	8.48
BOTTOM PRODUCT	8.67	6.00	0.52	8.15
TOP PRODUCT	8.46	96.57	8.17	0.28
CLOSURE ERROR-%	0.9		2.4	-0.5

ENERGY BALANCE

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	46.19	71.95
REFLUX	2.69	2.55
TOP PRODUCT		1.59
FEED	4.61	
STEAM	40.23	6.63
BOTTOM PRODUCT		3.37
TOTAL	93.73	86.11
HEAT LOSS		7.6

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 011048 01.06/76

FEED FLOW	= 16.970 G/SEC	DEV= 0.0428
REFLUX FLOW	= 13.528 G/SEC	DEV= 0.1883
STEAM FLOW	= 14.932 G/SEC	DEV= 0.2170
BOTTOM PROD	= 8.672 G/SEC	DEV= 0.0597
TOP PROD	= 8.462 G/SEC	DEV= 0.1205
COOL WATER	= 514.050 G/SEC	DEV= 15.0580
DIST COMP	= 96.578 WT% MEQH	DEV= 0.1134
BOTTOMS COMP	= 6.000 WT% MEQH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEQH	DEV= 0.0000
PRESSURE	= -30.357 KPA	DEV= 0.8900
COND LEVEL	= 17.942 CM	DEV= 0.0508
REB'R LEVEL	= 35.988 CM	DEV= 0.2913
DIFF PRESS	= -6.510 KPA	DEV= 0.0830
REB'R D'HEAD	= 93.6 DEG C	DEV= 0.1810
PLATE 1 TEMP	= 82.7 DEG C	DEV= 0.1679
PLATE 2 TEMP	= 77.4 DEG C	DEV= 0.1470
PLATE 3 TEMP	= 74.7 DEG C	DEV= 0.1305
PLATE 4 TEMP	= 74.2 DEG C	DEV= 0.1390
PLATE 5 TEMP	= 69.8 DEG C	DEV= 0.1295
PLATE 6 TEMP	= 67.2 DEG C	DEV= 0.1277
PLATE 7 TEMP	= 65.8 DEG C	DEV= 0.1168
PLATE 8 TEMP	= 64.6 DEG C	DEV= 0.1223
COND TEMP	= 62.7 DEG C	DEV= 0.1371
STEAM TEMP	= 106.7 DEG C	DEV= 0.1868
COND'T TEMP	= 105.2 DEG C	DEV= 0.1488
REFLUX TEMP	= 53.2 DEG C	DEV= 0.1411
FEED TEMP	= 38.6 DEG C	DEV= 0.1030
BOTTOMS TEMP	= 48.6 DEG C	DEV= 0.1145
REB'R TEMP	= 93.6 DEG C	DEV= 0.1829
FEED INLET	= 74.3 DEG C	DEV= 0.1361
REFLUX INLET	= 65.3 DEG C	DEV= 0.3502
COL D'HEAD	= 64.8 DEG C	DEV= 0.1147
WATER INLET	= 21.4 DEG C	DEV= 0.0970
WATER OUTLET	= 33.4 DEG C	DEV= 0.3214

STEADY STATE DATA
 RUN NO 01104E
 01/06/76

FEED FLOW	13.61G/SEC	BOTTOM PROD	6.24G/SEC
REFLUX FLOW	13.49G/SEC	TOP PROD	7.20G/SEC
STEAM FLOW	14.98G/SEC	COOL WATER	509.43G/SEC
FEED PLATE	4	FEED COMP	50.00WT% METH
DIST COMP	95.87WT% METH	BOTTOMS COMP	0.20WT% METH
FEED INLET	73.10DEG C	REFLUX INLET	64.30DEG C
STEAM TEMP	110.00DEG C	PRESSURE	-30.1KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	13.61	50.00	6.80	6.80
BOTTOM PRODUCT	6.24	0.20	0.01	6.23
TOP PRODUCT	7.20	95.87	6.91	0.29
CLOSURE ERROR-PC	-1.1		1.7	-4.0

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	42.26	67.53
REFLUX	2.63	2.50
TOP PRODUCT		1.33
FEED	3.63	
STEAM	40.46	6.92
BOTTOM PRODUCT		2.56
TOTAL	88.99	80.86
HEAT LOSS		8.1

