DYNAMIC AND STEADY STATE BEHAVIOUR OF AN EXTRACTIVE DISTILLATION COLUMN

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by

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Abstract

This project was the second part of a long-term study into the control of an extractive distillation unit. A fifteen stage extractive distillation column was re-built and operated over a range of operating levels. Overhead product acetone composition was limited to approximately 0.94 mole fraction acetone due to tray liquid priming. A steady state simulation was developed to aid in a better understanding of the process. Murphree vapour tray efficiencies were adjusted within the simulation to match experimental and simulated product composition results. The simulation was used to accurately predict unfitted experimental results. Dynamic behaviour of the column was studied by relating product composition responses to changes in disturbance and manipulated variables. Dynamic behaviour was analysed by frequency response and real time response fitting. Experimental data was relatively noisy. First order transfer function models with dead time, fitted in the real time domain, were satisfactory.

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To Venera

CHAPTER 1

INTRODUCTION

The history of computer control in chemical process industries stretches through the past decade. Applications were first undertaken in the late 1950s with digital computers using supervisory control enhancing the operation of conventional analog controllers. Computer applications continued to grow and a wide range of computer hardware became available with the introduction of mini computers in the late 1960s. This new technology gave greater user flexibility and a continued reduction in hardware cost but computer control did not gain a clear advantage over conventional analog methods.

At present the question still remains; will computers take over the control field? Smith (42) comments:

> "The fact is that while we have seen great advances in the computer itself, we have seen precious little advancement in what we know about how to utilize its capabilities effectively. It is the author's opinion that when substantial progress is made in this latter area, then the computer will "sweep the market"."

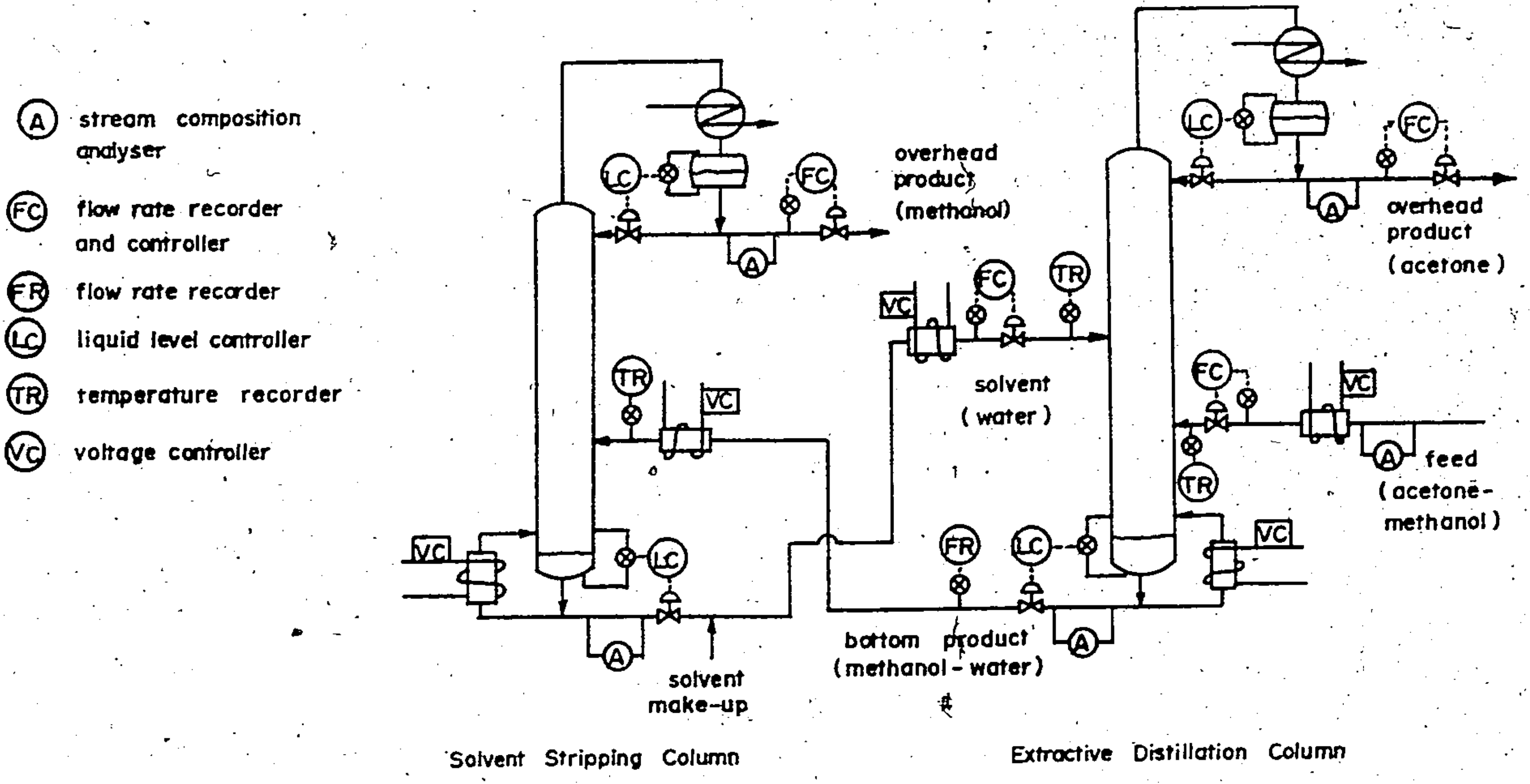
This project represents the second stage in a long-term computer control study of a complex chemical process. The general aim of this work is a realistic control application in which the advantages and disadvantages of computer control may be evaluated. With this experience, more effective control methods may be developed.

1.1 Process Description

The process chosen for this project was extractive distillation. A mixture of methanol and acetone was separated using water as a solvent. Figure 1-1 presents a proposed flow and instrumentation layout for the process. An acetone-methanol mixture is fed to the extractive distillation column. An acetone-methanol mixture forms an azeotrope of 0.80 mole fraction acetone at atmospheric pressure. In order to break this azeotrope, water enters the column above the feed point. Water increases the azeotropic acetone concentration and at approximately 0.30 mole fraction water the azeotrope is completely broken. Jackson (22) presents a detailed description of the vapour-liquid equilibrium relationships in extractive distillation.

A distillation column is an efficient vapour-liquid contacting device which separates components of different volatilities. In the present application, the extractive distillation column is operated to obtain an overhead product of acetone and a bottom product of methanol-water. Vapour, generated by an electrically heated reboiler, flows upward through the column trays. A water cooled condenser condenses the overhead vapour. A fraction of the liquid is drawn off as overhead product and the remainder returns to the column.

FIGURE I-I PROPOSED FLOW AND INSTRUMENTATION LAYOUT FOR THE EXTRACTIVE DISTILLATION UNIT



The bottom methanol-water mixture is fed to the second solvent stripping column. Methanol, the more volatile component, forms the overhead product and the bottom product, water, is returned with solvent make-up to the extractive distillation column. The present work focuses on the extractive distillation column. The solvent stripping column will be designed, constructed and studied in future work.

Figure 1-1 indicates the basic instrumentation required for the control of the extractive distillation unit. Major equipment (including pressure transducers for flow and liquid level indication, control valves, voltage regulators and stream composition analyzers) requires a substantial financial investment and in this respect present work was limited. These automatic instruments were not available and manual product stream composition analysis, flow rate measurement and control were substituted. Details of the flow and instrumentation scheme used in the present work are presented later. However, the long term objectives of the control work require full instrumentation of the extractive distillation column as depicted in Figure 1-1.

1.2 Distillation Importance and Control Difficulties

Distillation is a relatively common but important process in the chemical industries. Distillation is required to separate process products, often at the terminal stage of refinement in the process. The product must meet its specifications because it is most valuable at this point. To meet specifications in the face of large uncontrollable disturbances, product purity is regulated above the required level. Product purity overspecification reduces maximum feed throughput of the unit and increases energy requirements. Production of the entire process may be limited by the maximum distillation feed throughput. Energy requirements including cooling water and steam can be large and costly. Processes, in which component separation is difficult or high product purity is specified, require high reflux ratios. Significant energy savings may be obtained with tighter product purity control in these cases.

Yet distillation control is difficult. Columns are subject to frequent disturbances of widely different magnitudes. Some of these, including feed rate and composition, can be measured but others, including reboiler heat input and column heat losses, can not. Slow column response adds to control difficulties. Unmeasured disturbances enter the system and affect product purity. A controller detects this change and manipulates a variable to offset it. However, a column with a large number of trays responds slowly to corrections and the product is off specification during this period. Compounding this difficulty is the interacting nature of column variables. Distillation is typical, in this respect, of many multivariate systems in the chemical industry. Changing one system variable may affect many variables to differing degrees. In distillation, interaction of controlled variables as functions of manipulated variable changes are important.

1.3 Review of Previous Work and Summary of Project Objectives

The general long-term objective of this project is to evaluate the different control methods presently applied to distillation and with this experience develop more effective control methods. However, more specific work is required to obtain this overall objective.

Jackson's work (22) was an initial step in the long-term study of an extractive distillation unit. His work included building an extractive distillation column, evaluating its steady state performance, interfacing basic column instrumentation to a control computer and developing a basic real-time software package. A thirteen tray extractive distillation column was built with 5.08 cm diameter stripping and absorbing sections and 3.81 cm diameter solvent recovery section. The column was interfaced to the control computer. This included column and feed stream temperatures, feed stream flow measurements and relays operating process status lights and on/off feed temperature control. A real-time software package was developed to monitor manual column operation and perform on/off control to maintain feed stream temperature set points. Steady state experiments indicated that the small diameter solvent recovery section was severely limiting column operation. Vapour velocity increased in the solvent recovery section, causing tray liquid priming at moderate reboiler heat input. The bottom tray also weeped at these conditions and was caused by improper sealing between tray and column wall. Jackson recommended complete rebuilding of the extractive distillation column due to these major operating problems.

Therefore, an initial objective of this project was to rebuild the extractive distillation column and prevent excessive tray priming and weeping. Steady state performance and operating range of the column had to be evaluated. This was to include development of a steady state simulation that accurately described experimental results. Finally, a preliminary understanding of the dynamic behaviour of the column had to be undertaken. This required product composition responses to disturbance and manipulated variable changes. From this work, a process model had to be estimated in order that future control work could be conducted.

Control work could not be undertaken because automatic product composition analysers and controller instrumentation were not available. However, future work will include installation and development of this equipment. Then different methods of distillation control for the extractive distillation column may be evaluated. A description of these control methods is presented in the next chapter.

In summary the objectives of this work were:

(i) to rebuild the extractive distillation column and evaluate its operating range and steady state performance,

(ii) develop a steady state simulation that accurately describes the experimental results, and

(iii) undertake preliminary work to understand the dynamic behaviour of the column.

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CHAPTER 2

REVIEW OF DISTILLATION CONTROL

This chapter presents an overall review of distillation control. A large amount of work during the past twenty years has concerned distillation control and only a brief outline will be given. Williams (47) organized the literature by classifying distillation control schemes according to their control theory sophistication. These general control methods and their distillation control applications are presented.

2.1 Distillation Control Difficulties

Distillation and other chemical processes, including heat exchangers and reactors, have characteristics that make control difficult. These include, (i) non-linear relationship between variables, (ii) multiple interactions between process variables, (iii) poor or non-existant process measurements and (iv) large number of variables and possible control loops.

In distillation, the relationship between feed rate and overhead and bottom product compositions is non-linear and becomes more pronounced with higher product purities. A process model must consider these types of non-linear relationships for tight product composition control. However, deriving accurate process models is difficult for large, complex systems.

A second difficulty, multiple interactions, occur between many distillation variables. Interactions between overhead and bottom compositions is a major control difficulty. Frequently, overhead product rate controls overhead composition. However, due to interactions, bottom composition is also affected and must be controlled by a second manipulated variable to offset overhead product rate changes.

Quick, accurate process measurements improve control loop performance. But, good measurements may not be available in distillation. Composition measurements have large time lags associated with stream sampling and analysis. In some cases, compositions can not be measured and poorer measurements, such as temperature or pressure in multicomponent distillation, must be used.

Designing loops with good control performance is difficult because there is usually a large number of control and measurement variables. Overhead composition in distillation can be controlled by overhead product rate, column reflux rate or reboiler heat input. For good column control, choosing between these three possible manipulated variables may be difficult. From general experience, overhead product rate adjustment gives better control, but this is not a hard and fast rule. The dynamic behaviour of each distillation unit is unique and it is this characteristic that determines control loop design and performance.

2.2 Distillation Control Objectives

A wide range of distillation column control methods are available. These range from simple feedback control to non-linear time optimal control. Control model choice in industry is determined by plant safety and economic return. The control system must give stable operation within safe column operating limits and exclude open loop instabilities due to instrument failure. Economic return is a second major consideration. Product value and column fixed and operating costs are factors in the choice of a control model. In one extreme, automatic feedback control or even manual operator corrections have economical advantages because product value and operating costs are small. However, in the case of a propane-propylene fractionator, where the overhead concentration of propylene product must be 99 percent, large economic losses are encountered with product composition off specification. In the latter case, more sophisticated control schemes are necessary. The level of sophistication is dependent upon the rate of economic return, with manpower and equipment required to implement the control scheme taken into account.

The levels of sophistication of distillation column control, as classified by Williams (47), are given in Table 2-1. Reference control methods within these levels are presented

TABLE 2-1

Levels of Sophistication of Distillation Column Control

Level 5 Modal control and time optimal control (1, 12, 21)
Level 4 Adaptation of parameters
Level 3 Multivariable non-interactive control (8, 11, 25, 28, 32, 46)
Level 2 Dynamic predictive and utility optimization control (26, 27, 29, 31, 34, 38, 39, 40, 49)
Level 1 Basic dynamic control (6, 30, 41, 48)

there. Williams gives an extensive bibliography which includes 104 references. These are not included in Table 2-1. Control methods from each of these levels will be described along with their applications to distillation. Historically, simpler methods in Level 1 were initially used and more sophisticated levels developed. At present, Levels 1, 2 and 3 are generally used in industry. Levels 4 and 5 are being developed from more of a theoretical standpoint and initial applications are being attempted.

2.3 Distillation Control Methods

Level 1, basic dynamic control, is based on single input - single output linear design theory. Applications in distillation include feedback control of the more important product stream composition. Either overhead or bottom product composition is controlled by adjusting overhead product rate or reboiler heat input. Product composition may be measured indirectly (but quickly) by using temperature or column pressure. These measurements can be related directly to composition for a binary system with components of significantly different boiling points. But, composition must be measured directly in a multicomponent system, unless the key component of interest has a large effect upon temperature relative to the other components. Faster control loop responses are obtained by placing the composition sensor several trays from the controlled end of the column. There is an optimum sensor location for each particular distillation unit.

Controller settings are selected by standard design techniques when a process model is available. Ziegler and Nichols, Cohen and Coon, Lopez and Smith and Murrill have presented recommended controller settings based upon different performance criterion. These criteria at best provide initial estimates for controller settings which inevitably must be tuned on-line in real processes. Experience and on-line tuning are used if there is no process model. In Level 1 control, models describing the process are usually "black-box" type rather than a physical principles type model. The latter type uses basic engineering principles, including mass and energy balances and equilibrium relationships, to fully describe the distillation process. Models in this form have a large number of variables and are non-linear, but describe the system accurately. "Black-box" transfer function models consist of simple dynamic relationships between single inputs and outputs. These relationships are determined from process experiment or an accurate deterministic model of the unit.

A simple transfer function is obtained by fitting output data resulting from a pre-specified input. This may be done in the frequency (20) or time domain (35). These methods are described in detail in a latter section of this chapter. Transfer functions are low order approximations of the column response to variable changes. First and second order systems with dead time are usually fitted (2, 23, 49). Transfer functions describe the system in continuous time and are useful with analog controllers. However, with wider use of direct digital control, a discrete time, Z-transform approach has been used. In one method, the Z-transform is calculated from the S-transform (41). A second method identifies the transfer function model form via the Box and Jenkins approach (5) and the unknown parameters are least squares fitted (37). Controller settings are designed from either continuous or discrete time transfer functions.

Feedback control gives good results for columns where one product composition is of primary importance. Feedback control loops for both product compositions may be installed if interaction between overhead and bottom compositions is small. The degree of control loop interaction is indicated by the relative gain matrix (39) and resulting interaction index. It is defined as the ratio of interaction-induced variable change to the controlled variable change. This approach has been successfully applied by Nisenfeld and Stravinski (34) to predict control loop interaction and to design control loop variable pairings with the smallest interaction. However, interaction is still present and cannot be eliminated with this level of control theory. A second major problem is slow response in large columns under feedback control. Column feed upsets cannot be detected quickly in columns with large holdups. In these cases, dynamic predictive control has improved column control performance.

Level 2 control includes dynamic predictive and utility optimization control. Dynamic predictive control is used when disturbances entering the system can be measured and acted upon before system outputs are affected. In distillation, commonly measured disturbances include feed rate and composition. Feed-forward control can be implemented to offset these measured disturbances when a process model, relating the disturbance and controlled variables, is known. The process model may be in transfer function form or derived from energy and mass balance relationships with dynamic compensation.

In a transfer function approach, the process disturbance model relating feed rate and composition to product compositions, as well as the process manipulated model relating manipulated variables to product compositions, are experimentally determined. First or second order plus dead time transfer functions are usually adequate. The control model is the ratio of the process disturbance model to the process manipulated model for each disturbance-controlled variable pair.

Mass and energy balance control has been widely used in industry by Shinsky (39) and others (33, 34). This method is a simple but effective approach to feedforward control in distillation. Normally, overhead product rate and boil-up rate are the manipulated variables but, in special distillation cases, other variables can be selected. Overhead rate is defined in terms of control set points (overhead and bottom compositions) and disturbance variables (feed rate and compositions) using steady state total material and component mass balance relationships about the column. This mass balance equation is in a simple algebraic form. The second manipulated variable, boil-up rate, is adjusted to maintain the separation factor for the set point product compositions. Usually separation factor is a function of boil-up rate to feed rate ratio. Therefore, to maintain a constant separation factor, the boil-up rate to feed rate ratio is held constant by adjusting boil-up rate in proportion to feed rate disturbances. This is the form in which the energy balance equations are applied. These relationships between manipulated, disturbance and controlled variables are reasonably accurate at steady state conditions. However, during a response from one level to another, there is a transient error due to different dynamic characteristics between the disturbance-controlled variable and manipulated-controlled variable pairs. Therefore, dynamic compensation is incorporated into the above steady state model to account for these differences. Simple first order lags, adjusted on line, have proved effective.

Distillation control using feedforward control alone is not effective (49). Product compositions tend to drift around set points due to process model inaccuracies and unmeasured disturbance effects. Better control is obtained by a feedforward-feedback method. Feedforward methods, as described above, are combined with Level 1 feedback control. Measured disturbances are offset using predictive control and product composition errors, due to model inaccuracy or unmeasured disturbances, are corrected by feedback control.

Utility optimization control is also included in Level 2 control. Utility costs for condenser cooling water and preheater feed steam are significant in large distillation units. Cooling water flow is minimized by controlling to a desired cooling water temperature increase, as well as outlet condenser product temperature set point. Distillation feed is normally preheated by the bottom product stream to conserve heat energy in industry. A secondary steam preheater controls feed temperature but fluctuations in feed rate and bottom product temperature or rate are difficult to adjust for. By measuring these disturbances, feedforward-feedback control, as described by Williams (47), is used to obtain better feed temperature control.

The third level of control sophistication is multivariable non-interactive control. In this method, control is designed such that a change in set point of one variable influences only that variable. Control loops are non-interacting when the product of the process matrix and control transfer function matrix is a diagonal matrix. Then, controller settings for each loop may be designed separately, using single input-single output design theory or by on-line tuning.

Control problems may occur in distillation when both product compositions are under feedback control due to control loop interactions. Changing vapour boil-up to control bottom composition also affects overhead composition. Likewise, changing overhead product rate to control overhead composition disturbs bottom composition. An interaction index, as previously discussed, can measure these interactions. However, Level 1, basic dynamic control, cannot reduce these interactions.

In multivariate non-interactive control, the product of the process matrix and control matrix is a diagonal matrix. The process matrix describes the single input-single output relationships between the controlled and manipulated variables in transfer function form. These transfer functions are usually first and second order lags with dead time and lead-lag networks (28, 46). The choice of a control matrix has two restrictions. The product of the process matrix and control matrix must form a diagonal matrix and process matrix terms must be physically realizeable. Terms with negative dead time are unrealizeable. Values of the terms in the diagonal matrix are unrestricted, therefore terms in the control matrix can be given a wide range of values. Advantages of using specific forms of the control matrix are discussed by Waller (46). With the control matrix calculated, the process control loops are decoupled. Feedback controller settings are designed using single loop methods or on-line tuning.

Decoupled controllers simplify design of controller settings but there are disadvantages. Non-interactive control is designed to allow set point changes in one control loop not to affect the performance of other loops. However, as Foss (15) comments, chemical processes do not require servo performance but the diminuation of disturbances. (Servo performance is only required during a small fraction of equipment operating time which includes start up and set point changes.) Niederlinski (32) states that interacting control loops gave better disturbance attenuation than non-interacting control in the case that he studied. Therefore, decoupled control is a valuable method in cases where control loop interaction is severe, but of questionable value otherwise.