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 01104E 01/06/76

FEED FLOW	= 13.610 G/SEC	DEV= 0.0950
REFLUX FLOW	= 13.498 G/SEC	DEV= 0.1982
STEAM FLOW	= 14.988 G/SEC	DEV= 0.2437
BOTTOM PROD	= 6.243 G/SEC	DEV= 0.0788
TOP PROD	= 7.208 G/SEC	DEV= 0.0971
COOL WATER	= 509.431 G/SEC	DEV= 13.4008
DIST COMP	= 95.873 WT% MEQH	DEV= 0.1535
BOTTOMS COMP	= 0.200 WT% MEQH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEQH	DEV= 0.0000
PRESSURE	= -30.102 KPA	DEV= 0.5629
COND LEVEL	= 17.912 CM	DEV= 0.0555
REB'R LEVEL	= 34.907 CM	DEV= 0.2771
DIFF PRESS	= -7.609 KPA	DEV= 0.1013
REB'R D'HEAD	= 97.7 DEG C	DEV= 0.1794
PLATE 1 TEMP	= 95.1 DEG C	DEV= 0.1573
PLATE 2 TEMP	= 89.5 DEG C	DEV= 0.2057
PLATE 3 TEMP	= 82.3 DEG C	DEV= 0.1701
PLATE 4 TEMP	= 77.4 DEG C	DEV= 0.1746
PLATE 5 TEMP	= 71.2 DEG C	DEV= 0.1300
PLATE 6 TEMP	= 67.5 DEG C	DEV= 0.1286
PLATE 7 TEMP	= 65.5 DEG C	DEV= 0.1293
PLATE 8 TEMP	= 63.8 DEG C	DEV= 0.1240
COND TEMP	= 61.6 DEG C	DEV= 0.1450
STEAM TEMP	= 110.0 DEG C	DEV= 0.2194
COND'T TEMP	= 109.2 DEG C	DEV= 0.2217
REFLUX TEMP	= 51.8 DEG C	DEV= 0.1266
FEED TEMP	= 37.4 DEG C	DEV= 0.0970
BOTTOMS TEMP	= 45.6 DEG C	DEV= 0.1128
REB'R TEMP	= 97.7 DEG C	DEV= 0.1982
FEED INLET	= 73.1 DEG C	DEV= 0.2494
REFLUX INLET	= 64.3 DEG C	DEV= 0.2551
COL D'HEAD	= 64.1 DEG C	DEV= 0.1336
WATER INLET	= 19.8 DEG C	DEV= 0.0940
WATER OUTLET	= 31.7 DEG C	DEV= 0.1586

STEADY STATE DATA
 RUN NO 011058
 02/06/6

FEED FLOW	16.99G/SEC	BOTTOM PROD	8.92G/SEC
REFLUX FLOW	13.55G/SEC	TOP PROD	8.32G/SEC
STEAM FLOWW	14.80G/SEC	COOL WATER	287.10G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEON
DIST COMP	96.45WT% MEON	BOTTOMS COMP	4.40WT% MEON
FEED INLET	72.2DEG C	REFLUX INLET	63.5DEG C
STEAM TEMP	105.8DEG C	PRESSURE	-29.0KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.99	50.00	8.49	8.49
BOTTOM PRODUCT	8.92	4.40	0.39	8.53
TOP PRODUCT	8.32	96.45	8.02	0.29
CLOSURE ERROR-PC	1.5		-0.9	3.9

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	23.82	46.98
REFLUX	2.60	2.48
TOP PRODUCT		1.52
FEED	4.46	
STEAM	39.86	6.53
BOTTOM PRODUCT		3.46
TOTAL	70.75	60.99
HEAT LOSS		9.7

STEADY STATE CONDITIONS BASED ON 20 POINTS
 RUN NO 0L105B 02/06/6

FEED FLOW	= 16.992 G/SEC	DEV= 0.0348
REFLUX FLOW	= 13.552 G/SEC	DEV= 0.1919
STEAM FLOWW	= 14.801 G/SEC	DEV= 0.1979
BOTTOM PROD	= 8.927 G/SEC	DEV= 0.0458
TOP PROD	= 8.320 G/SEC	DEV= 0.1058
COOL WATER	= 287.107 G/SEC	DEV= 8.0902
DIST COMP	= 96.452 WT% MEOH	DEV= 0.0811
BOTTOMS COMP	= 4.400 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -29.037 KPA	DEV= 0.5072
COND LEVEL	= 17.714 CM	DEV= 0.0421
REB'R LEVEL	= 42.509 CM	DEV= 0.2438
DIFF PRESS	= -6.863 KPA	DEV= 0.0889
REB'R O'HEAD	= 93.0 DEG C	DEV= 0.1216
PLATE 1 TEMP	= 82.5 DEG C	DEV= 0.1451
PLATE 2 TEMP	= 76.5 DEG C	DEV= 0.1125
PLATE 3 TEMP	= 73.3 DEG C	DEV= 0.1308
PLATE 4 TEMP	= 72.7 DEG C	DEV= 0.0769
PLATE 5 TEMP	= 68.1 DEG C	DEV= 0.1269
PLATE 6 TEMP	= 65.5 DEG C	DEV= 0.0736
PLATE 7 TEMP	= 64.0 DEG C	DEV= 0.1072
PLATE 8 TEMP	= 62.8 DEG C	DEV= 0.0897
COND TEMP	= 61.2 DEG C	DEV= 0.0959
STEAM TEMP	= 105.8 DEG C	DEV= 0.1440
COND'T TEMP	= 104.6 DEG C	DEV= 0.1393
REFLUX TEMP	= 51.7 DEG C	DEV= 0.1143
FEED TEMP	= 35.8 DEG C	DEV= 0.0883
BOTTOMS TEMP	= 46.6 DEG C	DEV= 0.0976
REB'R TEMP	= 92.9 DEG C	DEV= 0.0977
FEED INLET	= 72.2 DEG C	DEV= 0.1439
REFLUX INLET	= 63.5 DEG C	DEV= 0.3441
COL O'HEAD	= 63.0 DEG C	DEV= 0.0897
WATER INLET	= 19.8 DEG C	DEV= 0.0779
WATER OUTLET	= 39.1 DEG C	DEV= 0.1477

STEADY STATE DATA
 RUN NO OL105E
 02/06/76

FEED FLOW	20.43G/SEC	BOTTOM PROD	12.12G/SEC
REFLUX FLOW	13.55G/SEC	TOP PROD	9.03G/SEC
STEAM FLOW	15.12G/SEC	COOL WATER	317.86G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEON
DIST COMP	96.45WT% MEON	BOTTOMS COMP	12.00WT% MEON
FEED INLET	72.4DEG C	REFLUX INLET	64.0DEG C
STEAM TEMP	102.2DEG C	PRESSURE	-28.9KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	20.43	50.00	10.21	10.21
BOTTOM PRODUCT	12.12	12.00	1.45	10.66
TOP PRODUCT	9.03	96.45	8.71	0.32
CLOSURE ERROR-PC	3.5		-0.4	7.5

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	27.11	51.83
REFLUX	2.62	2.49
TOP PRODUCT		1.68
FEED	5.39	
STEAM	40.64	6.37
BOTTOM PRODUCT		4.36
TOTAL	75.78	66.73
HEAT LOSS		9.0

STEADY STATE CONDITIONS BASED ON 20 POINTS
 RUN NO 0L105E 02/06/76 -

FEED FLOW	= 20.430 G/SEC	DEV= 0.0368
REFLUX FLOW	= 13.557 G/SEC	DEV= 0.1511
STEAM FLOWW	= 15.126 G/SEC	DEV= 0.2846
BOTTOM PROD	= 12.120 G/SEC	DEV= 0.4370
TOP PROD	= 9.031 G/SEC	DEV= 0.0895
COOL WATER	= 317.868 G/SEC	DEV= 18.0728
DIST COMP	= 96.455 WT% MEOH	DEV= 0.0791
BOTTOMS COMP	= 12.000 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -28.932 KPA	DEV= 0.6574
COND LEVEL	= 17.749 CM	DEV= 0.0410
REB'R LEVEL	= 44.476 CM	DEV= 1.7406
DIFF PRESS	= -6.116 KPA	DEV= 0.1655
REB'R O'HEAD	= 87.8 DEG C	DEV= 0.1393
PLATE 1 TEMP	= 77.8 DEG C	DEV= 0.1288
PLATE 2 TEMP	= 74.5 DEG C	DEV= 0.1237
PLATE 3 TEMP	= 72.9 DEG C	DEV= 0.1295
PLATE 4 TEMP	= 72.6 DEG C	DEV= 0.1295
PLATE 5 TEMP	= 68.5 DEG C	DEV= 0.1189
PLATE 6 TEMP	= 66.0 DEG C	DEV= 0.1168
PLATE 7 TEMP	= 64.5 DEG C	DEV= 0.1026
PLATE 8 TEMP	= 63.3 DEG C	DEV= 0.1080
COND TEMP	= 61.5 DEG C	DEV= 0.0993
STEAM TEMP	= 102.2 DEG C	DEV= 0.1333
COND'T TEMP	= 99.9 DEG C	DEV= 0.1416
REFLUX TEMP	= 52.3 DEG C	DEV= 0.1351
FEED TEMP	= 36.2 DEG C	DEV= 0.1023
BOTTOMS TEMP	= 49.8 DEG C	DEV= 0.0869
REB'R TEMP	= 87.8 DEG C	DEV= 0.1439
FEED INLET	= 72.4 DEG C	DEV= 0.1617
REFLUX INLET	= 64.0 DEG C	DEV= 0.3211
COL O'HEAD	= 63.5 DEG C	DEV= 0.1026
WATER INLET	= 20.4 DEG C	DEV= 0.0895
WATER OUTLET	= 39.0 DEG C	DEV= 0.2091

STEADY STATE DATA
 RUN NO 0L1068
 05/06/76

FEED FLOW	16.99G/SEC	BOTTOM PROD	9.06G/SEC
REFLUX FLOW	13.59G/SEC	TOP PROD	8.12G/SEC
STEAM FLOW	14.96G/SEC	COOL WATER	287.64G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MECH
DIST COMP	96.50WT% MECH	BOTTOMS COMP	3.80WT% MECH
FEED INLET	72.6DEG C	REFLUX INLET	63.9DEG C
STEAM TEMP	107.4DEG C	PRESSURE	-28.9KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.99	50.00	8.49	8.49
BOTTOM PRODUCT	9.06	3.80	0.34	8.71
TOP PRODUCT	8.12	96.50	7.83	0.28
CLOSURE ERROR-PC	1.1		-3.6	5.9

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	23.87	47.59
REFLUX	2.63	2.51
TOP PRODUCT		1.49
FEED	4.49	
STEAM	40.34	6.72
BOTTOM PRODUCT		3.55
TOTAL	71.35	61.88
HEAT LOSS		9.4