The fourth level of sophistication includes control with adaptation of parameters. In this method, control system parameters are updated from current operating conditions. This method has proved effective for drifting and non-linear processes including heat exchangers and chemical reactors. One drawback is the expensive digital computer and programming required to implement the updating algorithm. There has been very little work on this type of control in distillation to date.

The highest level of control sophistication includes modal and time optimal control. These two methods determine the control configuration in different ways but both use accurate non-linear models to initially describe the process.

Modal control is designed to increase the rate of response of the natural modes of the system (eigenvector-eigenvalue pairs of the state matrix). To accomplish this, the manipulated variables must be aligned with the modes and the modes of the system measured. Then the time constants (or eigenvalues) can be adjusted, through a proportional feedback control matrix, to decrease the closed loop system response time.

Modal control has been designed for several distillation cases (10, 12, 16). Initially a deterministic process model is developed from the non-linear differential equations describing the column and linearized about the normal operating level. For large systems, the order of the state matrix is reduced but important characteristics of the system (smallest eigenvalues) are retained. Modes of the reduced state matrix are calculated and the manipulated variables aligned as described by Gould (16). Finally, feedback controller constants are adjusted, increasing the value of the smaller eigenvalues and increasing the closed loop response of the distillation column.

In time optimal control, feedback and feedforward gains are evaluated that minimize a quadratic functional of process states and control inputs. A process model is usually determined from differential equations describing the system. Time optimal control has the disadvantage, as with modal control, that all the state variables must be measured or estimated. The estimation may be obtained from several methods including a Luenberger observer or Kalman-Bucy state estimator but Foss (15) gives several disadvantages with these estimations.

Modal and time optimal control of distillation has not been generally applied in industry. This is largely due to the almost impossible requirement that all of the states be either measured or estimated accurately. A second reason is the marginal economic improvement that these control methods will give over the present feedforward-feedback methods in most industrial applications.

2.4 Present Work in Relation to Distillation Control

A natural question now arises, how does the present project relate to the overall distillation control work that has been presented? This project is the second stage of a long term research program on the application of distillation control to a pilot plant column. Initially, basic experimental work must be carried out to obtain this objective. In the first stage, the extractive distillation column was re-built and operated over a wide range of column operating conditions. This work is presented in a later chapter. Basic dynamic behaviour of the column was experimentally determined in the second stage of this project. No control work was implemented due to the lack of instrumentation on the pilot plant column. Automatic composition analysis is required for the overhead and bottom product streams. Instrumentation is also required to adjust overhead product rate and boil-up rate and control product compositions.

The dynamic behaviour of the column was described by simplified transfer functions relating single input-output variable pairs. Disturbance variables (feed rate and composition) and manipulated variables (overhead product rate and boil-up rate) were related to the control variables (overhead and bottom product compositions). This was done in the frequency and time domain.

Pulse testing was conducted to determine the frequency response of the column. The column was operated at steady state conditions, then a rectangular pulse was manually introduced to the input variable. Product samples were taken at regular intervals and analysed. Frequency response was obtained by relating the input and output responses by a fast Fourier transform algorithm. Results were plotted in Bode form (amplitude ratio and phase angle versus frequency). Simple first and second order systems with dead time were fitted from the Bode plot. Low order transfer functions described the low frequency area (2, 23, 49).

Transfer functions were also fitted in the time domain. Column responses were obtained with rectangular pulse and step changes to the input. First and second order transfer functions with dead time were least squares fitted by a non-linear regression routine (Marquardt's compromise). The results for the dynamic behaviour of the column are given in a later chapter.

The above work was a "black-box" approach in describing the distillation column model. A second approach uses material and energy balances to develop a deterministic model. The next chapter describes a steady state simulation of the extractive distillation column. Pilot plant column operation can be better understood from this model and a deterministic model developed that accurately describes the column.

CHAPTER 3

STEADY STATE SIMULATION

Previous work in this project (22) presented a graphical hand calculation to represent stage-to-stage composition changes within the extractive distillation column. Using this calculation, the number of ideal stages is calculated for a given experimentally determined steady state. This is compared to the actual number column stages and the overall column efficiency calculated.

This calculational method has three drawbacks. The graphical hand solution takes about thirty minutes for a complete solution. This is too time consuming for a large number of cases. Secondly, this method requires reboiler product composition to start stage-to-stage calculations up the column. This restriction is satisfactory for calculation of overall column efficiency from experimental column data. But when column efficiency is known, calculation of reboiler product composition can not be determined without a lengthy iterative solution. The third drawback is over-simplifying assumptions. These include ignoring heat balance and assuming equimolar overflow. The effect of variations in feed temperature can not be determined. Equimolar overflow is a poor assumption because vapour flows increase fifteen percent up the column at experimental conditions, due to vapour composition changes.

3.1. Digital Simulation Advantages

By contrast with the manual calculation approach, quick, accurate steady state calculations are possible with a digital simulation. The complex distillation process is better understood with this model and large number of cases are calculated to determine the effect of different process variables on column performance. A second advantage is a better understanding of column dynamics. Process time lags and dead time can only be determined by a ~~dynamic~~ simulation. But steady state models determine process gains between disturbance variables and product composition. Magnitudes of input changes for experimental column testwork were designed using these results. A steady state model has a third advantage. It is a first step to a complete deterministic model. However, accurate tray efficiencies and proven equilibrium relationships are required for a deterministic model. These may be obtained through a steady state simulation.

3.2 Computational Methods for Distillation

There are three general groups of methods used in the analysis of distillation columns. These include (i) group methods, (ii) stage-to-stage calculation methods and (iii) successive approximation methods. Each method is applicable to different types of problems. The following description of these methods is an overview of an excellent presentation by King (24).

Group methods relate number of column stages directly to product compositions. This approach requires algebraic equations to represent the combined effect of all stages on product composition. These equations are developed using simplifying assumptions including linear equilibrium relationships and constant flow rates within the column. An example of this calculational method is the Underwood equations. This type of calculation is unsuitable to the extractive distillation column because the assumptions are unrealistic.

Stage-to-stage methods require fixed conditions at one end of the tower and sequential stage calculation along the column. The McCabe-Thiele diagram is an application for a binary system. A method for three components was presented in a graphical form for the extractive distillation column (22). It consists of alternative uses of equilibrium and material balance relations to go from one stage to the next. This method is attractive in design calculations and experimental work where product compositions and flows are specified, and the required number of stages for the separation is calculated. However, if the number of stages is set and either product compositions or flows are unknown, this calculation requires an iterative solution. This is not practical for a multicomponent system with a large number of stages.

Successive approximation methods are required for problems where the number of stages is fixed and the degree of separation is calculated. They are divided into two groups; relaxation methods and iteration methods.

Relaxation methods follow the transient behaviour of the column as it approaches steady state solution. Initially all stage flows, compositions and temperatures are assumed. The calculational method considers each stage separately. Conditions for each stage (i) are recalculated to obtain enthalpy and component balances. These new conditions change the calculated balances on adjacent stages (i-1) and (i+1). When these stages are in turn rebalanced, stage (i) still is imbalanced and must be recalculated on the next iteration. Relaxation methods are highly stable but converge slowly, especially as the final solution is approached. Therefore, they are used for difficult problems which cannot be solved by other methods.

Iteration methods also assume conditions for all stages. Stage compositions are calculated for the column using mass and equilibrium relationships. Since the original assumed conditions are usually in error, calculated compositions will be wrong. A correction procedure using these compositions determines a more accurate set of conditions for each stage. These will be used in the next iteration. The Thiele-Geddes (43) and θ-convergence methods (19) solve stage compositions by a stage to stage calculation. However, these methods are subject to computer truncation errors and computation difficulties arise with multiple feeds. In the Thomas tridiagonal matrix method, stage compositions are calculated, with adjacent stage interactions taken into account by a series of linear equations in matrix form. This method is four times faster than the Thiele-Geddes method (3). After stage compositions are determined, more accurate stage conditions are estimated by a correction procedure. This may include a multivariate Newton scheme or a bubble-point calculation. The multivariate Newton scheme calculates new stage flows and temperatures from equilibrium and enthalpy balance equations with stage interactions. This procedure requires a large number of calculations. The bubble-point method calculates new conditions for each individual stage. This calculation is much faster but because stage interactions are neglected requires more iterations for convergence. The choice between these two methods is dependent upon the properties of the column to be simulated.

The Thomas tridiagonal matrix method is used to calculate stage compositions in the simulation of the extractive distillation column. Bubble-point and enthalpy balance calculations correct stage flows and temperatures. These new stage conditions are used in the next iteration.

3.3 Extractive Distillation Column Simulation

The overall convergence scheme for the steady state simulation is given in Figure 3-1. A general description is given here. A more detailed description, including program listings and flowcharts, is given in Appendix B.

Initially, column operating conditions and parameters must be defined. These include (i) number of stages, (ii) feed plate locations, (iii) feed compositions, flow rates and temperatures, (iv) pressure, (v) stage efficiencies, (vi) reflux ratio and (vii) either overhead product rate or reboiler duty. No product compositions are specified as might be given in a design problem case.

Stage temperatures, flows and liquid compositions are estimated for the particular column operating conditions. Naturally, better initial estimates will require a fewer number of iterations for convergence. Unlike most distillation systems, initial liquid compositions are required because equilibrium relationships are strongly dependent upon composition rather than temperature.

Mass balance equations are solved for component liquid flows using initial estimates of stage conditions. The Modified Thomas method was used to solve the tridiagonal matrix rather than the Thomas method. The latter produces computer truncation errors when the equilibrium ratio (K = y/x) approaches 1.0 (4).

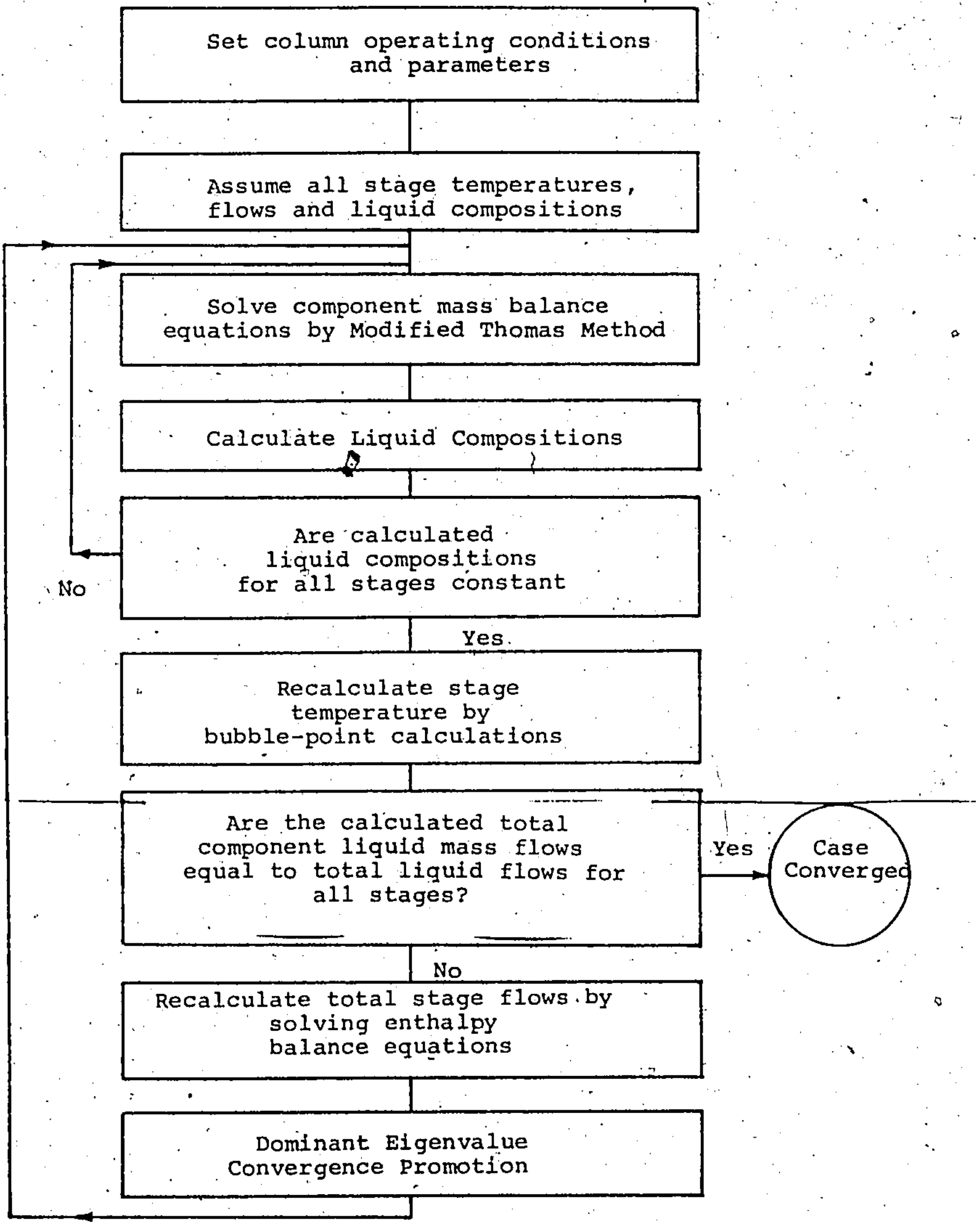


FIGURE 3-1 Convergence Scheme for the Extractive Distillation Column

Equilibrium ratio for methanol ranges from 0.7 to 4.0 for the acetone-methanol-water system.

Initial estimates of stage conditions are wrong, therefore the sum of component liquid flows will not correspond to the desired total liquid flow. However, normalized liquid compositions are determined from the component flows. These compositions are compared to the compositions calculated during the previous pass. If the compositions are different by more than 0.025 per cent, component liquid flows are recalculated. Flows change because the component mass balance equations are a function of equilibrium relationships which are dependent upon temperature and liquid composition.

Stage temperatures are determined by the bubble-point method when calculated compositions are constant. In this calculation with liquid composition given, stage temperature is varied to obtain vapour mole fractions summing to 1.0. Equilibrium data for the acetone-methanol-water system is supplied by the Enthalpic Wilson equation. This method has been shown (7) to give better results than other methods. Vapour compositions as predicted by the Enthalpic Wilson equation were compared to those given experimentally by Griswold (17) over the range of column operating conditions. The average error for acetone, methanol and water for 16 points were 0.0168, 0.0182 and 0.0134 mole fractions, respectively and the largest errors were 0.035, 0.034 and 0.036 mole fractions, respectively. These errors are introduced into the simulation.

More accurate equilibrium relationships will have to be developed if these errors cannot be tolerated.

Next in the simulation, calculated total component liquid flows are compared to set liquid flows for all stages. If flows for any stage differ by more than 0.05 per cent, the iteration procedure continues. Total stream flows are reset by solving enthalpy balance equations by Hanson's (18) Method I when overhead product rate is set or Method II when reboiler duty is set.

After the enthalpy balance calculations, stage temperatures and vapour flows are recalculated by the Dominant Eigenvalue Convergence Promotion routine (36). This algorithm has been included to assist difficult cases to converge. The vast majority of cases require no assistance from this routine.

The next iteration is again started by calculating component liquid flows. Most cases require between five and twelve iterations to converge. This corresponds to ten to twenty seconds of computer CPU time on a CDC 6400. Therefore, a large number of cases are analysed to determine the effect of many different variables.

The first step in the simulation study was to determine model accuracy. Jackson's work (22) presents a graphical solution of a 10 tray column. This problem was solved by the simulation and the results are given in Table 3-1.

TABLE 3-1

COMPARISON OF GRAPHICAL SOLUTION AND SIMULATION

	Overhead Product (mole fraction)			Bottom Product (mole fraction)		
	Acetone	Methanol	Water	Acetone	Methanol	Water
Graphical Solution	0.920	0.017	0.063	0.0035	0.1615	0.8350
Simulation	0.916	0.016	0.068	0.0057	0.1617	0.8326

The graphical solution and simulation results compare very favourably. All column operating conditions and parameters are calculated or estimated accurately in the simulation except for stage efficiencies. Correlations are available but unreliable for small diameter columns. Column stage efficiencies were varied to match experimentally determined product compositions in the second part of the simulation work. Using these efficiencies, column conditions were altered to determine their effect upon column operation. This work is presented later.

A simulation relates a mathematical model, through theory and empirical data, to a real process. This process is a fifteen stage extractive distillation column which is described in the next chapter.

CHAPTER 4

PROCESS EQUIPMENT

The first stage of this work was rebuilding the extractive distillation column as recommended by Jackson (22). This was required due to several process problems.

The previous column's capacity was limited by liquid priming in the solvent recovery section. This section was 3.81 cm in diameter compared to the 5.08 cm diameter absorbing and stripping sections. Vapour velocity increased by 75 per cent due to column diameter change. This created high liquid tray levels in the solvent recovery section.

Tray weeping was another major operating problem. Previous column trays were strung on a steel rod and inserted into a 91.44 cm x 5.08 cm I.D. Q.V.F. glass pipe. Teflon collars were machined to fit the outer edge of the plates and provide a tight fit against the inner glass wall. But due to the semi-fluid characteristics of teflon, plate collars deformed and leakage occurred. Therefore, it was recommended that the column be re-built. Tray weeping around the teflon collars was eliminated by machining 7.62 cm diameter sieve plates and sandwiching them between sections of 15.24 cm x 5.08 cm I.D. Q.V.F. glass pipe.

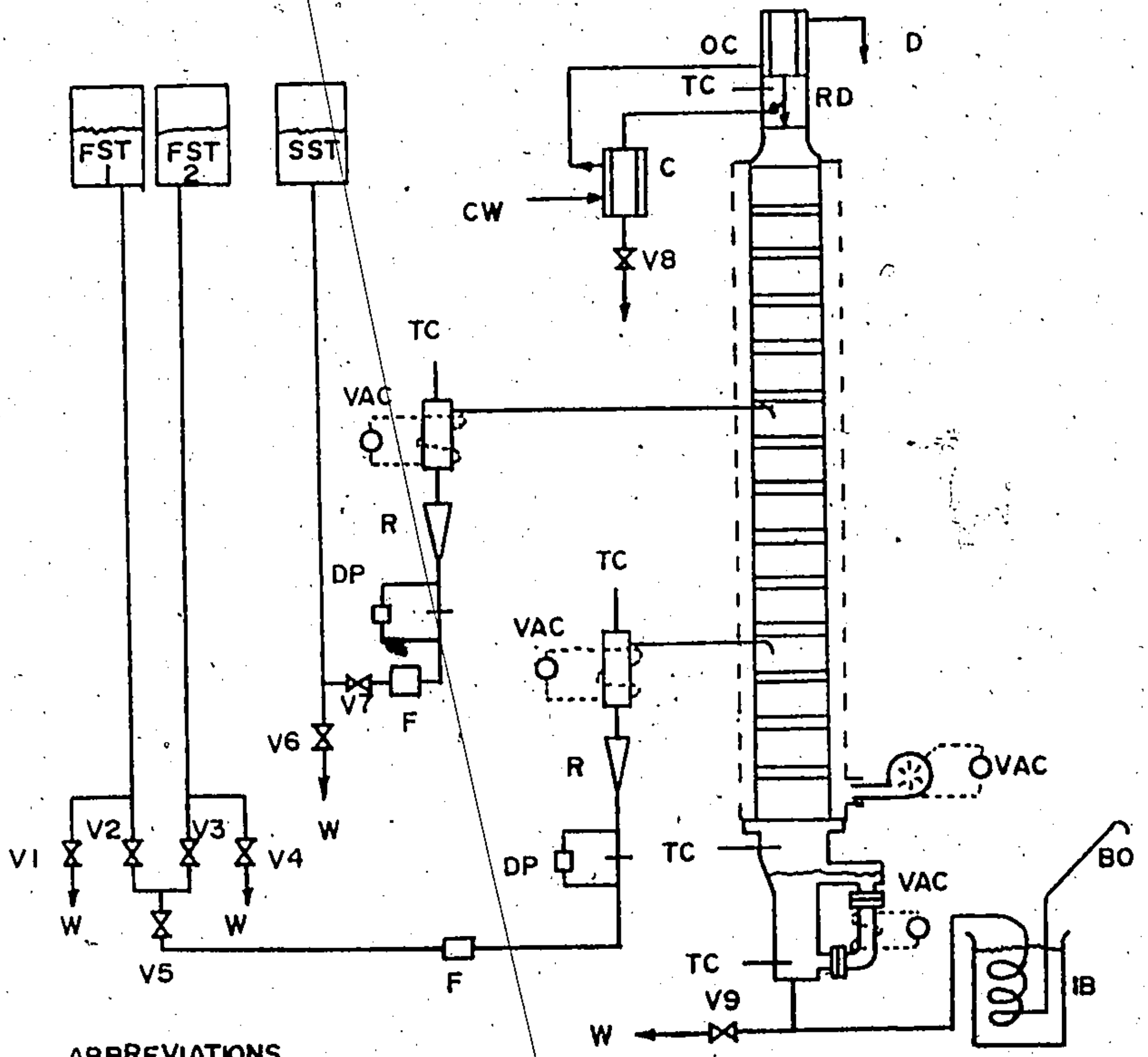
A third problem was associated with the Vibrostaltic feed pumps. These pumps produced flow by high frequency mechanical pinching of 0.96 cm surgical tubing. This tubing would deteriorate with use, producing a gummy residue that accumulated in the feed systems. The Vibrostaltic pumps were also unreliable. Feed flows would change or completely stop and constant operator attention was required for steady flows. The pumps were discarded due to these problems and feed storage tanks were placed above the column for a constant pressure head. This arrangement produced reliable flows over the required operating range.