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 0L106B 05/06/76

FEED FLOW	= 16.990 G/SEC	DEV= 0.0473
REFLUX FLOW	= 13.596 G/SEC	DEV= 0.1908
STEAM FLOWW	= 14.967 G/SEC	DEV= 0.1997
BOTTOM PROD	= 9.062 G/SEC	DEV= 0.0583
TOP PROD	= 8.120 G/SEC	DEV= 0.1266
COOL WATER	= 287.641 G/SEC	DEV= 14.4864
DIST COMP	= 96.509 WT% MEOH	DEV= 0.1507
BOTTOMS COMP	= 3.800 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -28.931 KPA	DEV= 0.6172
COND LEVEL	= 17.166 CM	DEV= 0.0888
REB'R LEVEL	= 40.636 CM	DEV= 0.2578
DIFF PRESS	= -5.913 KPA	DEV= 0.2248
REB'R O'HEAD	= 93.9 DEG C	DEV= 0.1889
PLATE 1 TEMP	= 83.4 DEG C	DEV= 0.1621
PLATE 2 TEMP	= 77.2 DEG C	DEV= 0.1366
PLATE 3 TEMP	= 114.0 DEG C	DEV= 0.2070
PLATE 4 TEMP	= 73.2 DEG C	DEV= 0.1469
PLATE 5 TEMP	= 68.5 DEG C	DEV= 0.1456
PLATE 6 TEMP	= 66.0 DEG C	DEV= 0.1403
PLATE 7 TEMP	= 64.5 DEG C	DEV= 0.1246
PLATE 8 TEMP	= 63.3 DEG C	DEV= 0.1238
COND TEMP	= 61.6 DEG C	DEV= 0.1374
STEAM TEMP	= 107.4 DEG C	DEV= 0.2012
COND'T TEMP	= 106.3 DEG C	DEV= 0.2012
REFLUX TEMP	= 52.1 DEG C	DEV= 0.1289
FEED TEMP	= 36.4 DEG C	DEV= 0.1035
BOTTOMS TEMP	= 47.1 DEG C	DEV= 0.1141
REB'R TEMP	= 93.9 DEG C	DEV= 0.1989
FEED INLET	= 72.6 DEG C	DEV= 0.1581
REFLUX INLET	= 63.9 DEG C	DEV= 0.2741
COL O'HEAD	= 63.5 DEG C	DEV= 0.1369
WATER INLET	= 19.8 DEG C	DEV= 0.1111
WATER OUTLET	= 39.5 DEG C	DEV= 0.2420

STEADY STATE DATA
 RUN NO OL106E
 05/06/76

FEED FLOW	16.99G/SEC	BOTTOM PROD	10.49G/SEC
REFLUX FLOW	16.18G/SEC	TOP PROD	6.71G/SEC
STEAM FLOW	14.99G/SEC	COOL WATER	327.14G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEQH
DIST COMP	97.09WT% MEQH	BOTTOMS COMP	17.00WT% MEQH
FEED INLET	72.0DEG C	REFLUX INLET	63.4DEG C
STEAM TEMP	101.2DEG C	PRESSURE	-28.8KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.99	50.00	8.49	8.49
BOTTOM PRODUCT	10.49	17.00	1.78	8.70
TOP PRODUCT	6.71	97.09	6.51	0.19
CLOSURE ERROR-PC	1.2		-2.2	4.8

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	26.09	51.79
REFLUX	3.09	2.94
TOP PRODUCT		1.22
FEED	4.45	
STEAM	40.26	6.24
BOTTOM PRODUCT		3.64
TOTAL	73.90	65.84
HEAT LOSS		8.0

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 01106E 05/06/76

FEED FLOW	= 16.990 G/SEC	DEV= 0.0533
REFLUX FLOW	= 16.186 G/SEC	DEV= 0.1587
STEAM FLOW	= 14.993 G/SEC	DEV= 0.1928
BOTTOM PROD	= 10.492 G/SEC	DEV= 0.0899
TOP PROD	= 6.711 G/SEC	DEV= 0.1211
COOL WATER	= 327.146 G/SEC	DEV= 12.3664
DIST COMP	= 97.099 WT% MEOH	DEV= 0.1334
BOTTOMS COMP	= 17.000 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -28.878 KPA	DEV= 0.4981
COND LEVEL	= 17.283 CM	DEV= 0.0521
REB'R LEVEL	= 43.577 CM	DEV= 0.4113
DIFF PRESS	= -4.372 KPA	DEV= 0.1229
REB'R O'HEAD	= 85.8 DEG C	DEV= 0.1758
PLATE 1 TEMP	= 75.7 DEG C	DEV= 0.1477
PLATE 2 TEMP	= 72.7 DEG C	DEV= 0.1421
PLATE 3 TEMP	= 116.0 DEG C	DEV= 0.1967
PLATE 4 TEMP	= 71.0 DEG C	DEV= 0.1204
PLATE 5 TEMP	= 66.7 DEG C	DEV= 0.1393
PLATE 6 TEMP	= 64.4 DEG C	DEV= 0.1089
PLATE 7 TEMP	= 63.2 DEG C	DEV= 0.1293
PLATE 8 TEMP	= 62.2 DEG C	DEV= 0.1088
COND TEMP	= 61.0 DEG C	DEV= 0.1091
STEAM TEMP	= 101.2 DEG C	DEV= 0.1889
COND'T TEMP	= 98.7 DEG C	DEV= 0.1907
REFLUX TEMP	= 51.4 DEG C	DEV= 0.1250
FEED TEMP	= 36.7 DEG C	DEV= 0.1063
BOTTOMS TEMP	= 47.4 DEG C	DEV= 0.1093
REB'R TEMP	= 85.7 DEG C	DEV= 0.1957
FEED INLET	= 72.0 DEG C	DEV= 0.1502
REFLUX INLET	= 63.4 DEG C	DEV= 0.3800
COL O'HEAD	= 62.4 DEG C	DEV= 0.1189
WATER INLET	= 19.0 DEG C	DEV= 0.0980
WATER OUTLET	= 37.8 DEG C	DEV= 0.2002

STEADY STATE DATA
 RUN NO QL107B
 06/05/76

FEED FLOW	16.97G/SEC	BOTTOM PROD	9.12G/SEC
REFLUX FLOW	13.47G/SEC	TOP PROD	8.06G/SEC
STEAM FLOWW	14.94G/SEC	COOL WATER	398.11G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEONH
DIST COMP	96.51WT% MEONH	BOTTOMS COMP	4.40WT% MEONH
FEED INLET	71.5DEG C	REFLUX INLET	62.7DEG C
STEAM TEMP	105.6DEG C	PRESSURE	-30.2KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.97	50.00	8.48	8.48
BOTTOM PRODUCT	9.12	4.40	0.40	8.71
TOP PRODUCT	8.06	96.51	7.78	0.28
CLOSURE ERROR-PC	1.2		-3.5	6.0

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	30.32	55.92
REFLUX	2.54	2.43
TOP PRODUCT		1.45
FEED	4.41	
STEAM	40.24	6.58
BOTTOM PRODUCT		3.50
TOTAL	77.53	69.90
HEAT LOSS		7.6

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 0L1078 06/05/76

FEED FLOW	= 16.971 G/SEC	DEV= 0.0417
REFLUX FLOW	= 13.476 G/SEC	DEV= 0.1639
STEAM FLOWW	= 14.946 G/SEC	DEV= 0.2142
BOTTOM PROD	= 9.121 G/SEC	DEV= 0.0495
TOP PROD	= 8.067 G/SEC	DEV= 0.1027
COOL WATER	= 398.115 G/SEC	DEV= 8.6673
DIST COMP	= 96.516 WT% MEOH	DEV= 0.1481
BOTTOMS COMP	= 4.400 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -30.246 KPA	DEV= 0.3451
COND LEVEL	= 17.225 CM	DEV= 0.0446
REB'R LEVEL	= 37.696 CM	DEV= 0.2523
DIFF PRESS	= -6.206 KPA	DEV= 0.0919
REB'R O'HEAD	= 92.1 DEG C	DEV= 0.1861
PLATE 1 TEMP	= 81.4 DEG C	DEV= 0.1602
PLATE 2 TEMP	= 75.5 DEG C	DEV= 0.1064
PLATE 3 TEMP	= 121.0 DEG C	DEV= 0.1964
PLATE 4 TEMP	= 71.9 DEG C	DEV= 0.1390
PLATE 5 TEMP	= 67.2 DEG C	DEV= 0.1323
PLATE 6 TEMP	= 64.7 DEG C	DEV= 0.1265
PLATE 7 TEMP	= 63.3 DEG C	DEV= 0.1236
PLATE 8 TEMP	= 62.1 DEG C	DEV= 0.1236
COND TEMP	= 60.5 DEG C	DEV= 0.1502
STEAM TEMP	= 105.6 DEG C	DEV= 0.2012
COND'T TEMP	= 104.3 DEG C	DEV= 0.2204
REFLUX TEMP	= 50.7 DEG C	DEV= 0.1471
FEED TEMP	= 35.0 DEG C	DEV= 0.1010
BOTTOMS TEMP	= 45.6 DEG C	DEV= 0.1070
REB'R TEMP	= 92.1 DEG C	DEV= 0.1699
FEED INLET	= 71.5 DEG C	DEV= 0.1625
REFLUX INLET	= 62.7 DEG C	DEV= 0.4650
COL O'HEAD	= 62.3 DEG C	DEV= 0.1242
WATER INLET	= 18.2 DEG C	DEV= 0.0873
WATER OUTLET	= 33.6 DEG C	DEV= 0.1622

STEADY STATE DATA
 RUN NO 0107E
 05/06/76

FEED FLOW	16.99G/SEC	BOTTOM PROD	8.35G/SEC
REFLUX FLOW	10.79G/SEC	TOP PROD	8.82G/SEC
STEAM FLOWW	14.97G/SEC	COOL WATER	282.43G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEOH
DIST COMP	94.67WT% MEOH	BOTTOMS COMP	0.10WT% MEOH
FEED INLET	71.6DEG C	REFLUX INLET	62.9DEG C
STEAM TEMP	109.3DEG C	PRESSURE	-30.2KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.99	50.00	8.49	8.49
BOTTOM PRODUCT	8.35	0.10	0.00	8.34
TOP PRODUCT	8.82	94.67	8.35	0.47
CLOSURE ERROR-PC	1.0		-1.5	3.7