The above problems required major changes to the extractive distillation column. However, feed and solvent stream instrumentation, reboiler and condenser systems and computer interfacing and software remained unchanged from the work of Jackson. The following is a description of the equipment after final modifications. During the initial stages of this work, sieve tray design was modified to prevent tray weeping. Initial tray design and reasons for changing to the present design are presented in the next chapter.

Figure 4-1 presents the flow diagram for the extractive distillation column. Discussion of process equipment is presented under the following sections:

1. Feed and Solvent Streams
2. Extractive Distillation Column

FIGURE 4-1 FLOW DIAGRAM - EXTRACTIVE DISTILLATION COLUMN



ABBREVIATIONS

BO	bottom overflow
C	cooler
CW	cooling water
D	drain
DP	differential pressure
F	filter
FST	feed storage tank
IB	ice bath
OC	overhead condenser
R	rotameter
RD	reflux divider
SST	solvent storage tank
TC	thermocouple
V	valve
VAC	variable AC power
W	waste

3. Reboiler and Condenser Systems
4. Computer Interfacing and Software
5. Stream Analysis

4.1 Feed and Solvent Streams

Feed and solvent stream layouts are given in Figure 4-1. The 10 liter storage tanks are placed on the laboratory rack above the column. Feed flows from these tanks, through 0.63 cm stainless steel tubing, to the on/off valves (V1, V2, V3, V4). Stainless steel and glass are used in the feed system because acetone disolves tygon and rubber tubing. Feed tanks #1 and #2 are selected by valves V2 and V3 respectively. Feed flow is adjusted by a needle valve (V5). A micron filter is placed inline to prevent foreign material clogging the glass orifice. Differential pressure across the orifice measures flow rate and visual measurements are made with a F & P Co. Precision Flow-rater. The preheater is a 30.5 cm x 1.27 cm O.D. tube, wound with resistance wire. A thermocouple measures outlet stream temperature and a Powerstat transformer varies power input to the preheater. The preheater outlet is connected to the feed port of the tenth tray from the top of the column. Total feed system liquid holdup from valve (V5) to column inlet is 65 ml.

Solvent and feed streams are similar. However, only one solvent storage tank is required and lines to the glass orifice are tygon, rather than stainless steel tubing. The outlet from the preheater is connected to the feed port in the fifth tray.

Feed and solvent streams give reliable flows which have to be manually adjusted only occasionally. This is largely due to removing the Vibrostolic pumps and moving the storage tanks overhead.

4.2 Extractive Distillation Column

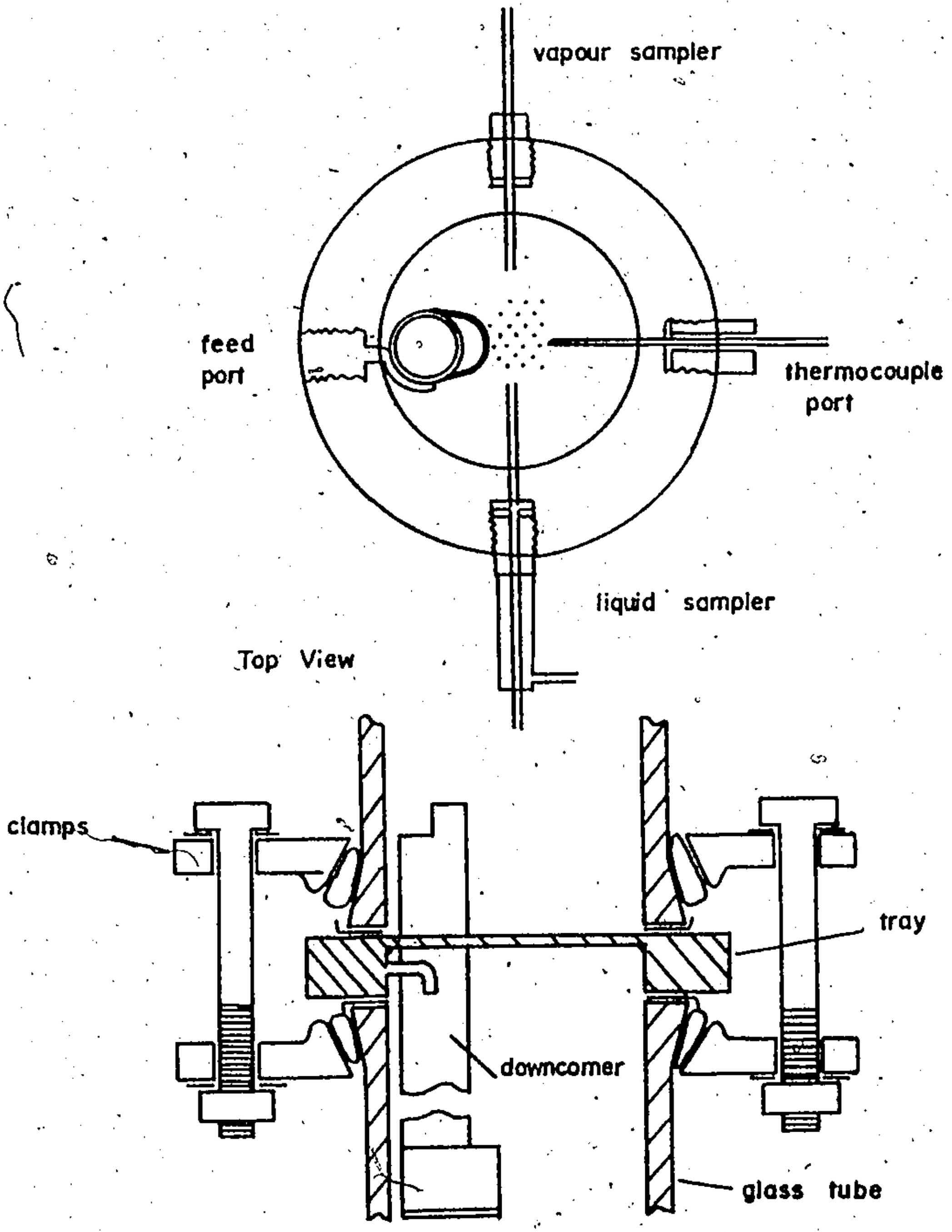
The extractive distillation column consists of thirteen sieve plates. These are separated by 15.24 cm x 5.08 cm I.D. Q.V.F. glass pipe, tapered on both ends. Each pair of glass pipes are clamped together by a set of flanges (Figure 4-2). Plastic rings seal the glass pipe - sieve tray joints.

Liquid overflows the weir and flows down the downcomer in normal column operation. A liquid seal at the bottom prevents vapour by-passing the plate through the downcomer. The 0.16 cm thick sieve plate has 93 holes, 0.089 cm diameter, through which vapour passes and mixes with the liquid.

The plates have been designed in order that operating conditions within the column may be obtained. Temperature of vapour entering the tray is measured by a thermocouple inserted through the sieve plate wall.

Vapour and liquid samples can be taken from each plate. An inlet tray vapour sample may be obtained through a tube that is inserted below the plate. A liquid sample is taken from the plate, through the sieve plate wall and heat exchanger by

FIGURE 4-2 SIEVE PLATE LAYOUT



Side View

scale: $\frac{3}{4}$ actual size

0.32 cm O.D. tubing. The air cooled heat exchanger, 7.62 cm x 0.96 cm O.D. tubing, prevents liquid sample from vapourizing. Each plate has a feed port located near the downcomer. A feed stream may enter this port and flow down the side of the downcomer, onto the tray below. Column feed plate location can be changed to obtain optimum operating conditions.

A hot air jacket, surrounding the column, allows observation of the operating column and insulation from room temperature. The jacket was constructed from two sections of 13.34 cm I.D. Perspex plastic pipe with holes drilled in appropriate locations for stage liquid and vapour samplers, thermocouples and feed parts. Each section was cut lengthwise into two pieces in order that the jacket could be placed around the column. Hot air is directed inside the jacket at the base of the column by a hot air gun with variable temperature control. Temperatures at the bottom and top of the air jacket are 80°C and 55°C, respectively at normal operating conditions. This corresponds to the approximate tray temperatures at these locations.

Vapour is generated by a reboiler and flows upward through the trays to a condenser during column operation. Here the vapour is condensed, part is drawn off as overhead product, and the remainder flows from tray to tray down the column.

4.3 Reboiler and Condenser Systems

Both the reboiler and condenser have been described in detail by Jackson (22). A general description of the equipment and modifications will be given here.

The stainless steel reboiler supports the column and rests on angle-irons clamped to the laboratory frame. It is normally operated with the liquid level slightly below the outlet of the electrically heated sidearm. Liquid holdup is 327 ml. Tests were conducted with reboiler heat output measured over a range of power inputs. These results are given in Appendix A.

Bottom product flow was modified from that described by Jackson. In that arrangement, liquid level was measured by an inclined mercury manometer with contact points for upper and lower level limits. A Vibrostaltic pump turned on and off at these limits. Unfortunately, manometer contact points corroded, giving poor contact and erratic reboiler level control behaviour. Therefore, this liquid level controller was replaced by a simple reboiler overflow arrangement. The bottom product flows by gravity and column pressure through an ice bath and overflows at a set exit tube height. This height is adjusted to obtain the desired reboiler liquid level. The holdup between reboiler and tube exit is 25 ml.

The condenser and reflux divider have been described by Jackson. The condenser is water cooled and totally condenses the overhead vapour. Liquid flows down the condenser, through the reflux divider and onto the top tray of the column. A fraction of the liquid passing through the reflux divider is directed outside the unit by a magnetically activated funnel. This overhead product stream is cooled by a water jacketed graduated cylinder and flows to a waste bottle.

4.4 Computer Interfacing and Software

The extractive distillation column is a relatively complex unit in which a number of variables have to be measured and controlled for steady operation. Jackson interfaced the column to the control computer and developed software for a preliminary monitor/control package as a first step towards direct digital control. This work has been described in detail by Jackson (22) and a general description will be given here.

The 32K Nova 2 digital control computer is connected to the process site by two Belden twisted shielded pair transmission cables. Process thermocouple mV measurements (reboiler, condenser, feed preheater and solvent preheater temperatures) are converted to current signals by Acromag transducers. They are transmitted to the computer where a voltage signal is obtained by placing a resistor in series. Feed and solvent stream flow rates, measured by differential pressure cells across the orifices, are transmitted as voltage signals. These voltage signals are inputed to an analog to digital converter (ADC). A relay box at the process contains eight relay switches. These are used as on/off switches for 115 volts AC supply in the process. They operate five signal lights, two feed preheaters and a reflux divider. Computer relays activate these switches with a six volt supply. Two manual switches at the process are monitored by the control computer through contact switch inputs. When product samples are taken during an experiment, the switches are activated and the control computer notes the time. These times are outputed on the teletype and give a summary of the samples taken.

Computer software was written to measure and log process variables, control feed stream temperatures and monitor feed stream flowrates. Reboiler, condenser, feed stream and solvent stream measurements are obtained at regular intervals, converted to engineering units, and outputed to the teletype. Calibration curves for the conversion from A/D units to engineering units are given in Appendix A. Feed stream temperatures are controlled to the desired set point by on/off control of power supplied to the preheaters. Preheaters are turned off or on when above or below the set points, respectively. Stream flow-rates are monitered, logged and compared to the set points. If outside a given limit,a light is turned on at the process telling the operator that the flow is in error and manual adjustment is necessary.

A parameter change routine is included in the software package. Program parameters which determine program periods, digital to engineering units conversion constants, set points and limits may be checked and changed on-line if necessary.

Logged data is punched onto paper tape and converted to computer cards. From these, data is presented graphically and averaged for an experimental run.

The control computer has made the operation of the extractive distillation column and analysis of the measured variables easier. Unfortunately, automatic analysis of liquid product streams could not be implemented and samples were taken and analysed manually with a gas chromatograph.

4.5 Stream Analysis

During an experimental run, overhead and bottom product stream 5 ml samples are taken every nine minutes. These samples are analysed manually with a Varian 90-P3 gas chromatograph. Porapak Q column packing separates the components and a thermal conductivity detector produces a chromatograph signal. Signal area, corresponding to each component, is determined by a Hewlett-Packard 3370B Integrator. Chromatograph and integrator settings are given by Jackson.

Calibration curves relating peak area to moles of pure component are given in Appendix A. The calibration is linear over the range 0.0 - 1.0 μ-liters. Sample composition is determined by converting component peak area to moles and then to mole fractions. In this calculation sample size is independent. Each sample is analysed twice and an average taken.

An estimate of the reproducability of the gas chromatograph analysis is given in Table 4-1.

These results indicate that the analysis is very reproduceable. Overhead samples are more reproduceable than bottom samples.

This chapter has presented a description of the process equipment associated with the extractive distillation column. In the next chapter, steady state results for the column will be given. This will include equipment modifications, column operating range and comparison of steady state simulation and experimental results.

TABLE 4-1

REPRODUCABILITY OF STREAM COMPOSITION ANALYSIS

	Acetone mole fraction	Methanol mole fraction	Water mole fraction
Overhead Sample average	0.90254	0.06016	0.03730
(10 analyses) standard deviation	0.00040	0.00010	0.00015
Bottom Sample average	0.00476	0.18613	0.80911
(10 analyses) standard deviation	0.00003	0.00110	0.00118

CHAPTER 5

STEADY STATE RESULTS

This chapter presents experimental results relating to the steady state operation of the extractive distillation column. The first section is a short description of tray modifications. These modifications prevented tray weeping at design boil-up rates. Column operating level and limits are presented in the second section. Manipulated and disturbance variable limits, as restricted by tray weeping and priming are given. In the final section the steady state simulation is related to experimental results and the effect of disturbance and manipulated variables are predicted.

5.1 Column Modifications

The first step in this project was rebuilding the extractive distillation column. Tray weeping and an under-sized solvent recovery section made this necessary. A new thirteen tray column was designed with sections of 15.24 cm x 5.08 cm I.D. Q.V.F. glass pipe between each tray. Tray design parameters are found in standard distillation textbooks (24, 45). The following range of sieve tray parameters are generally applied in industry; hole diameter 0.16 - 1.27 cm, free area 5 - 15 per cent, plate thickness 0.16 - 1.27 cm and weir height 1.27 - 7.62 cm. In view of these recommendations, trays were built with 178, 0.095 cm diameter holes and 9.83 per cent free area. Plate thickness was 0.16 cm and weir height 1.91 cm. These plates were tested with acetone and water to represent the column's solvent recovery and stripping sections. No weeping occurred with boil-up rates greater than 55 kcal/hr, for acetone. However with water, weeping was observed throughout the reboiler boil-up range (0 - 320 kcal/hr). This difference in behaviour is due to the chemical properties of acetone and water. Surface tension of acetone and water are significantly different. Acetone, as with most organic compounds, has low surface tension (20 dynes/cm), whereas water has high surface tension (60 dynes/cm). This difference could account for the observed weeping.

Trays were rebuilt with 93 holes, 0.089 cm in diameter to reduce weeping with water. This corresponded to 2.85 per cent free area, in contrast to the original free area design of 9.83 per cent. These trays were tested and weeping occurred at 30 and 130 kcal/hr for acetone and water, respectively. This was well below the design boil-up rate of 190 kcal/hr. Therefore the tray design was satisfactory with respect to weeping.

Tests were next conducted with design feed and product rates to determine the lower boil-up operating limit of the column. Trays 6 and 9 weeped with a boil-up rate of 160 kcal/hr. It was felt that tray unlevelness was partially responsible for this weeping. Therefore, a 1.91 cm spacer was inserted between column and laboratory frame, thus tilting the column 1.2°. Experiments were repeated and trays 12 and 13 weeped but at a higher boil-up rate (210 kcal/hr) than before. The lowest boil-up without weeping (150 kcal/hr) was obtained with the column tilted 0.4° from the original position. These tests indicate that weeping characteristics are very sensitive to tray levelness.

5.2 Column Operating Level and Limits

Equipment operating levels in industrial processes are set to maximize economic return within safe operating limits. However, the extractive distillation column is not experimentally simulating a physical plant, therefore column operating level is not set. This level is somewhat arbitrary but must be within the column operating limits and give an acetone-methanol separation that is typical of industrial columns. Ideal operating level is midrange between the upper and lower limits of disturbance and manipulated variables. In this case, the column is disturbed and controlled over the widest possible range.

The "base case" operating level of the column is given in Table 5-1. The extractive distillation column is brought to

TABLE 5-1

Base Case Operating Level of the Extractive Distillation Column - Run 23-3

Feed rate	15.86	gm moles/hr
Feed temp	45.0	deg. C.
Feed composition	0.5910	mole fraction acetone
	0.4027	mole fraction methanol
	0.0063	mole fraction water
Feed tray	11	
Solvent rate	27.32	gm moles/hr
Solvent temp.	70.0	deg. C.
Solvent composition	1.0	mole fraction water
Solvent tray	6	
Overhead product rate	10.28	gm moles/hr
Overhead product composition (approximate)	0.895	mole fraction acetone
	0.065	mole fraction methanol
	0.040	mole fraction water
Reflux ratio	1.756	
Boil-up rate	200	kcal/hr
Bottom product composition (approximate)	0.004	mole fraction acetone
	0.170	mole fraction methanol
	0.826	mole fraction water
Total trays	13 + reboiler + total condenser	

this steady state level at the beginning of each dynamic test. Disturbance and control parameters are then perturbed, with step or pulse changes, to obtain overhead and bottom composition dynamic responses. Dynamic behaviour of the column is presented in the next chapter.

Base case operating level varies within a series of experiments (Table 5-2). Manual setting of feed and boil-up rates produce this error. Overhead product rate is set indirectly by boil-up rate and strongly influences overhead product composition. Installing an automatic overhead product rate controller would reduce the overhead composition range (0.894 - 0.910 mole fraction acetone) and give better base case reproducability.

Base case tray liquid heights and volumetric holdups are given in Table 5-3. Liquid heights are measured visually and range from 3.2 to 9.5 cm. Heights are lowest in the solvent recovery section and highest on trays below the solvent and feed trays. Volumetric tray holdup is determined indirectly from pressure drop across an operating tray and tray cross-sectional area. Dry tray pressure drop is very small. Tray holdups are relatively constant throughout the column (32 - 42 ml). The reboiler has a large effect upon column dynamic behaviour. This is due to the large reboiler holdup in comparison to tray holdups.