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	21.18	43.95
REFLUX	2.06	1.99
TOP PRODUCT		1.63
FEED	4.42	
STEAM	40.41	6.87
BOTTOM PRODUCT		3.40
TOTAL	68.08	57.85
HEAT LOSS		10.2

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 01107E 05/06/76

FEED FLOW	= 16.991 G/SEC	DEV= 0.0566
REFLUX FLOW	= 10.793 G/SEC	DEV= 0.1696
STEAM FLOWW	= 14.975 G/SEC	DEV= 0.2333
BOTTOM PROD	= 8.351 G/SEC	DEV= 0.0692
TOP PROD	= 8.824 G/SEC	DEV= 0.1163
COOL WATER	= 282.433 G/SEC	DEV= 12.8215
DIST COMP	= 94.672 WT% MEOH	DEV= 0.1492
BOTTOMS COMP	= 0.100 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -30.249 KPA	DEV= 0.5832
COND LEVEL	= 17.139 CM	DEV= 0.0497
REB'R LEVEL	= 34.797 CM	DEV= 0.3009
DIFF PRESS	= -7.203 KPA	DEV= 0.1133
REB'R O'HEAD	= 96.7 DEG C	DEV= 0.1641
PLATE 1 TEMP	= 94.6 DEG C	DEV= 0.1709
PLATE 2 TEMP	= 90.3 DEG C	DEV= 0.2493
PLATE 3 TEMP	= 119.7 DEG C	DEV= 0.1588
PLATE 4 TEMP	= 78.3 DEG C	DEV= 0.1772
PLATE 5 TEMP	= 73.0 DEG C	DEV= 0.1375
PLATE 6 TEMP	= 68.9 DEG C	DEV= 0.1315
PLATE 7 TEMP	= 66.2 DEG C	DEV= 0.1428
PLATE 8 TEMP	= 63.8 DEG C	DEV= 0.1087
COND TEMP	= 61.3 DEG C	DEV= 0.1027
STEAM TEMP	= 109.3 DEG C	DEV= 0.2055
COND'T TEMP	= 108.6 DEG C	DEV= 0.2279
REFLUX TEMP	= 51.1 DEG C	DEV= 0.1225
FEED TEMP	= 35.4 DEG C	DEV= 0.1009
BOTTOMS TEMP	= 44.8 DEG C	DEV= 0.1058
REB'R TEMP	= 96.6 DEG C	DEV= 0.1784
FEED INLET	= 71.6 DEG C	DEV= 0.1419
REFLUX INLET	= 62.9 DEG C	DEV= 0.3479
COL O'HEAD	= 64.3 DEG C	DEV= 0.1384
WATER INLET	= 17.9 DEG C	DEV= 0.1002
WATER OUTLET	= 37.2 DEG C	DEV= 0.2129

STEADY STATE DATA
 RUN NO 0L108B
 06/05/76

FEED FLOW	16.95G/SEC	BOTTOM PROD	9.12G/SEC
REFLUX FLOW	13.49G/SEC	TOP PROD	8.09G/SEC
STEAM FLOWW	15.04G/SEC	COOL WATER	405.47G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEQH
DIST COMP	96.51WT% MEQH	BOTTOMS COMP	4.50WT% MEQH
FEED INLET	71.4DEG C	REFLUX INLET	62.8DEG C
STEAM TEMP	105.6DEG C	PRESSURE	-30.2KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.95	50.00	8.47	8.47
BOTTOM PRODUCT	9.12	4.50	0.41	8.71
TOP PRODUCT	8.09	96.51	7.80	0.28
CLOSURE ERROR-PC	1.5		-3.0	6.1

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	30.94	56.84
REFLUX	2.55	2.44
TOP PRODUCT		1.46
FEED	4.40	
STEAM	40.50	6.62
BOTTOM PRODUCT		3.50
TOTAL	78.41	70.87
HEAT LOSS		7.5

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 0L1088 06/05/76

FEED FLOW	= 16.951 G/SEC	DEV= 0.0831
REFLUX FLOW	= 13.492 G/SEC	DEV= 0.2041
STEAM FLOW	= 15.042 G/SEC	DEV= 0.2244
BOTTOM PROD	= 9.124 G/SEC	DEV= 0.0723
TOP PROD	= 8.091 G/SEC	DEV= 0.0968
COOL WATER	= 405.472 G/SEC	DEV= 11.6224
DIST COMP	= 96.517 WT% MEOH	DEV= 0.1345
BOTTOMS COMP	= 4.500 WT% MEOH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEOH	DEV= 0.0000
PRESSURE	= -30.264 KPA	DEV= 0.4101
COND LEVEL	= 17.230 CM	DEV= 0.0583
REB'R LEVEL	= 37.735 CM	DEV= 0.2907
DIFF PRESS	= -6.127 KPA	DEV= 0.0974
REB'R O'HEAD	= 92.1 DEG C	DEV= 0.1685
PLATE 1 TEMP	= 81.3 DEG C	DEV= 0.1681
PLATE 2 TEMP	= 75.5 DEG C	DEV= 0.1332
PLATE 3 TEMP	= 121.2 DEG C	DEV= 0.1682
PLATE 4 TEMP	= 71.9 DEG C	DEV= 0.1396
PLATE 5 TEMP	= 67.3 DEG C	DEV= 0.1473
PLATE 6 TEMP	= 64.7 DEG C	DEV= 0.1240
PLATE 7 TEMP	= 63.3 DEG C	DEV= 0.1450
PLATE 8 TEMP	= 62.1 DEG C	DEV= 0.1074
COND TEMP	= 60.6 DEG C	DEV= 0.1451
STEAM TEMP	= 105.6 DEG C	DEV= 0.2384
COND'T TEMP	= 104.3 DEG C	DEV= 0.2010
REFLUX TEMP	= 50.8 DEG C	DEV= 0.1592
FEED TEMP	= 35.0 DEG C	DEV= 0.1160
BOTTOMS TEMP	= 45.7 DEG C	DEV= 0.1241
REB'R TEMP	= 92.1 DEG C	DEV= 0.1732
FEED INLET	= 71.4 DEG C	DEV= 0.1919
REFLUX INLET	= 62.8 DEG C	DEV= 0.4666
COL O'HEAD	= 62.3 DEG C	DEV= 0.1389
WATER INLET	= 18.2 DEG C	DEV= 0.0917
WATER OUTLET	= 33.5 DEG C	DEV= 0.1571