TABLE 5-2

Range of Base Case Operating Level

Overhead product composition (mole fraction acetone)	0.910 to 0.894
Overhead product rate (gm mole/hr)	10.8 to 10.3
Feed rate (gm moles/hr)	16.4 to 15.9
Feed composition (mole fraction acetone)	0.593 to 0.588
Solvent rate (gm moles/hr)	28.1 to 26.6

TABLE 5-3

Base Case Tray Heights and Volumetric Holdups

	Liquid Heights (cm)	Volumetric Holdups (ml)
Condenser		35
Tray 1	3.2	32
" 2	3.2	32
" 3	3.2	32
" 4	3.2	32
" 5	3.8	32
" 6	5.7	40
" 7	9.5	32
" 8	7.6	40
" 9	4.4	38
" 10	3.2	42
" 11	6.4	40
" 12	7.6	33
" 13	3.8	32
Reboiler Level	(high)	409
	(normal)	327
	(low)	257

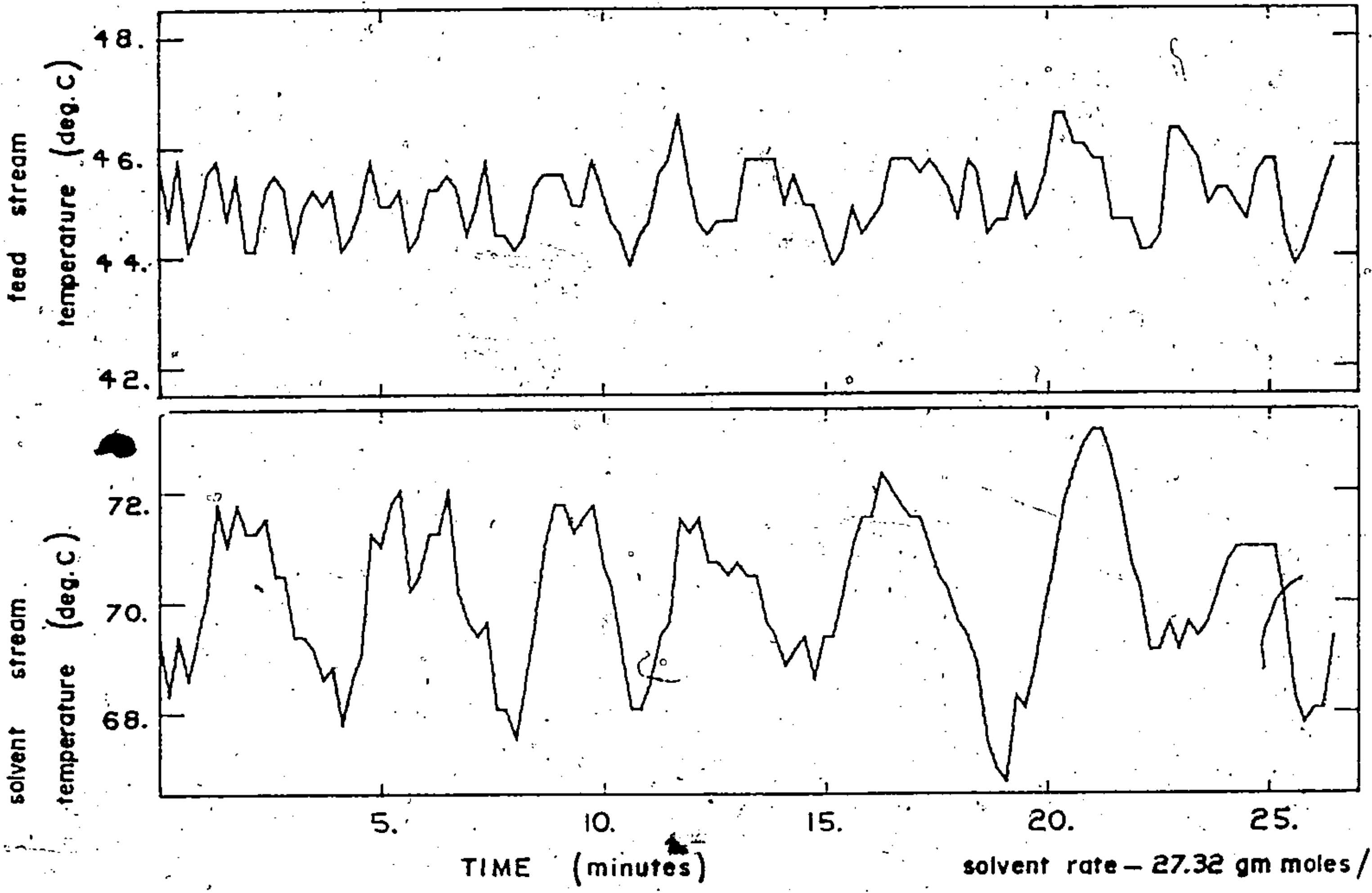
Reboiler holdup is normally 327 ml. Upper and lower limits of 409 and 257 ml occur during column dynamic response testing. Reboiler level varies with total holdup on the trays. This is affected by boil-up rate and liquid rates within the column. To obtain constant reboiler holdup, the bottom product gravity overflow system must be replaced by an automatic level controller.

Base case feed and solvent stream temperatures are set at 45.0 and 70.0 deg. C., respectively. The control computer uses on/off control to regulate stream set point temperatures. Figure 5-1 presents stream temperatures taken at 13 second intervals and base case flows. The temperature controller checked stream temperatures and took control action once per second. Stream temperatures have a maximum error of 1.58 and 3.29 degree C for feed and solvent streams, respectively. This error may be reduced by regulating applied preheater voltage with a PID controller.

A distillation column has a number of variables that may limit the operating range. Inadequate feed flow, undersized reboiler, limited condenser duty or tray priming may set the upper limit. Low internal column flows, with tray weeping resulting, set the lower limit. The upper limit for the extractive distillation column is priming on trays 7 or 11. Feed flow rate, reboiler duty and condenser duty are restricted

FIGURE 5-1 Feed and Solvent Stream Temperatures

feed rate – 15.86 gm moles/hr
preheater voltage – 15



solvent rate – 27.32 gm moles/hr
preheater voltage – 27

below maximum outputs. The lower limit is weeping on trays 6 or 13.

Table 5-4 gives the extractive distillation column steady state operating limits. Upper and lower limits were determined for disturbance variables (feed rate and composition) and manipulated variables (boil-up rate and overhead product rate). This data was obtained from steady state operation. The column operated satisfactorily for short time periods outside these limits during experiments investigating the dynamic behaviour of the system. Feed rate is normally set at 15.9 gm moles/hr and has an upper limit of 18.0 gm moles/hr due to tray 7 or 11 priming. Priming occurs when high liquid level on a tray carries over with vapour to the tray above. This is unsatisfactory because one equilibrium stage is lost due to liquid back mixing. Tray liquid level and priming vary with liquid rate, vapour rate and tray position within the column. The acetone rich solvent recovery section has low tray liquid heights (3.2 cm). In contrast, the absorbing and stripping sections have high tray liquid heights (3.2 - 9.5 cm) and higher methanol-water compositions. Acetone concentration and tray liquid height decrease rapidly toward the bottom of the stripping section. High tray liquid level coincides with high acetone concentration in a predominantly methanol-water mixture. When feed rate increases, more acetone enters the column and tray 7 or 11 liquid heights increase and

TABLE 5-4

Extractive Distillation Column Operating Limits

	Upper Limit*	Normal Operating Level	Lower Limit*
Feed rate (gm moles/hr)	18.0 (tray 7 or 11 priming)	15.9	UNKNOWN (<14.9) (tray 13 weeping)
Feed composition (mole fraction acetone)	0.656 (tray 7 or 11 priming)	0.591	UNKNOWN (<0.560) (tray 13 weeping)
Boil-up rate (kcal/hr)	235 (tray 7 priming)	200	150 (tray 6 weeping)
Overhead product rate (gm moles/hr)	UNKNOWN (>10.7) (weeping)	10.3	9.0 OR 94% acetone in overhead (tray 7 priming)

*Limits greater when pulses shorter than 10 minutes priming occurs.

The feed rate lower limit is tray 13 weeping. This tray weeps slightly during normal operation. Liquid is lost on tray 13 when total liquid rate is less than the tray weeping rate. This did not occur with the lowest experimental feed rate of 14.9 gm moles/hr.

Feed composition is normally 0.591 mole fraction acetone and has the same effect upon column operation as feed rate. Trays 7 or 11 prime at the upper limit of 0.656 mole fraction acetone. At the lower limit (less than 0.560 mole fraction acetone) tray 13 weeps.

The normal boil-up rate operating level is 200 kcal/hr. High vapour and liquid rates at the upper limit of 235 kcal/hr produce priming. Tray 6 weeps at the lower boil-up limit of 150 kcal/hr.

Upper limit for the overhead product rate is unknown. The column operates satisfactorily at a product rate of 10.7 gm moles/hr. Tray weeping occurs at an upper limit when increased overhead rates reduce tray liquid rates significantly. The lower overhead product rate limit is 9.0 gm moles/hr with tray priming. This corresponds to an overhead product concentration of 0.94 mole fraction acetone. At this overhead concentration, acetone is pushed lower down the column. This increases tray liquid levels and produces priming. Column modifications are required for overhead acetone concentrations greater than 0.94

mole fraction. Liquid height on tray 7 would be reduced by increasing hole diameter and downcomer cross sectional area or decreasing weir height. Introducing an anti-foam agent into the solvent feed stream is an alternative solution.

5.3 Simulation and Experimental Steady State Results

This section relates the steady state simulation to experimental results. Mathematical models of a process often have parameters that cannot be calculated accurately from basic engineering principles. Catalyst activity and decay are reactor examples. Murphree tray vapour efficiencies are difficult to predict in distillation.

Tray efficiencies are influenced by liquid rate, vapour rate, tray physical parameters and chemical properties of the distilled mixtures. A wide range of efficiencies have been reported. They can be negative in multicomponent systems due to component diffusion interactions in liquids (44) and greater than 100 per cent on trays with incomplete liquid mixing. High plate efficiencies (60-100 percent) are reported (14) for sieve plates. For multicomponent distillation with n components, efficiencies may be different for (n-1) independent components. Saturated liquid temperature requirements set the n-th component efficiency. Stream samples were taken at base case operating levels in Run 23-3 (operating conditions given in Table 5-1). Samples included overhead and bottom products, tray 5 and 10 liquid, tray 6 and 11 vapour and reboiler vapour

(Table 5-5). A series of simulation cases were computed using different efficiencies to fit experimental compositions. Table 5-5 gives the final fitted simulation compositions and Murphree vapour efficiencies used. Experimental product and vapour compositions compare well to the simulation, but liquid compositions do not. High acetone concentrations in the experimental liquid results can be attributed to poor sampling. The sample is drawn off the tray, carrying with it vapour that is bubbling through the liquid. The vapour has a higher acetone composition thus giving unrepresentative liquid samples. Relocating the liquid sampler in the downcomer liquid seal is a solution to this sampling problem.

Reboiler component Murphree vapour efficiencies are calculated directly from bottom product compositions, known vapour equilibrium relationships and reboiler vapour compositions. For Run 23-3, calculated acetone, methanol and water efficiencies are 198, 120 and 132 per cent respectively. The reboiler is generating vapour higher in acetone and methanol composition than an equilibrium stage. These components have lower boiling points than water and are vapourized easier. Vapour and liquid do not attain equilibrium because vapour-liquid contact time is short. The side-arm has a small volume and vapour leaves the side-arm above the reboiler liquid level. Reboiler efficiencies may be affected by reboiler liquid level and heat input but results were only obtained at base case operating conditions.

TABLE 5-5

Comparison of Run 23-3 Experimental and Simulation Results

	Composition (mole fraction)		
	Acetone	Methanol	Water
Overhead product - experiment	0.8976	0.0596	0.0428
- simulation	0.8940	0.0521	0.0539
Tray 5 liquid - experiment	0.6707	0.0651	0.2642
- simulation	0.5578	0.0948	0.3474
Tray 10 liquid - experiment	0.1147	0.1758	0.7095
- simulation	0.0912	0.1541	0.7547
Bottom product - experiment	0.0036	0.1725	0.8239
- simulation	0.0056	0.1779	0.8165
Tray 6 vapour - experiment	0.7324	0.0644	0.2032
- simulation	0.6904	0.0780	0.2316
Tray 11 vapour - experiment	0.5324	0.2966	0.1710
- simulation	0.5171	0.2793	0.2036
Reboiler vapour - experiment	0.0960	0.5793	0.3247
- simulation	0.1248	0.5763	0.2989
Murphree vapour efficiencies			
solvent recovery section	90% for all components		
stripping section	75% for all components		
absorbing section	90% for all components		
reboiler	198% for acetone 120% for methanol 132% for water		

Tray efficiencies of 90, 75 and 90 per cent were obtained for the solvent recovery, stripping and absorbing sections, respectively. These efficiencies were manually adjusted until close agreement was obtained between simulation and experiment results. A non-linear regression analysis was not used in order that the sensitivity and interaction among the efficiencies could be understood.

The steady state simulation results are relatively insensitive to plate efficiency variations. Changing all tray efficiencies from 60 to 100 per cent will only produce a 0.05 mole fraction increase in overhead acetone composition. Sensitivity of overhead composition to changes in individual section Murphree vapour efficiencies is given in Table 5-6. Overhead acetone composition is sensitive to the absorbing section efficiencies and both overhead methanol and water to the efficiencies in the absorbing and solvent recovery sections. The stripping section efficiencies have a relatively small effect upon overhead composition.

Other steady state runs were simulated using Murphree efficiencies obtained in fitting Run 23-3 data. These runs are at the base case operating level or at other levels obtained during step changes in dynamic testing (Table 5-7). Parameters ranged as follows; feed rate from 14.83 to 17.07 gm moles/hr, feed composition from 0.575 to 0.608 mole fraction acetone,

TABLE 5-6

Sensitivity of Overhead Composition Simulation Results to Murphree Vapour Efficiency

	Murphree Efficiency* per cent	Overhead composition change (mole fraction)		
		Acetone	Methanol	Water
Solvent Recovery Section	75 to 100	+0.004	+0.011	-0.015
Stripping Section	75 to 100	+0.003	-0.003	--
Absorbing Section	75 to 100	+0.012	-0.012	--

*Efficiencies of all other sections at 75 per cent

TABLE 5-7

Experimental Steady State Conditions

Run No.	Feed Rate gm moles/hr	Feed Comp. M.F.Acetone	Solvent Rate gm moles/hr	Overhead Rate gm moles/hr	Boil-up Rate Kcal/hr
18-1	14.830	0.606	28.314	10.521	200
18-2	17.070	0.606	28.314	11.200	200
20-1	16.164	0.608	27.648	10.840	200
20-2	16.465	0.575	27.648	10.840	200
22-2	16.372	0.589	28.048	10.389	200
22-3	16.321	0.589	28.148	10.193	200
23-2	16.280	0.591	26.749	10.098	155
23-3	15.864	0.591	27.315	10.284	200
24-1	16.329	0.593	26.649	10.491	200
24-2	16.329	0.593	26.649	10.101	165

overhead rate from 10.101 to 11.20 gm moles/hr and boil-up rate from 155 to 200 kcal/hr. Comparison between simulation and experimental product compositions are given in Table 5-8. The predicted overhead acetone and methanol compositions tend to be lower than the experimental and water higher. But in general, the simulation accuracy is good. The poorest agreement is obtained in cases with low boil-up rates (Runs 23-2, 24-2). This may be due to different reboiler efficiencies at these conditions. Future experimental work should include the effect of boil-up rate and reboiler level on efficiency.

The simulation model predicted the effect of disturbance and manipulated variable changes upon overhead and bottom composition. The effect of feed disturbance changes are given in Figure 5-2. Cases were run at different levels of feed composition and rate with Run 23-3 reflux ratio and boil-up rate held constant. Constant overhead acetone composition and bottom water-free acetone composition lines were interpolated from these cases. Both feed acetone composition and rate increases produce higher acetone concentrations in the product streams. The distillation column produces non-linear effects from feed composition and rate changes. Steady state open loop disturbance gains can be calculated from this data.

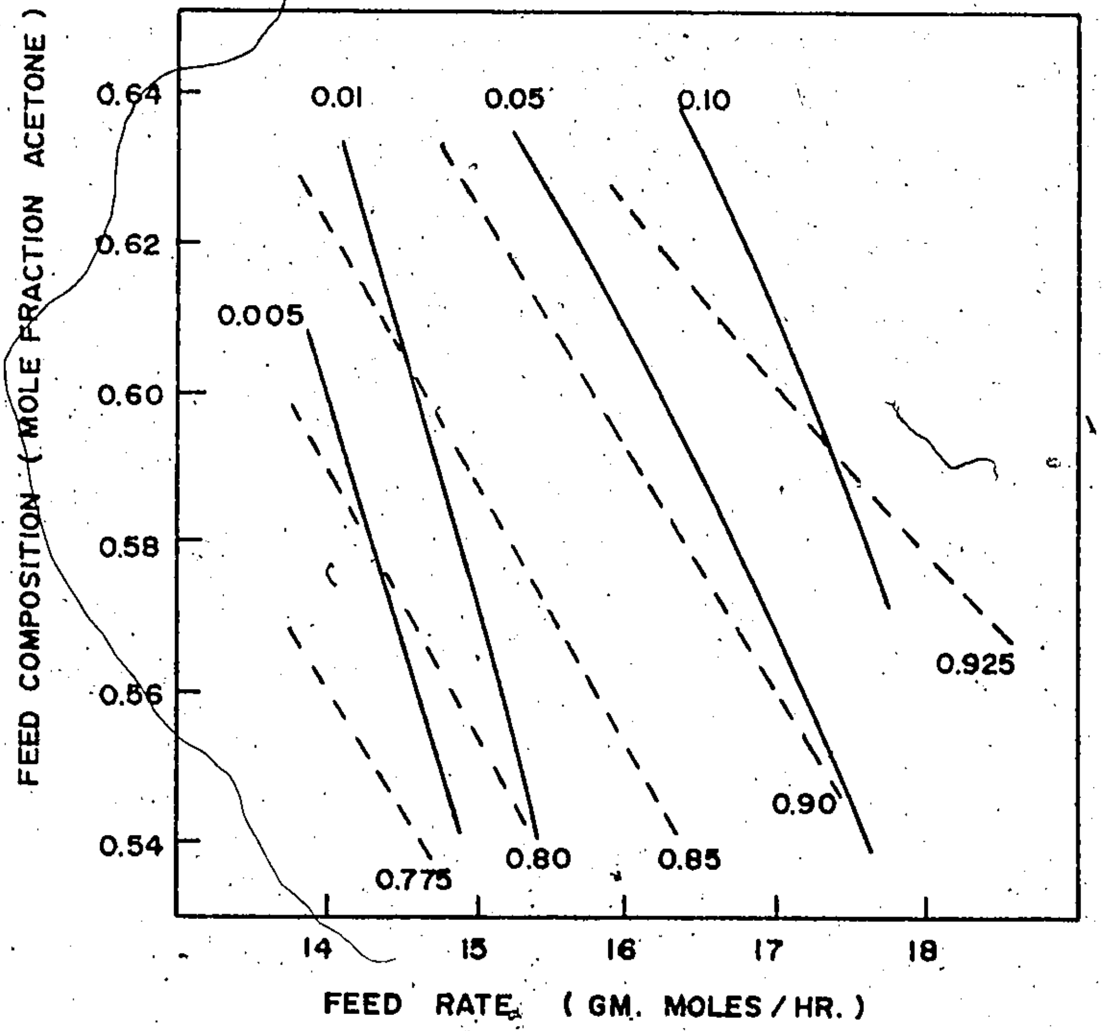
Figure 5-3 shows the effect of manipulated variables on product compositions. Results were obtained by varying

TABLE 5-8

Comparison Between Simulation and Experimental Product Composition Using Run 23-3 Fitted Murphree Efficiencies

		Overhead Composition (mole fraction)			Bottom Composition (mole fraction)		
Run	Results	Acetone	Methanol	Water	Acetone	Methanol	Water
18-1	Experimental	0.8481	0.1239	0.0280	0.0018	0.1365	0.8617
	Simulation	0.8502	0.0988	0.0510	0.0014	0.1443	0.8543
18-2	Experimental	0.9017	0.0613	0.0370	0.0070	0.1714	0.8216
	Simulation	0.9038	0.0424	0.0538	0.0066	0.1796	0.8138
20-1	Experimental	0.8936	0.0747	0.0317	0.0038	0.1615	0.8347
	Simulation	0.8922	0.0542	0.0536	0.0047	0.1708	0.8245
20-2	Experimental	0.8623	0.1086	0.0291	0.0034	0.1724	0.8242
	Simulation	0.8631	0.0850	0.0519	0.0036	0.1778	0.8186
22-2	Experimental	0.9097	0.0503	0.0400	0.0049	0.1783	0.8168
	Simulation	0.9018	0.0430	0.0552	0.0079	0.1822	0.8099
22-3	Experimental	0.9207	0.0351	0.0442	0.0059	0.1781	0.8160
	Simulation	0.9112	0.0338	0.0550	0.0093	0.1833	0.8074
23-2	Experimental	0.9134	0.0428	0.0438	0.0121	0.1851	0.8030
	Simulation	0.8880	0.0467	0.0653	0.0200	0.1847	0.7953
23-3	Experimental	0.8976	0.0596	0.0428	0.0036	0.1725	0.8239
	Simulation	0.8940	0.0521	0.0539	0.0056	0.1779	0.8165
24-1	Experimental	0.9068	0.0587	0.0345	0.0044	0.1856	0.8100
	Simulation	0.9001	0.0459	0.0540	0.0074	0.1877	0.8049
24-2	Experimental	0.9142	0.0451	0.0407	0.0114	0.1901	0.7985
	Simulation	0.8892	0.0455	0.0653	0.0213	0.1861	0.7926
AVERAGE ERROR		0.0085	0.0151	0.0187	0.0031	0.0052	0.0079

FIGURE 5-2
FEED DISTURBANCE EFFECTS ON PRODUCT COMPOSITIONS
BASE CASE OPERATING LEVEL , RUN 23-3



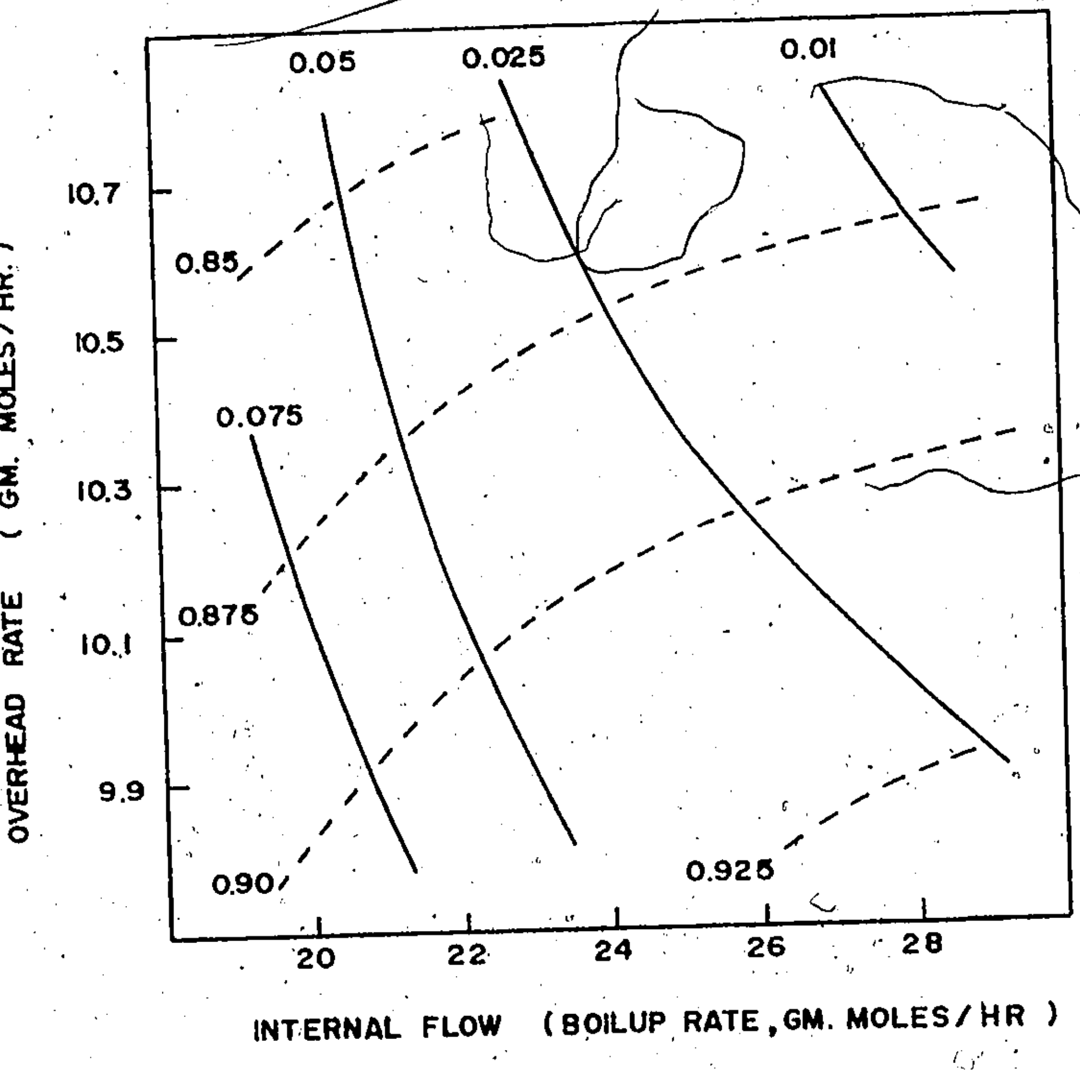
—— bottom product (mole fraction acetone water free)

– – overhead product (mole fraction acetone)

FIGURE 5-3

MANIPULATED VARIABLE EFFECTS ON PRODUCT COMPOSITIONS

BASE CASE OPERATING LEVEL, RUN 23-3



— bottom product (mole fraction acetone water free)

-- overhead product (mole fraction acetone) overhead product rate and boil-up rate with constant feed rate and composition (Run 23-3 conditions). Product compositions are very sensitive to overhead rate. Changes in internal flow rates have a moderate effect at low rates and small effect at high rates. These variables produce non-linear effects on product compositions.

The simulation results are valuable in determining steady state gains. They were used in designing the magnitude of the variable changes for the experimental dynamic testing. The dynamic behaviour of the distillation column is described in the next chapter.

CHAPTER 6

ANALYSIS OF THE DYNAMIC BEHAVIOUR

This work is the second part of a long term project on the application of distillation control. The objective of the extractive distillation project is to evaluate the different levels of sophistication of distillation control. These methods have been described in Chapter 2.

However, in order to design a control scheme, a process model is required relating the disturbance and manipulated variables to the control variables. This model can be either in a physical or "black box" form. A physical model describes the system by a series of non-linear differential equations based on basic engineering principles. These may include mass and energy balances and equilibrium relationships. The non-linear equations are linearized around the desired operating conditions and the order of the resulting process matrix reduced to make the controller design computations easier. In contrast, "black box" models relate single input - single output process variable pairs in transfer function form. Transfer functions are obtained by fitting the output data from a pre-specified input. Input-output data can be obtained from either the process equipment or a dynamic simulation of the unit.

The "black box" model approach is presented in this chapter. It is felt that the "black box" model based on process experiments would be more accurate than a physical model. The latter model requires estimation of several important process characteristics which cannot be accurately predicted. These include stage efficiencies and molar holdup as a function of liquid and vapour rates.

Preliminary work was conducted to develop a dynamic simulation of the extractive distillation column. Differential equations describing the unsteady state behaviour of the extractive distillation column (total and component stream continuity equations and energy continuity equations) were solved using a predictor-corrector method (Adams-Moultan-Shell Method). Steady state output results from the steady state simulation were used to initialize the dynamic simulation calculations. Time step sizes were varied to determine stable steady state level calculations with the largest step size. Relatively small time steps (0.18 - 1.8 seconds) were required. This preliminary study indicated that a large amount of work would be required to fully develop an accurate dynamic model of the extractive distillation column. Therefore, it was felt that experimentally determined dynamic responses would produce reliable results more quickly. Future work may include dynamic simulation that may be verified with experimentally determined responses.

The relationship of disturbance (feed rate and composition) and manipulated (overhead rate and internal column flow) variables to controlled variables (overhead and bottom compositions) were investigated by experimental column responses. Each disturbance and manipulated variable was manually regulated in the form of step and pulse inputs to determine the responses of the controlled variables. Transfer functions relating the input and output variables were determined from both the frequency and time domains.

6.1 Frequency Response and Time Domain Methods

In the frequency approach, the Bode plot (log amplitude ratios vs. log frequency and phase angles vs. log frequency) is obtained from the ratio of the Fourier transforms of the output and input responses. The process mathematical model may be determined from the Bode plot (31), or controller constants estimated by the Ziegler-Nichols method (9). Pulse testing is required to determine the frequency response of a system. An input variable is manually adjusted from a normal steady state level and the output variables measured. The amplitude and shape of the input pulse affects the frequency response results. The amplitude should be large enough to give reasonable measurement accuracy but it should not drive the system non-linear or outside the operating region of the equipment. A rectangular pulse is usually not as good as a triangular shaped pulse. The advantages and disadvantages of different pulse shapes are discussed by Hougen (20). Improved high frequency results are obtained by shortening the pulse duration. However, there is a deterioration of accuracy above a "breakdown frequency". This frequency is a function of data accuracy and the time interval between data points. To obtain frequency data out to frequency W_r radians/time unit, a theoretical minimum sampling rate can be determined from Shannon's Sampling Theorem

$$\Delta T = \frac{\pi}{W_r}$$

where ΔT is the time interval between data points.

A rectangular input pulse approximately nine minutes in duration was used in the present work. Accurate Bode plots were obtained for a distillation column using a rectangular pulse in frequency response data presented by Wood and Pacey (49). A rectangular pulse was used rather than a triangular pulse because it was easier to implement experimentally. Product compositions were sampled at 4.5 minute intervals. This corresponds to a response accurate to a frequency of 0.7 radians/minute.

The frequency response data relating the input and output variables was calculated from a real-valued version of the Fast Fourier Transform presented by Dollar (13). This algorithm was reproduced and checked by comparing computational examples presented by Dollar.

Experimental frequency response data presented in Bode plot form was fitted by first and second order transfer functions with dead times using a non-linear regression routine (Marquardt's Compromise). These results are compared to the transfer function parameters obtained from the time domain.

The experimental time response data may be fitted directly by first and second order transfer functions with dead time using a non-linear regression routine. The rectangular input pulse is approximated by two step changes. One step change is at the start of the pulse and the second step change is in the opposite direction at the end of the pulse. Experimental step change responses may be fitted by a single step change transfer function model.

Experimental results obtained by step changes are widely used in industry. Process responses can be fitted by first or second order systems with dead time graphically, whereas pulse testing requires computer computations. However, pulse testing using well designed inputs obtains more process model information, especially at higher frequencies. Both pulse testing and step changes are used in the present work to evaluate these two methods.

6.2 Experimental Results Relating to the Dynamic Behaviour

Bottom and overhead product composition responses were determined for changes in disturbance and manipulated variables.

Responses to a single input variable were investigated during each experimental run. The extractive distillation column was operated at normal operating input variable levels (Table 5-1) for the first four hours of operation to obtain column steady state. Then, product stream sampling was started and step or pulse changes were introduced into the column. Product sampling was conducted at 9 and 4-1/2 minute invervals for step and pulse changes, respectively. Sampling continued for approximately ninety minutes, at which time, a new steady state level was obtained. Normally two step changes and one or two pulse changes were introduced during an experimental run.

Approximately 140 samples were taken during each run. These samples were analysed manually using a gas chromatograph unit described previously. Each sample was analysed twice with the results averaged. Sample analyses required approximately twenty five hours per experimental run.

A software package monitored the operation of the extractive distillation column. This included logging overhead and bottom column temperatures, feed flow rate and temperature and solvent flow rate and temperature. Warning lights at the process site were turned on by the control computer when flow rates were outside specified limits. The operator would manually correct the flows. Feed and solvent temperatures were controlled around a set point by on-off control. A detailed description of the software package and process interfacing is given by Jackson (22).

Column responses were determined for input variable changes in feed rate, feed composition, overhead rate and internal flow. A large experimental effort is required to fully describe the dynamic behaviour of the column. The behaviour is primarily dependent upon the column's physical characteristics and chemical properties of the components separated. However, important secondary effects include the operating level of the column, magnitude of input variable changes (due to the non-linear column effects) and interaction among input variable changes. Only a preliminary investigation into the dynamic behaviour of the column was conducted due to the limited amount of experimental time and the large amount of time to conduct and analyse a run (5-6 days).

Responses to feed rate step and pulse changes are presented in Figures 6-1 and 6-2 respectively. Overhead product composition is represented by mole fraction acetone and bottom product composition by mole fraction acetone, water free. The latter represents the product of a second column, the solvent recovery column. The bottom product of the extractive distillation column would be fed to this column where the water would be completely separated. The design and construction of this column will be carried out in future work of this project.

The product composition responses are essentially complete within ninety minutes of the feed rate change. Frequency response results in Bode plot form for feed rate pulse responses in overhead and bottom product compositions are given in Figures 6-3

FIGURE 6-1 FEED RATE STEP RESPONSES
RUN 18-2

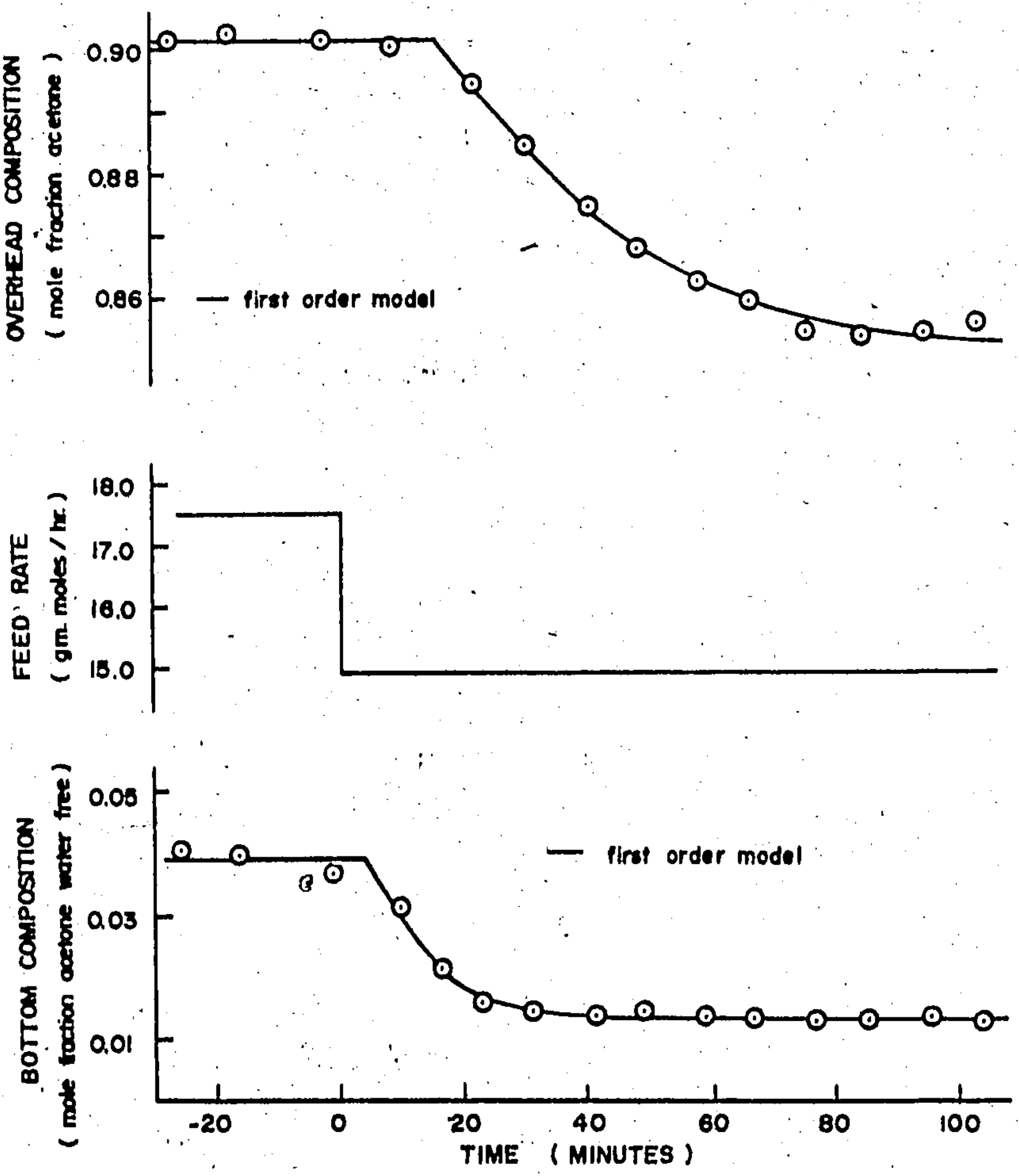
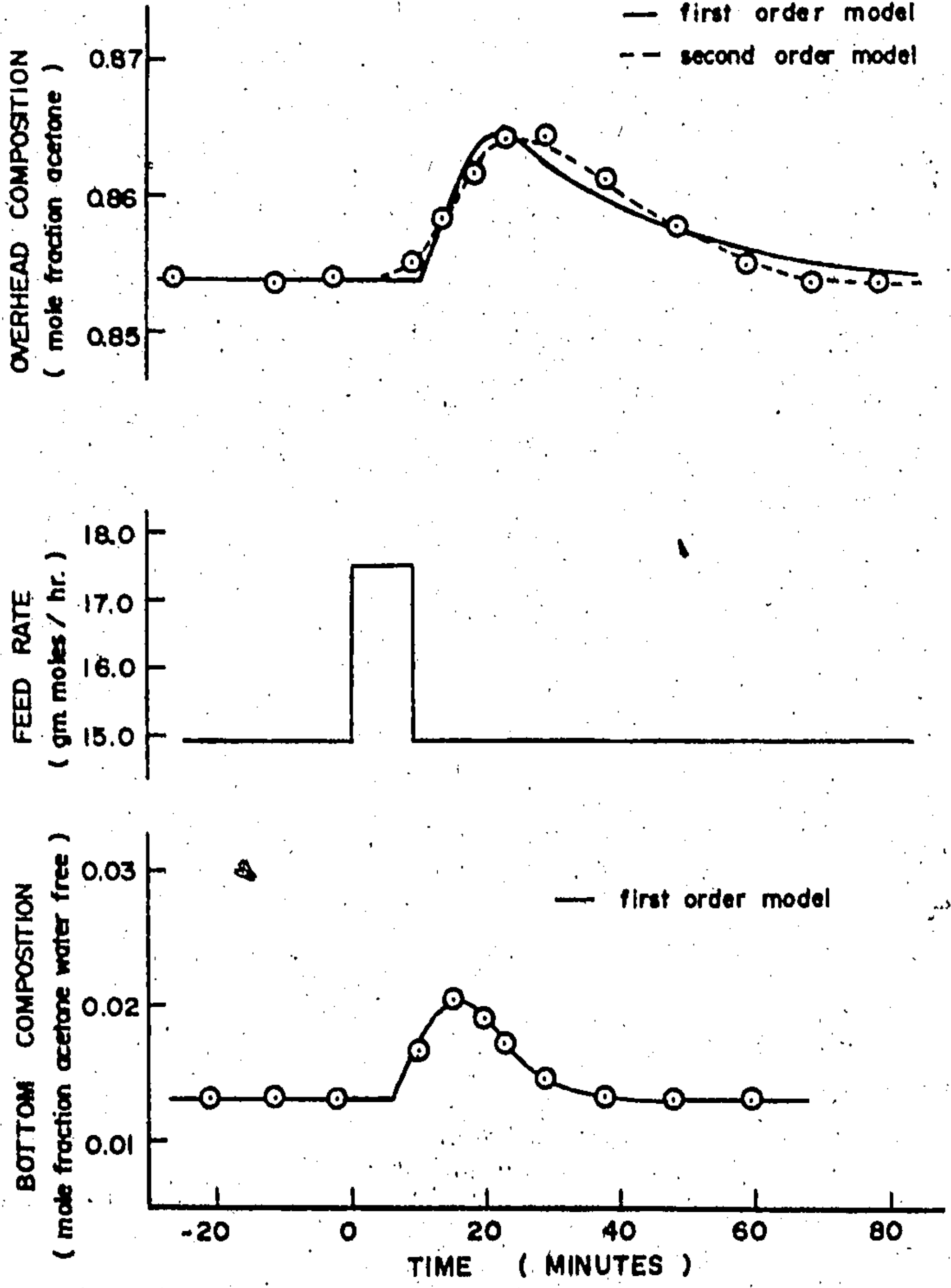


FIGURE 6-2 FEED RATE PULSE RESPONSES
RUN 18-3



and 6.4 respectively. Frequency response can be seen past the first cornering frequency. The theoretical breakdown frequency as predicted by Shannon's Sampling Theorem is indicated. In overhead composition response, the frequency response breaks down before the theoretical frequency due to experimental noise. However the bottom composition frequency response is exceptionally good and data is obtained up to the theoretical frequency.

Parameters fitted by a non-linear regression routine for first and second order transfer function models in overhead composition are presented in Tables 6-1 and 6-2. Responses using these models have been compared to experimental responses in Figures 6-1 to 6-4. A first order transfer model describes the experimental results accurately except for the response to the rectangular pulse in Run 18-3. In this case, a second order model is required.

The transfer function gain is larger for a step response than for a rectangular pulse. This is a common observation for all of the input variables studied and is likely due to non-linear effects. More extensive testing with different magnitude variables changes is required to confirm this.