STEADY STATE DATA
 RUN NO 0L108E
 06/05/76

FEED FLOW	16.99G/SEC	BOTTOM PROD	8.43G/SEC
REFLUX FLOW	13.53G/SEC	TOP PROD	8.70G/SEC
STEAM FLOWW	16.49G/SEC	COOL WATER	513.54G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEON
DIST COMP	95.66WT% MEON	BOTTOMS COMP	0.30WT% MEON
FEED INLET	72.3DEG C	REFLUX INLET	63.5DEG C
STEAM TEMP	110.9DEG C	PRESSURE	-30.2KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.99	50.00	8.49	8.49
BOTTOM PRODUCT	8.43	0.30	0.02	8.40
TOP PRODUCT	8.70	95.66	8.33	0.37
CLOSURE ERROR-PC	0.8		-1.6	3.3

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	41.28	69.22
REFLUX	2.60	2.50
TOP PRODUCT		1.61
FEED	4.48	
STEAM	44.56	7.67
BOTTOM PRODUCT		3.45
TOTAL	92.93	84.47
HEAT LOSS		8.4

STEADY STATE CONDITIONS BASED ON 50 POINTS
 RUN NO 01108E 06/05/76

FEED FLOW	= 16.997 G/SEC	DEV= 0.0451
REFLUX FLOW	= 13.536 G/SEC	DEV= 0.1656
STEAM FLOWW	= 16.496 G/SEC	DEV= 0.2495
BOTTOM PROD	= 8.435 G/SEC	DEV= 0.0600
TOP PROD	= 8.709 G/SEC	DEV= 0.1090
COOL WATER	= 513.544 G/SEC	DEV= 9.7596
DIST COMP	= 95.664 WT% MECH	DEV= 0.1545
BOTTOMS COMP	= 0.300 WT% MECH	DEV= 0.0000
FEED COMP	= 50.000 WT% MECH	DEV= 0.0000
PRESSURE	= -30.280 KPA	DEV= 0.3768
COND LEVEL	= 17.169 CM	DEV= 0.0472
REB'R LEVEL	= 34.881 CM	DEV= 0.3674
DIFF PRESS	= -5.969 KPA	DEV= 0.1001
REB'R O'HEAD	= 97.3 DEG C	DEV= 0.1646
PLATE 1 TEMP	= 94.4 DEG C	DEV= 0.1684
PLATE 2 TEMP	= 88.6 DEG C	DEV= 0.2272
PLATE 3 TEMP	= 123.8 DEG C	DEV= 0.2272
PLATE 4 TEMP	= 77.2 DEG C	DEV= 0.1816
PLATE 5 TEMP	= 71.3 DEG C	DEV= 0.1198
PLATE 6 TEMP	= 67.6 DEG C	DEV= 0.1389
PLATE 7 TEMP	= 65.4 DEG C	DEV= 0.1458
PLATE 8 TEMP	= 63.6 DEG C	DEV= 0.1333
COND TEMP	= 61.5 DEG C	DEV= 0.1199
STEAM TEMP	= 110.9 DEG C	DEV= 0.2292
COND'T TEMP	= 110.0 DEG C	DEV= 0.1935
REFLUX TEMP	= 52.0 DEG C	DEV= 0.1458
FEED TEMP	= 36.1 DEG C	DEV= 0.1039
BOTTOMS TEMP	= 46.0 DEG C	DEV= 0.1300
REB'R TEMP	= 97.2 DEG C	DEV= 0.1692
FEED INLET	= 72.3 DEG C	DEV= 0.1532
REFLUX INLET	= 63.5 DEG C	DEV= 0.5608
COL O'HEAD	= 64.0 DEG C	DEV= 0.1285
WATER INLET	= 19.2 DEG C	DEV= 0.0874
WATER OUTLET	= 32.2 DEG C	DEV= 0.1602