Table 6-3 presents first order model parameters fitted to the bottom product composition response. Bottom product composition gains and time constants are smaller than that for the overhead. Therefore bottom product composition is affected

FIGURE 6-3 BODE PLOT OF OVERHEAD COMPOSITION RESPONSE TO FEED RATE PULSE RUN 18-3

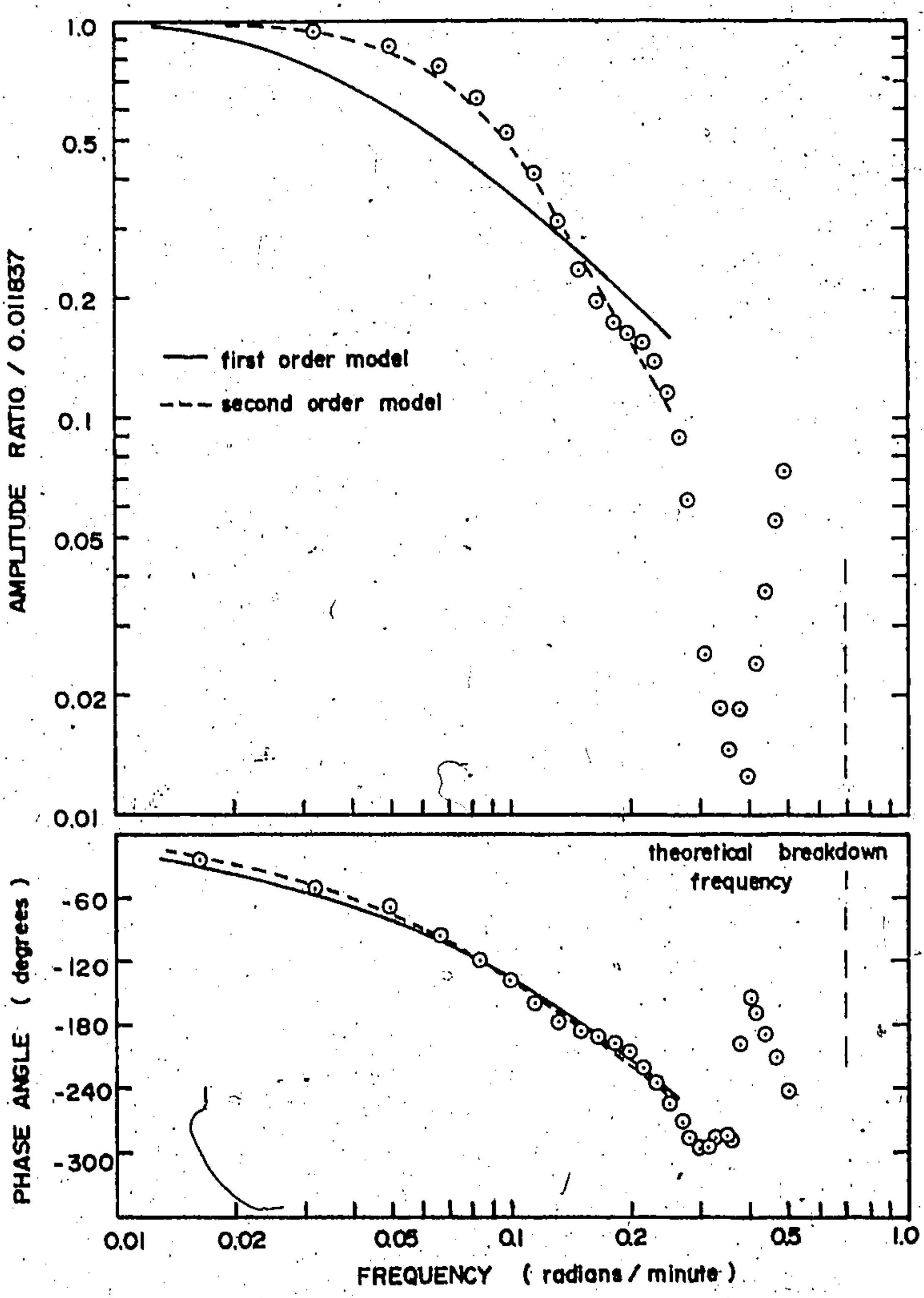


FIGURE 6-4 BODE PLOT OF BOTTOM COMPOSITION RESPONSE TO FEED RATE PULSE

RUN 18-3

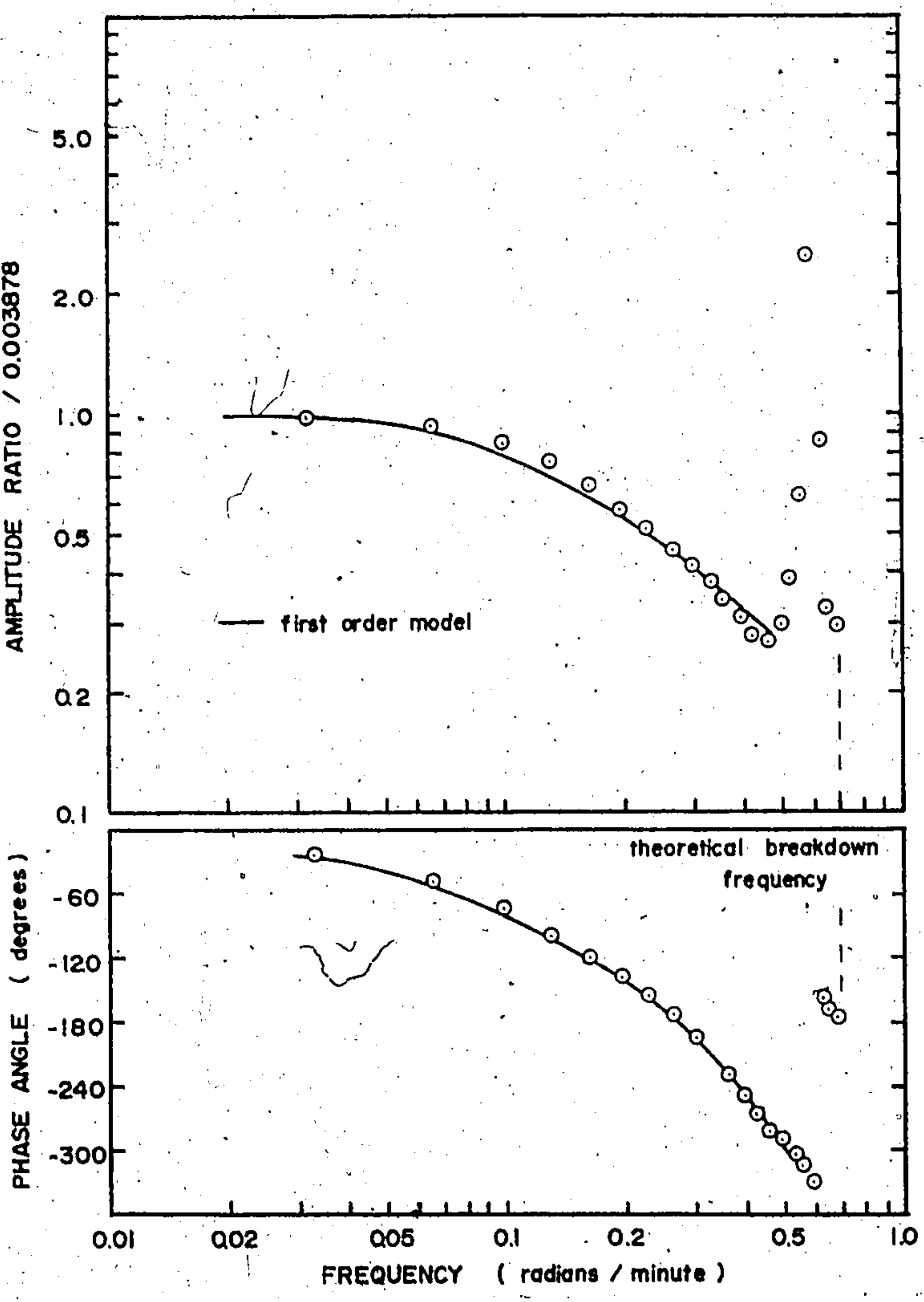


TABLE 6-1

First Order Model Parameters for Overhead Composition Responses to Feed Rate Changes

$$\frac{\Delta\text{Overhead composition (mole fraction acetone)}}{\Delta\text{Feed rate (gm moles/hr)}} = \frac{Ke^{-\tau_d s}}{\tau_1 s + 1}$$

Feed Rate Change	K	τ_d	τ_1	Variance of residuals	Degrees of freedom
Run 18-1 step change +2.63 gm moles/hr, time response fit	0.0209	10.65	24.55	2.52×10^{-6}	12
Run 18-2 step change -2.63 gm moles/hr, time response fit	0.0198	18.25	28.39	1.25×10^{-6}	10
Run 18-3 rectangular pulse +2.63 gm moles/hr, time response fit	0.0145	11.48	22.97	2.09×10^{-6}	9
Run 18-3 rectangular pulse +2.63 gm moles/hr, Bode plot fit	0.0118	12.07	25.05	-----	--

TABLE 6-2

Second Order Model Parameters for Overhead Composition Responses to Feed Rate Changes

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Feed rate (gm moles/hr)}} = \frac{K e^{-\tau_d s}}{\tau^2 s^2 + 2\xi\tau s+1}$$

Feed Rate Change	K	τ_d	τ	ξ	Variance of residuals	Degrees of freedom
Run 18-1 step change +2.63 gm moles/hr, time response fit	0.0206	0.95	17.24	0.8488	8.25×10^{-7}	11
Run 18-2 step change -2.63 gm moles/hr, time response fit	0.0181	9.40	18.32	0.8064	6.79×10^{-7}	9
Run 18-3 rectangular pulse +2.63 gm moles/hr, time response fit	0.0125	5.71	13.15	0.6781	1.37×10^{-7}	8
Run 18-3 rectangular pulse +2.63 gm moles/hr, Bode plot fit	0.0118	6.67	12.19	0.8229	-----	--

TABLE 6-3

First Order Model Parameters for Bottom Composition Responses to Feed Rate Changes

$$\frac{\Delta \text{ Bottom composition (mole fraction acetone, water free)}}{\Delta \text{ Feed rate (gm moles/hr)}} = \frac{K e^{-\tau_d s}}{\tau_1 s + 1}$$

Feed Rate Change	K	τ_d	τ_1	Variance of residuals	Degrees of freedom
Run 18-1 step change +2.63 gm moles/hr, time response fit	0.0103	6.05	20.21	1.69×10^{-6}	13
Run 18-2 step change -2.63 gm moles/hr, time response fit	0.0098	6.44	7.28	1.03×10^{-6}	10
Run 18-3 rectangular pulse +2.63 gm moles/hr, time response fit	0.0044	8.15	6.85	5.28×10^{-9}	7
Run 18-3 rectangular pulse +2.63 gm moles/hr, Bode plot fit	0.0039	7.80	7.58	-----	--

faster and to a smaller degree than the overhead. This is due to the high proportion of acetone in the feed stream (0.60 mole fraction acetone) and the position of the feed tray (3 trays above reboiler but 8 trays below the condenser).

Product composition responses to feed composition step and pulse changes are presented in Figures 6-5 and 6-6, respectively. These results are very noisy compared to the feed rate change results. Feed composition changes have a very small effect upon the bottom product composition (gain of 0.076 Δ mole fraction acetone, water free/Δ mole fraction acetone in feed for the step change).

The frequency response of the overhead product composition to a feed composition pulse is given in Figure 6 - 7. The range in which the Bode plot is accurate is reduced due to experimental noise but the cornering frequency can still be seen. No Bode plot could be obtained for the bottom product composition because the response was obscured by composition measurement noise. This noise is due to poor reboiler mixing (resulting in unrepresentative samples) and gas chromatograph analysis error. Bottom product composition is approximately 0.82 mole fraction water. Small errors in the acetone and methanol compositions are magnified when a water free composition basis is calculated.

First and second order transfer function model parameters [illegible]e presented in Tables 6-4 and 6-5 for overhead composition

FIGURE 6-5 FEED COMPOSITION STEP RESPONSES
RUN 20-2

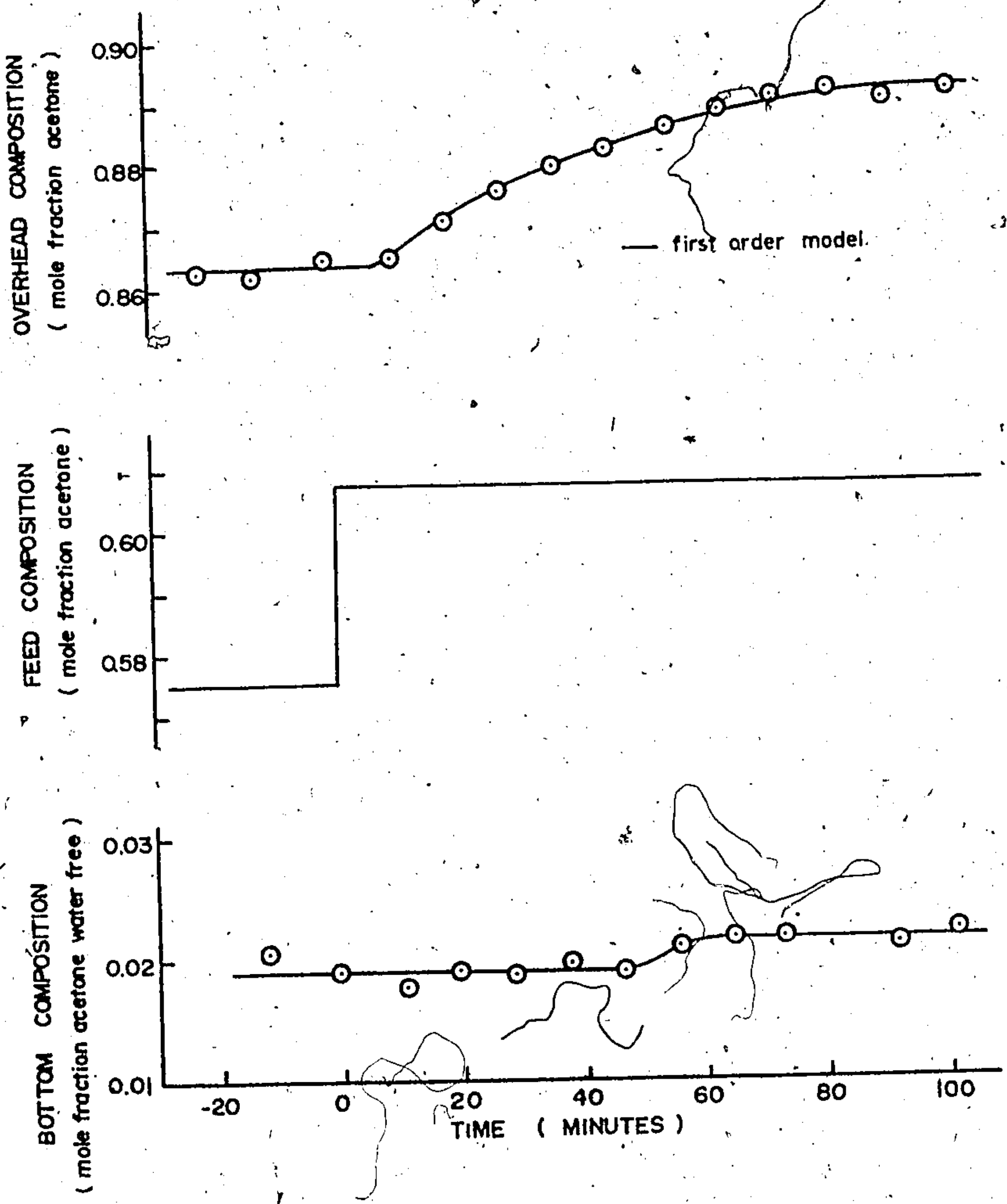


FIGURE 6-6 FEED COMPOSITION PULSE RESPONSES
RUN 20-3

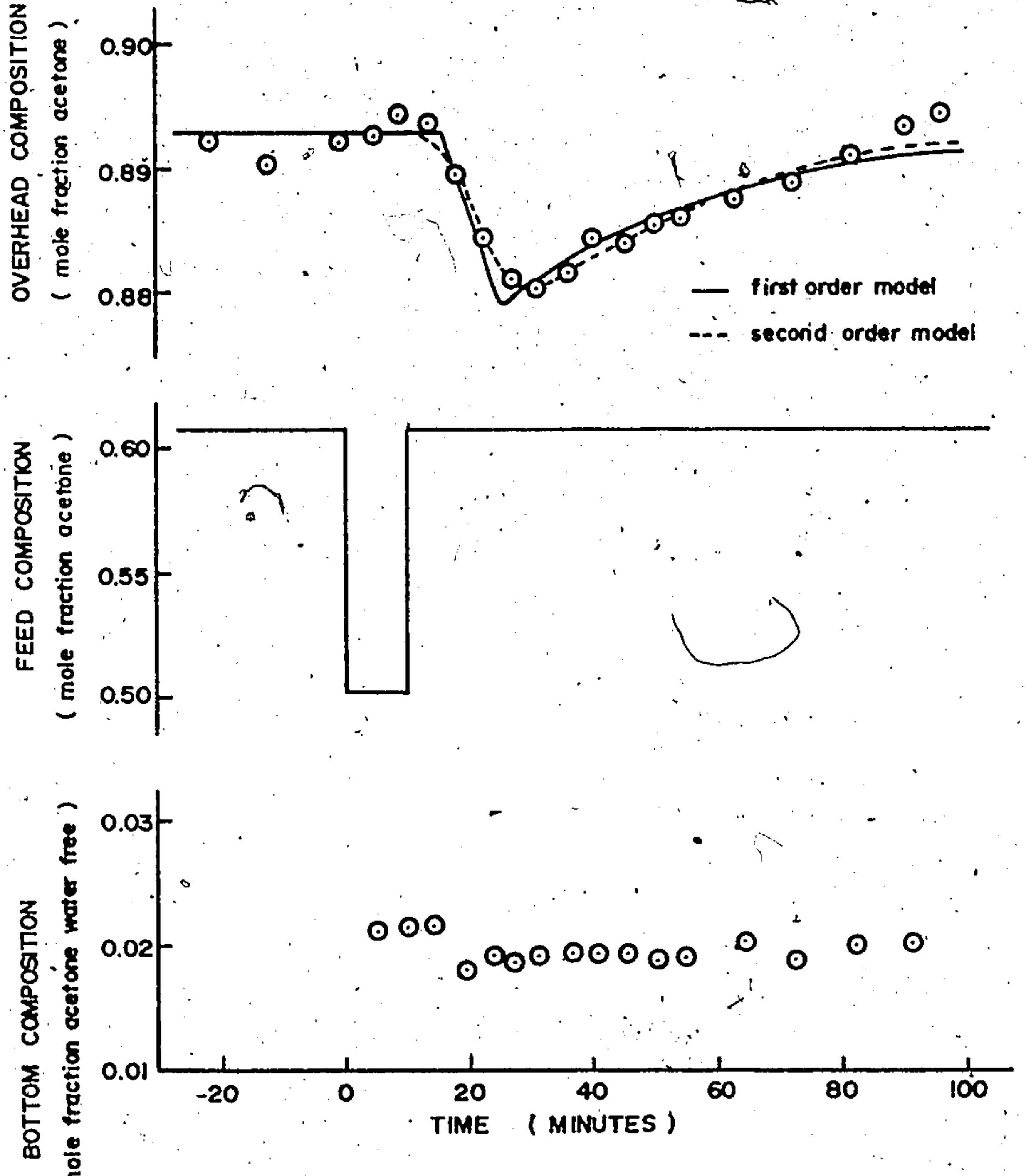
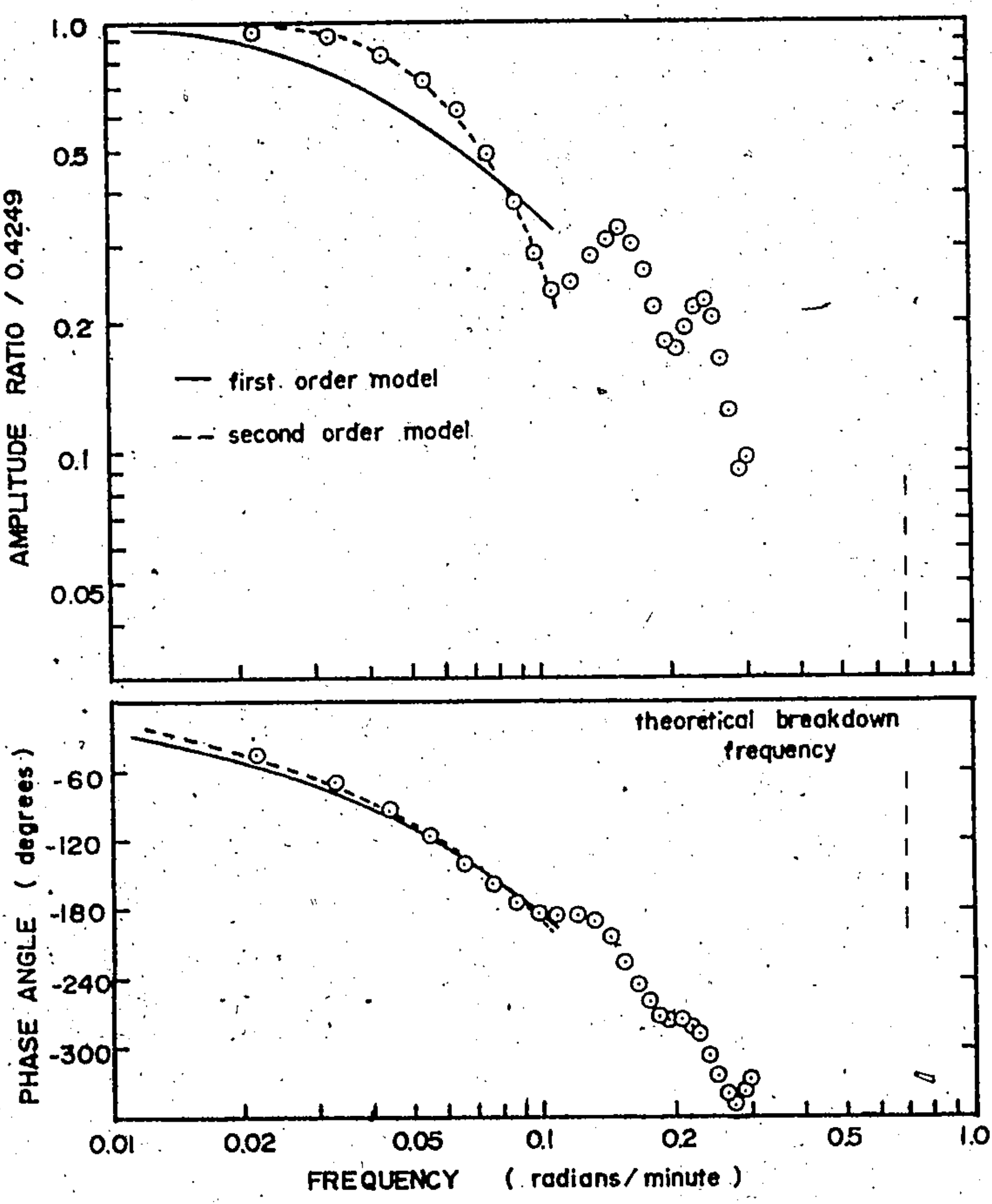


FIGURE 6-7 BODE PLOT OF OVERHEAD COMPOSITION RESPONSE TO FEED COMPOSITION PULSE
RUN 20-3



responses to feed composition changes. There is no significant improvement in fitting a second order model to the experimental data. A second order model may describe the response better, especially to a pulse input, if experimental measurement noise is reduced.

Product composition responses to overhead rate step and pulse changes are presented in Figures 6-8, 6-9 and 6-10. These results are similar to the feed composition change responses. The product composition measurements are noisy and the bottom composition is affected only slightly (gain of 0.025 Δ mole fraction acetone, water free/Δgm mole per hour of overhead rate).