STEADY STATE DATA
 RUN NO 0L109B
 06/05/76

FEED FLOW	16.96G/SEC	BOTTOM PROD	9.07G/SEC
REFLUX FLOW	13.51G/SEC	TOP PROD	8.13G/SEC
STEAM FLOW	14.96G/SEC	COOL WATER	529.49G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEQH
DIST COMP	96.56WT% MEQH	BOTTOMS COMP	4.60WT% MEQH
FEED INLET	72.8DEG C	REFLUX INLET	64.0DEG C
STEAM TEMP	106.7DEG C	PRESSURE	-30.2KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.96	50.00	8.48	8.48
BOTTOM PRODUCT	9.07	4.60	0.41	8.66
TOP PRODUCT	8.13	96.56	7.85	0.27
CLOSURE ERROR-PC	1.4		-2.4	5.4

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	41.02	68.36
REFLUX	2.61	2.49
TOP PRODUCT		1.50
FEED	4.50	
STEAM	40.31	6.65
BOTTOM PRODUCT		3.52
TOTAL	88.46	82.54
HEAT LOSS		5.9

STEADY STATE COND. BASED ON 50 POINTS
 RUN NO. 06/05/76

FEED FLOW	= 16.963 G/SEC	DEV= 0.0561
REFLUX FLOW	= 13.513 G/SEC	DEV= 0.1970
STEAM FLOW	= 14.961 G/SEC	DEV= 0.2033
BOTTOM PROD	= 9.079 G/SEC	DEV= 0.0513
TOP PROD	= 8.134 G/SEC	DEV= 0.0933
COOL WATER	= 529.492 G/SEC	DEV= 1.5044
DIST COMP	= 96.566 WT% MEQH	DEV= 0.1578
BOTTOMS COMP	= 4.600 WT% MEQH	DEV= 0.0000
FEED COMP	= 50.000 WT% MEQH	DEV= 0.0000
PRESSURE	= -30.266 KPA	DEV= 0.4615
COND LEVEL	= 17.287 CM	DEV= 0.0525
REB'R LEVEL	= 37.295 CM	DEV= 0.2577
DIFF PRESS	= -6.082 KPA	DEV= 0.0948
REB'R O'HEAD	= 93.1 DEG C	DEV= 0.1704
PLATE 1 TEMP	= 82.2 DEG C	DEV= 0.1647
PLATE 2 TEMP	= 76.5 DEG C	DEV= 0.1535
PLATE 3 TEMP	= 126.0 DEG C	DEV= 0.1669
PLATE 4 TEMP	= 73.0 DEG C	DEV= 0.1379
PLATE 5 TEMP	= 68.4 DEG C	DEV= 0.1427
PLATE 6 TEMP	= 65.9 DEG C	DEV= 0.1196
PLATE 7 TEMP	= 64.5 DEG C	DEV= 0.1340
PLATE 8 TEMP	= 63.3 DEG C	DEV= 0.1331
COND. TEMP	= 61.6 DEG C	DEV= 0.1390
STEAM TEMP	= 106.7 DEG C	DEV= 0.2126
COND'T TEMP	= 105.3 DEG C	DEV= 0.1868
REFLUX TEMP	= 51.7 DEG C	DEV= 0.1463
FEED TEMP	= 36.7 DEG C	DEV= 0.1178
BOTTOMS TEMP	= 46.6 DEG C	DEV= 0.1001
REB'R TEMP	= 93.1 DEG C	DEV= 0.1575
FEED INLET	= 72.8 DEG C	DEV= 0.1547
REFLUX INLET	= 64.0 DEG C	DEV= 0.3542
COL O'HEAD	= 63.6 DEG C	DEV= 0.1142
WATER INLET	= 18.5 DEG C	DEV= 0.0859
WATER OUTLET	= 30.8 DEG C	DEV= 0.1308

STEADY STATE DATA
 RUN NO 0L109E
 06/05/76

FEED FLOW	16.95G/SEC	BOTTOM PROD	10.30G/SEC
REFLUX FLOW	13.47G/SEC	TOP PROD	6.91G/SEC
STEAM FLOWW	13.52G/SEC	COOL WATER	530.00G/SEC
FEED PLATE	4	FEED COMP	50.00WT% MEQH
DIST COMP	96.88WT% MEQH	BOTTOMS COMP	15.00WT% MEQH
FEED INLET	73.0DEG C	REFLUX INLET	64.2DEG C
STEAM TEMP	101.0DEG C	PRESSURE	-30.2KPA

M A T E R I A L B A L A N C E

	FLOW (G/SEC)	COMP (WT PCT)	METHANOL (G/SEC)	WATER (G/SEC)
FEED	16.95	50.00	8.47	8.47
BOTTOM PRODUCT	10.30	15.00	1.54	8.75
TOP PRODUCT	6.91	96.88	6.69	0.21
CLOSURE ERROR-PC	1.5		-2.7	5.8

E N E R G Y B A L A N C E

	ENTHALPY IN (KJ/SEC)	ENTHALPY OUT (KJ/SEC)
COOLING WATER	40.70	67.80
REFLUX	2.61	2.48
TOP PRODUCT		1.27
FEED	4.51	
STEAM	36.30	5.63
BOTTOM PRODUCT		3.64
TOTAL	84.14	80.85
HEAT LOSS		3.2