Overhead rate pulse changes were made in opposite directions from the steady state in Figures 6-9 and 6-10. No well defined bottom composition response was obtained with an overhead rate pulse increase of 3.933 gm moles/hr. However, a response was observed for an overhead rate pulse decrease of 3.196 gm moles/hr (Figure 6 - 10). It is interesting to note the experimentally determined compositions at approximately 32 and 50 minutes. These points are significantly below the trend of the response and analysis error is unlikely. A possible explanation may be associated with the liquid flow characteristics of the reboiler. In this case, the overhead product rate is decreased, forcing a larger acetone rich flow down the column into the reboiler. This acetone rich liquid

TABLE 6-4

First Order Model Parameters for Overhead Composition Responses to Feed Composition Changes

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Feed composition (mole fraction acetone)}} = \frac{K e^{-\tau_d s}}{\tau_L s + 1}$$

Feed Composition Change	K	τ_d	τ_1	Variance of residuals	Degrees of freedom
Run 20-1 step change -0.0323 mole fraction acetone time response fit	1.0282	5.18	36.99	3.53×10^{-6}	12
Run 20-2 step change +0.0323 mole fraction acetone time response fit	0.9985	8.10	39.21	1.24×10^{-6}	12
Run 20-3 rectangular pulse -0.1058 mole fraction acetone time response fit	0.4978	16.05	32.44	2.26×10^{-6}	16
Run 20-3 rectangular pulse -0.1058 mole fraction acetone Bode plot fit	0.4249	20.46	25.74	----	--

TABLE 6-5

Second Order Model Parameters for Overhead Composition Responses to Feed Composition Changes

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Feed Composition (mole fraction acetone)}} = \frac{K\,e^{-\tau_d s}}{\tau^2 s^2 + 2\xi\tau s + 1}$$

Feed Composition Change	K	τ_d	τ	ξ	Variance of residuals	Degrees of freedom
Run 20-1 step change -0.0323 mole fraction acetone time response fit	1.0279	4.80	3.75	4.98	3.85×10^{-6}	11
Run 20-2 step change +0.0323 mole fraction acetone time response fit	0.9922	6.51	8.72	2.31	1.34×10^{-6}	11
Run 20-3 rectangular pulse -0.1058 mole fraction acetone time response fit	0.4448	11.73	13.70	1.04	1.74×10^{-6}	15
Run 20-3 rectangular pulse -0.1058 mole fraction acetone Bode plot fit	0.4249	9.55	18.12	0.70	-----	--

FIGURE 6-8 OVERHEAD RATE STEP RESPONSES
RUN 22-2

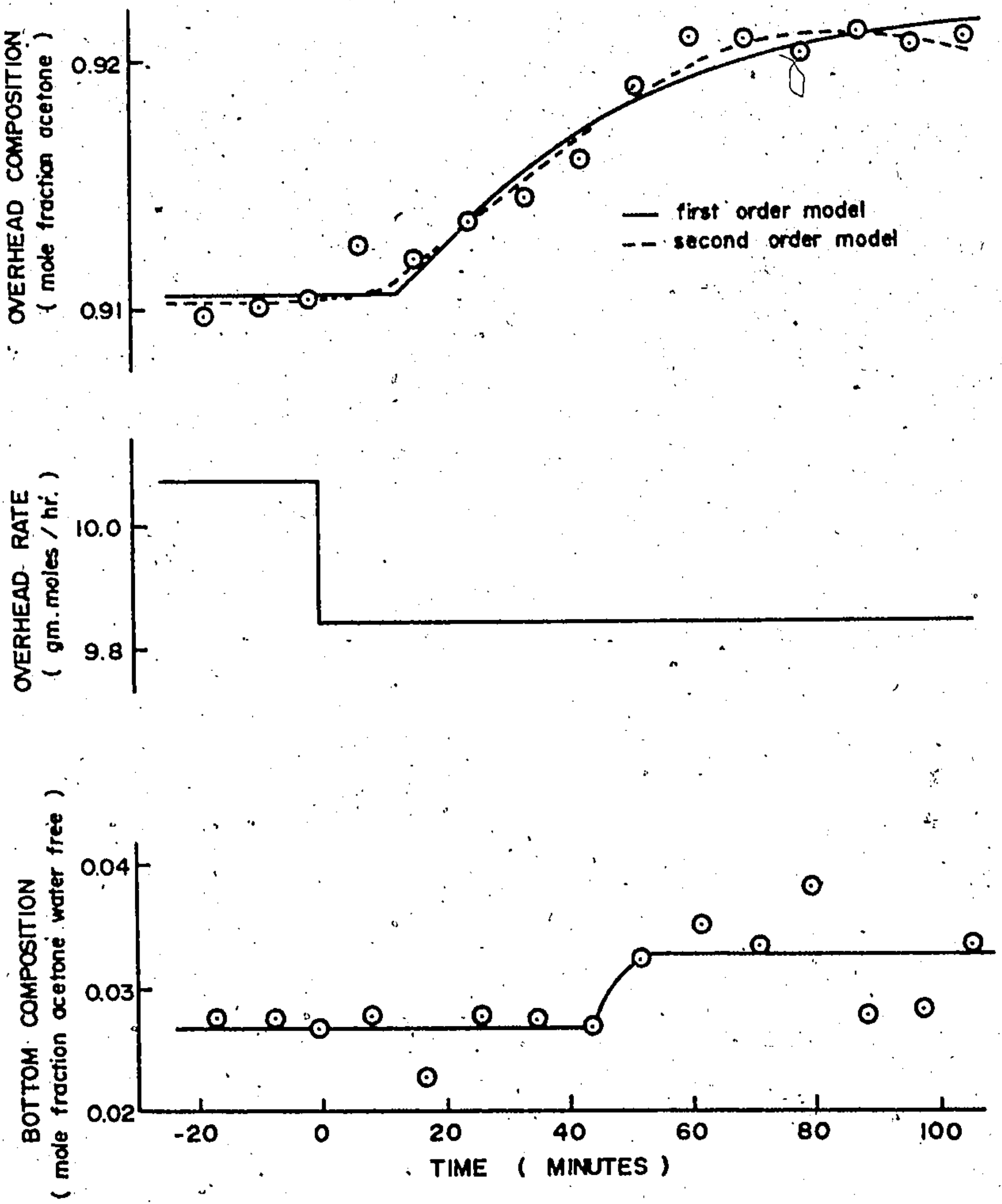


FIGURE 6-9 OVERHEAD RATE PULSE RESPONSES
RUN 22-3

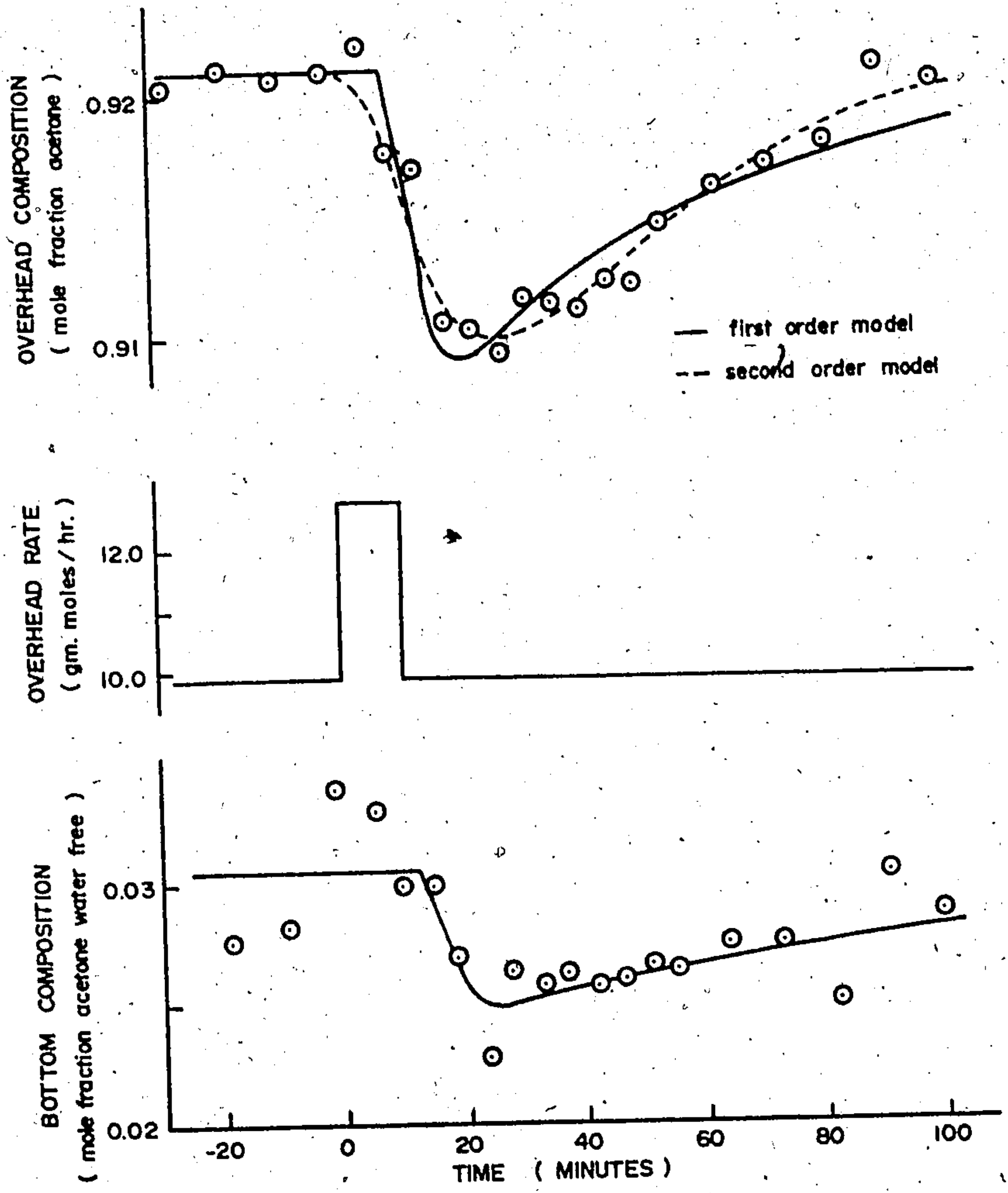
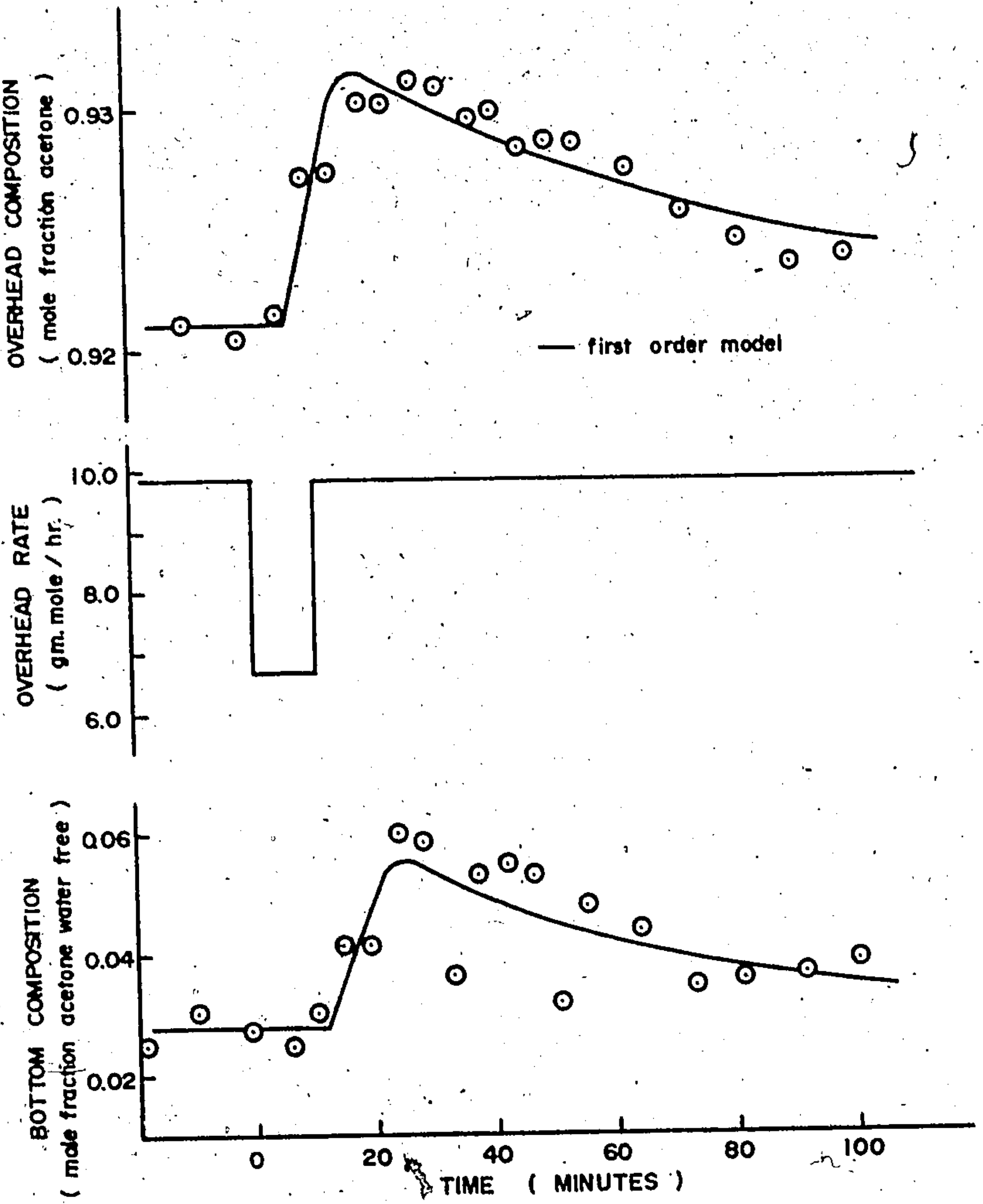


FIGURE 6-10 OVERHEAD RATE PULSE RESPONSES
RUN 22-4



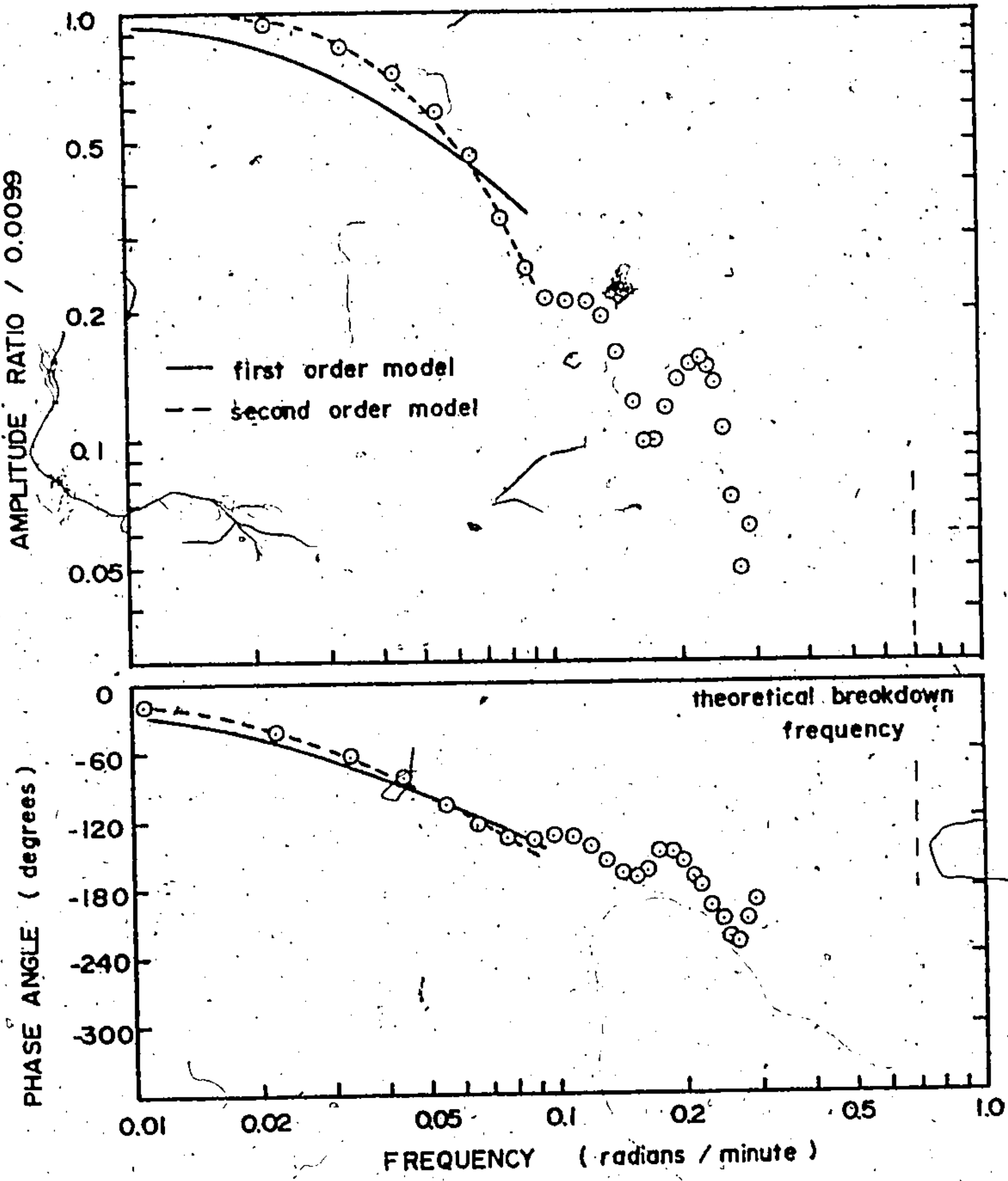
flow returns to normal when the overhead product rate is increased to its initial steady state value. However, the liquid flow within the reboiler may move in a plug-like fashion down the reboiler. A fraction exits through the bottom product stream and the remainder flows through the side-arm heater where a small amount is vapourized. The unvapourized liquid returns back to the reservoir section of the reboiler. If the overhead rate pulse duration is less than the recirculation rate within the reboiler, two slugs of liquid, one acetone rich and one acetone depleted, would recirculate within the reboiler until mixing occurred. This may explain the bottom product response in Figure 6-10.

Final response values are different than the initial steady states values in Figure 6-10. This run shows experimental problems associated with column parameters changing during a response. Slight changes in feed rate, solvent rate or reboiler liquid level may change the product compositions significantly. However, responses generally returned to within acceptable limits after pulse and step changes.

Frequency response results of overhead composition to an overhead rate pulse is presented (Figure 6 - 11). The range of accurate frequency response results is reduced due to composition measurement noise.

First and second order transfer function model parameters are presented in Tables 6-6 and 6-7 for overhead composition

FIGURE 6-11 BODE PLOT OF OVERHEAD COMPOSITION RESPONSE TO OVERHEAD RATE PULSE
RUN 22-3



response to overhead product rate changes. Second order models improve the fit of the experimental results slightly. Changing steady state levels during a response (Run 22-4) produce poor results. First order time lag for Run 22-4 was calculated as 72.2 minutes, as compared to an average of 38.3 minutes for the other cases.

The final variable to be investigated was product composition response to internal flow changes. Experimentally, this was difficult because two variables had to be manually manipulated, reboiler heat input and reflux ratio. A change in reboiler heat input would change the overhead product rate as well as internal column flows. Therefore, reflux ratio was manipulated to obtain constant overhead product rate. However, in the two runs attempted, this was never accomplished to the accuracy required. Previous work on the effect of overhead product rate changes on overhead product composition indicates that a 0.1 gm mole/hr error in overhead rate will produce a 0.0051 mole fraction acetone error in the overhead composition. Overhead product composition is very sensitive to overhead product rate. The problem was intensified because the overhead product flow was intermittant (requiring manual measurement) and the reflux ratio was difficult to adjust to an accurate set point.

Product composition response to a step change in reboiler heat input is presented in Figure 6-12. The amount of component

TABLE 6-6

First Order Model Parameters for Overhead Composition Responses to Overhead Rate Changes

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Overhead product rate (gm moles/hr)}} \quad \frac{K e^{-\tau_d s}}{\tau_1 s + 1}$$

Overhead Product Rate Change	K	τ_d	τ_1	Variance of residuals	Degrees of freedom
Run 22-2 step change -o.23 gm moles/hr time response fit	-0.0512	13.74	36.08	1.17×10^{-6}	11
Run 22-3 rectangular pulse +3.933 gm moles/hr time response fit	-0.0161	8.03	47.63	1.95×10^{-6}	17
Run 22-3 rectangular pulse +3.933 gm moles/hr Bode plot fit	-0.0099	14.22	31.04		
Run 22-4 rectangular pulse -3.196 gm moles/hr time response fit	-0.0239	5.25	72.24	1.39×10^{-6}	15

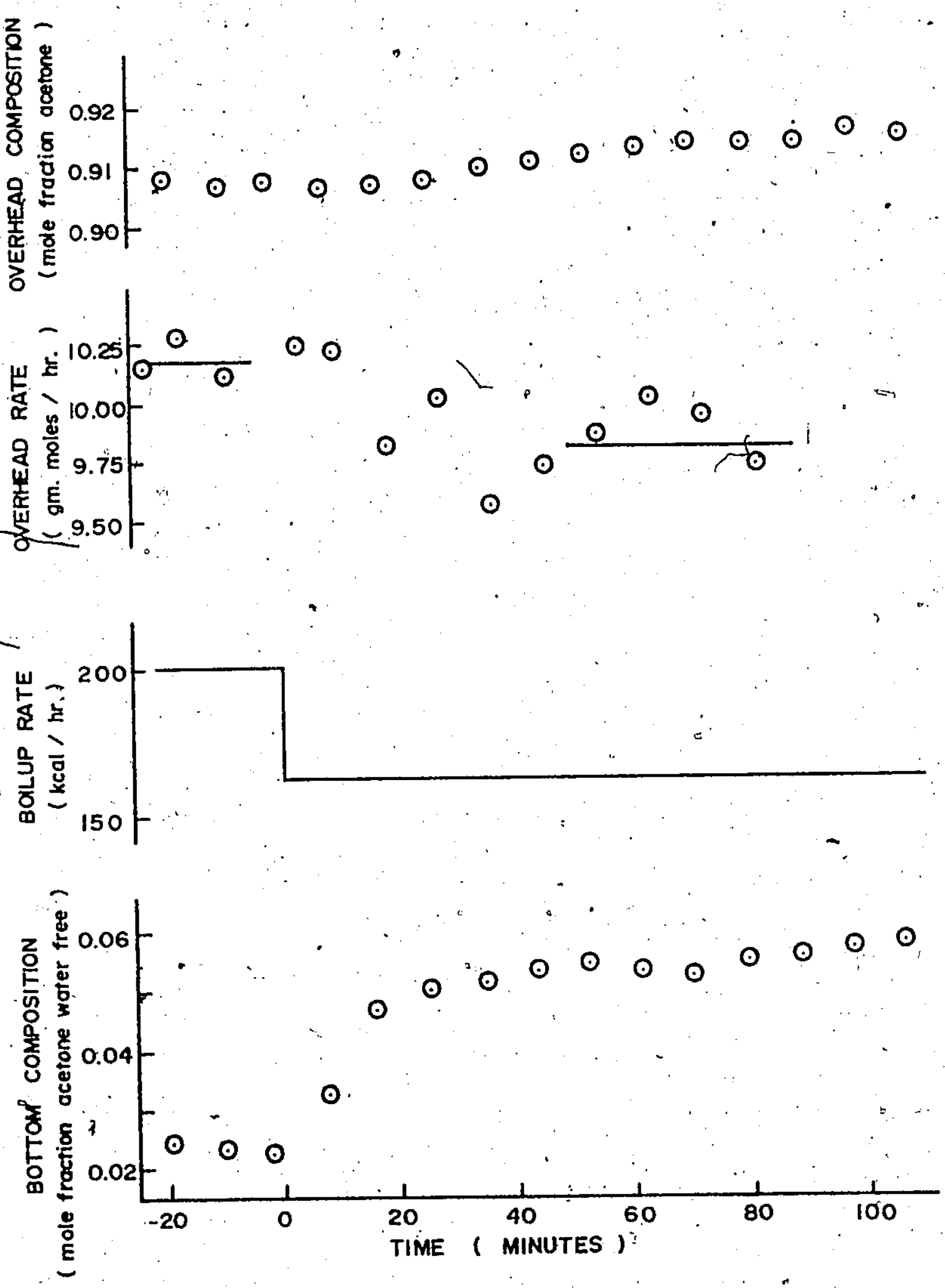
TABLE 6-7

Second Order Model Parameters for Overhead Composition Responses to Overhead Rate Changes

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Overhead product rate (gm moles/hr)}} = \frac{K\, e^{-\tau_d s}}{\tau^2 s^2 + 2\tau\xi s + 1}$$

Overhead Product Rate Change	K	τ_d	τ	ξ	Variance of residuals	Degrees of freedom
Run 22-2 step change -0.23 gm moles/hr time response fit	-0.0333	0.0	25.73	0.2870	7.40×10^{-7}	10
Run 22-3 rectangular pulse +3.933 gm moles/hr time response fit	-0.0136	1.42	19.94	0.9016	7.58×10^{-7}	16
Run 22-3 rectangular pulse +3.933 gm moles/hr Bode plot fit	-0.0099	1.33	21.62	0.7286	---	--

FIGURE 6-12 INTERNAL FLOW STEP RESPONSES
RUN 24-1



separation is reduced with a decrease in internal flow. This increases bottom product acetone concentration and decreases overhead product acetone composition. However, in Run 24-1 the overhead acetone composition increased because the overhead product rate decreased by 0.37 gm moles per hour. Experimental work to determine responses to internal flow rate changes was terminated because it was felt that overhead product rate could not be controlled manually to the required accuracy.

6.3 Appraisal of Experimental Results

From the three successful studies relating the product composition responses to input variable changes, it is concluded that a first order model with dead time satisfactorily represents the extractive distillation column. Recommended transfer functions are presented in Table 6-8. Second and higher order models are not significantly better due to the relatively large noise associated with the product composition responses.

Bottom product composition response is noisier than the overhead. This is largely due to poor mixing in the reboiler, resulting in unrepresentative sampling. However, tighter control of feed rate, feed temperature and reboiler liquid level would improve column frequency response results.

Determining the process transfer function model in the time domain using step changes proved satisfactory. This was due to the low order models fitted because process responses

TABLE 6-8

Recommended Transfer Functions Relating Product Compositions to Disturbance and Manipulated Variables

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Feed rate (gm moles/hr)}} = \frac{0.0198e^{-18.25s}}{28.39\ s+1}$$

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Feed composition (mole fraction acetone)}} = \frac{0.9985e^{-8.10s}}{39.21\ s+1}$$

$$\frac{\Delta \text{ Overhead composition (mole fraction acetone)}}{\Delta \text{ Overhead product rate (gm moles/hr)}} = \frac{0.0512e^{-13.74s}}{36.08\ s+1}$$

$$\frac{\Delta \text{ Bottom composition (mole fraction acetone, water free}}{\Delta \text{ Feed rate (gm moles/hr)}} = \frac{0.0098e^{-6.44s}}{7.28\ s+1}$$

$$\frac{\Delta \text{ Bottom composition (mole fraction acetone, water free}}{\Delta \text{ Feed composition (mole fraction acetone)}} = 0.076$$

$$\frac{\Delta \text{ Bottom composition (mole fraction acetone, water free)}}{\Delta \text{ Overhead rate (gm moles/hr)}} = 0.025$$

were relatively noisy. But, if response noise and sampling period are reduced, frequency response techniques are useful. High frequency characteristics of the process can be investigated only by pulse testing methods.

More work is required to fully understand the dynamic behaviour of the extractive distillation column. Non-linear effects due to different sized input disturbances were not determined. But experimental results indicated that process gains for pulse changes were smaller than those for step changes. Not only may the size of the change be important but also the direction. A comparison between Run 22-3 and 22-4 shows significant dynamic behaviour differences with input variables changed in opposite directions.

Overhead acetone concentration was affected strongly but bottom acetone composition weakly by changes in feed rate, feed composition and overhead rate. This was due to the steady state operating conditions of the column. Overhead product composition was normally at 0.90 mole fraction acetone. Therefore, changes in molar acetone feed could be translated to the overhead directly. Bottom product acetone composition would be more sensitive if the top section of the column was operated in more of a pinch region. However, tray priming will occur at high acetone concentrations in the overhead product. Therefore, tray modifications to prevent tray priming are required to increase bottom composition sensitivity.

This work has only presented a preliminary study into the dynamic behaviour of the extractive distillation column. More work is required to determine the process model and design a control strategy.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

Initial work in this project included rebuilding the fifteen stage extractive distillation column. Tray hole size and free area were critical design variables for column operation free of tray weeping. The column was operated at a wide range of operating levels to determine the operating range. Overhead product acetone concentration was limited to approximately 0.94 mole fraction due to liquid priming on tray 7.

A steady state simulation was developed to aid in a better understanding of the process. Column liquid and vapour samples were taken during experimental work and simulation results were matched by adjusting Murphree tray vapour efficiencies. These results indicated tray efficiencies were 75-90 per cent and reboiler efficiency approximately 150 per cent. These efficiencies were used in the simulation and predicted experimentally determined product compositions of unfitted experimental results with reasonable accuracy.

The dynamic behaviour of the column was studied by relating product composition responses to changes in disturbance and manipulated variables. Dyanmic behaviour was analysed by frequency response and real time response fitting. Pulse and step changes were made and low ordered transfer function models obtained. Experimental data was relatively noisy. First order transfer function models with dead time, fitted in the real time domain, were satisfactory. Pulse testing using frequency response techniques is superior in determining high order models with high frequency characteristics.

Reducing response noise and sampling interval is required to determine column dynamic behaviour with more accuracy. Tighter control of feed rate, feed temperature, reboiler liquid level and overhead product rate will reduce response noise.

The next major step in the extractive distillation project is to control the column product compositions using different levels of control methods. However, process measurements and controllers are required to implement computer control. A major effort is needed to develop an automatic on-line product and feed stream composition analyser. Instrumentation must be evaluated for this application and interfaced to the process and control computer. Measurement dead time associated with sample line flow and sample analysis must be minimized. Finally, instrumentation and computer interfacing is required to control overhead product rate and reboiler heat input.

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APPENDIX A

STREAM MEASUREMENTS

Process measurements included feed and solvent flow rates, column temperatures and stream composition analysis.

Feed and solvent flow rates were indicated by differential pressure cells measuring pressure drop across glass orifices. Measurements at specified flow rates changed slightly from run to run due to transmitter drift and traces of air in pressure tap lines. Therefore, flow calibrations were conducted before each run. Measurements were logged in analog-to-digital converter units at specified flows around the operating level. Flows were logged in analog-to-digital units during runs and converted to engineering units in subsequent data sorting and reduction.

Reboiler, condenser, feed stream and solvent stream temperatures were measured by thermocouples. These readings were converted to Celsius units and logged. Thermocouple transmitters were calibrated at three temperatures and fitted to the following equation,

$$T \text{ (deg. Celsius x 100)} = 518. + 2881.\, x - 44.\, x^2$$

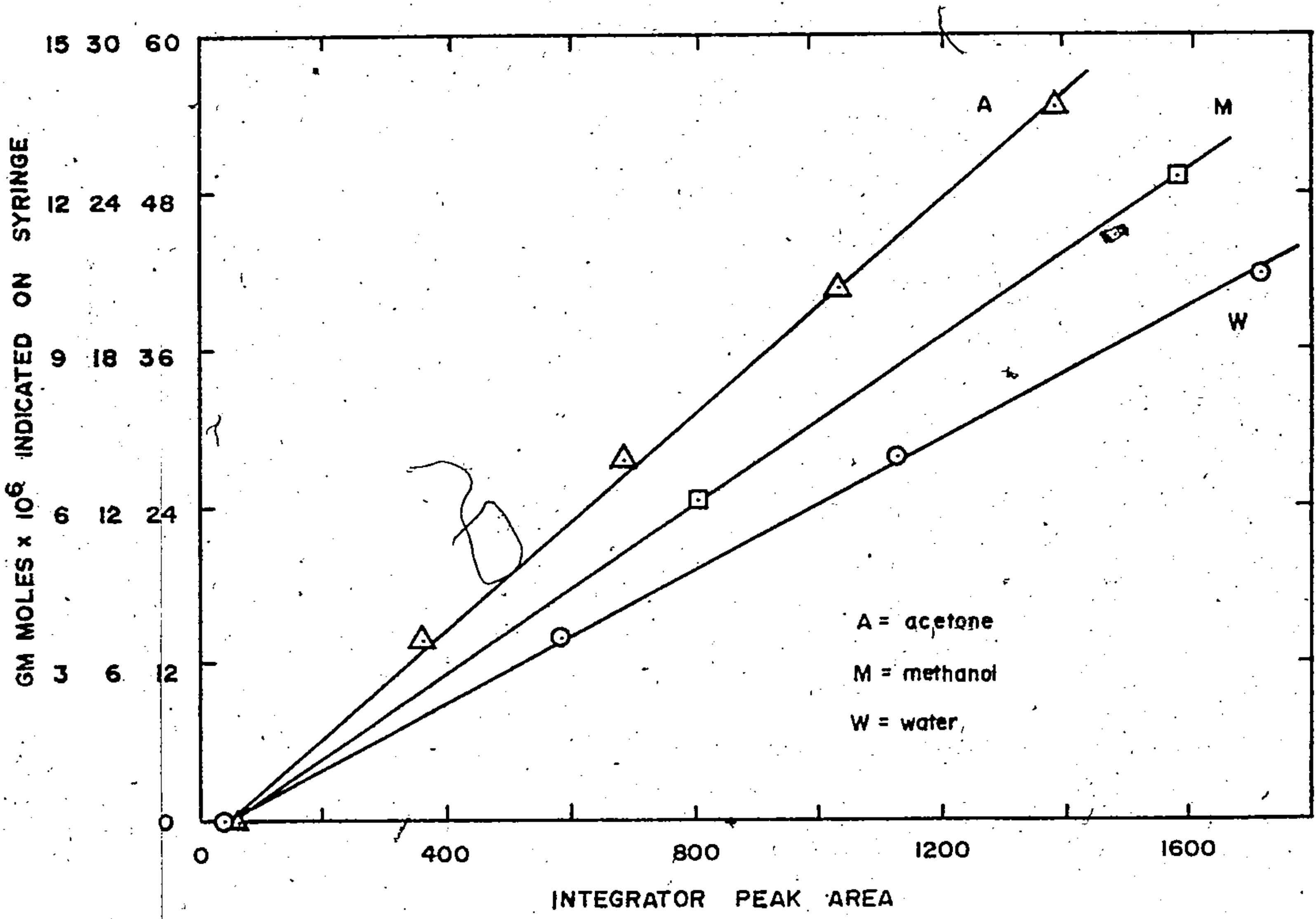
where x is the thermocouple A/D reading minus the reference thermocouple A/D reading.

Samples from the process were analysed with a Varian 90-P3 gas chromatograph and a Hewlett-Packard 3370B Integrator. Details for the chromatograph and integrator settings are given by Jackson (22). Initially, the chromatograph was calibrated by injecting 1 µl samples of known composition and plotting the number of moles of each component in each sample versus component peak area. However, the resulting calibration curves were non-linear, in contrast to the linear relationships generally obtained from thermal conductivity cells. Therefore, the calibration was repeated for each component by injecting pure samples of varying size and plotting the number of moles versus component peak area (Figure A-1). These results gave the expected linear behaviour, but when the syringe indicated no sample, an integrator peak area occurred. This is due to small amount of residual liquid inside the needle when the syringe was fully depressed. The residual liquid would be vapourized in the gas chromatograph sample injection port and produce a non-linear calibration when sample size was constant and composition varied.

Sample size reproducability was very important during gas chromatograph calibration. A clean syringe and air tight teflon seal around the plunger wire was required.

During routine sample analysis, a syringe holding the sample within the needle (Hamilton #7101) was preferred, rather than a syringe holding liquid within a glass barrel (Hamilton

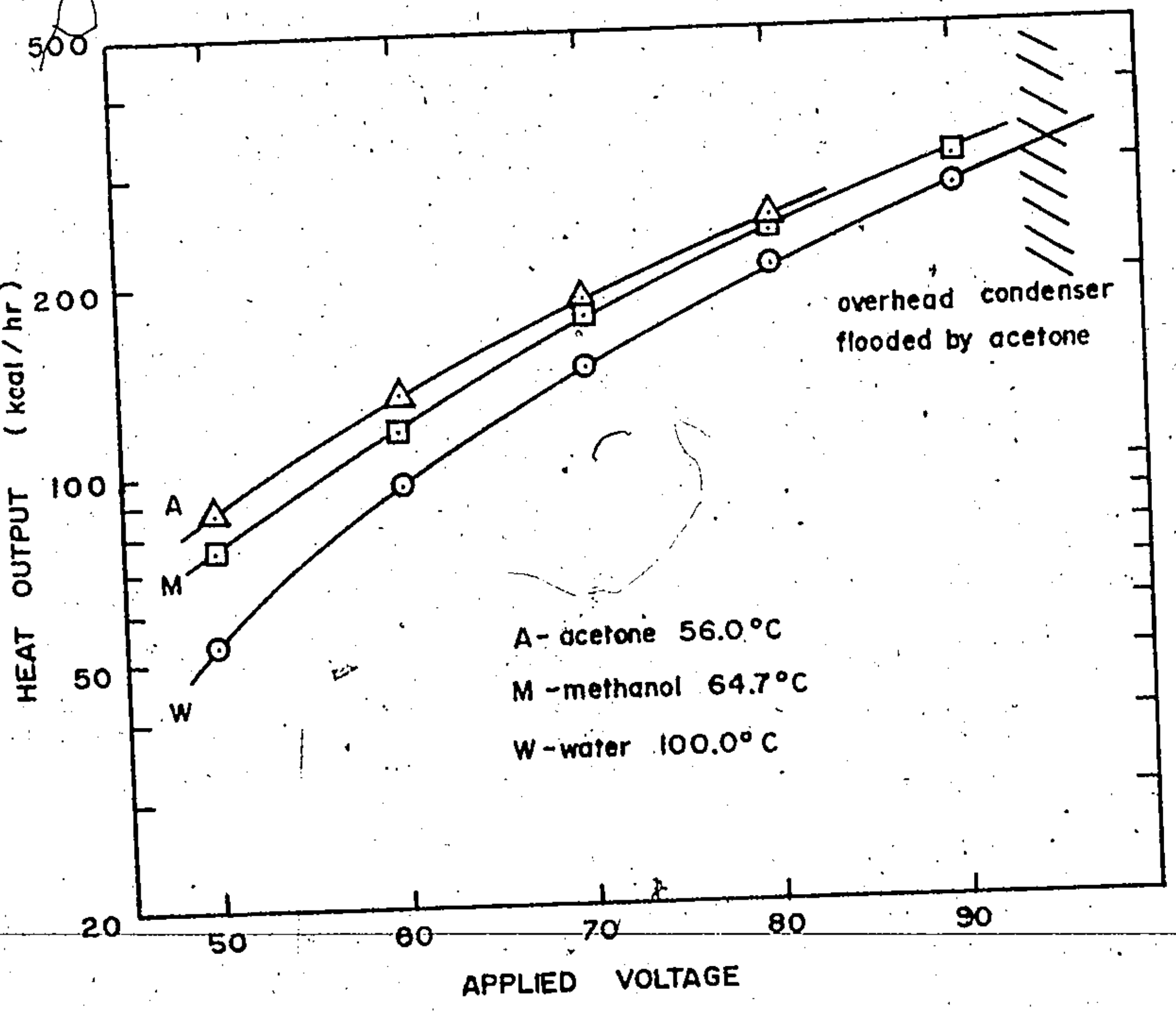
FIGURE A-I GAS CHROMATOGRAPH CALIBRATION CURVES USING PURE COMPONENTS AND VARIABLE SAMPLE SIZE



#701). Analysis with the latter syringe produced results higher in the more volatile components. Sample in the needle type syringe would completely vapourize but only a fraction of the liquid (richer in the more volatile components)would vapourize with the glass barrel type syringe.

Figure A-2 presents calibration of the electrically heated reboiler heat output as a function of applied voltage. The condenser was attached directly to the reboiler during these tests. Pure solutions of acetone, methanol and water were boiled at constant applied voltages and the output vapour condensed and measured. Separate calibration curves were obtained for each component and differed by as much as 20 per cent. This behaviour is likely due to different reboiler heat losses. Acetone, with the lowest reboiler temperature, corresponded to the highest measured heat output.

FIGURE A-2 REBOILER HEAT OUTPUT AS A FUNCTION OF APPLIED VOLTAGE



APPENDIX B

STEADY STATE SIMULATION FLOW CHARTS AND LISTINGS

Figures B-1 to B-4 are logic flow diagrams for the steady state simulation of the extractive distillation column. Program TOWER is the main line program. Data is initially read and variables initialized. This program calculates component mass flow rates using the tri-diagonal matrix form of the mass balance equations. A general form of these equations is presented by Boston (4). The specific form for the extractive distillation column (feed and solvent streams, and liquid product streams) is given below:

$$(1 + W_{L1}/L_1)\, l_1 - S_2 l_2 = f_1 \qquad \ldots\ldots(1)$$

$$-l_{n-1} + (1 + S_n) l_n - S_{n+1} l_{n+1} = f_n \quad (n=2,\ 3,\ \ldots,\ N-1)$$

$$-l_{N-1} + (1 + S_N) l_N = f_N$$

where $S_n = E_n K_n V_n / L_n$

W_{L1} = total overhead molar product rate

L_n = total tray (n) molar liquid rate

V_n = total tray (n) molar vapour rate

K_n = equilibrium ratio

E_n = vapour efficiency

l_n = tray (n) molar component liquid rate

f_n = component molar feed rate to tray (n)

(non zero for feed and solvent trays)

Equations (1) are solved for each of the acetone, methanol and water components by the modified Thomas method (4). Equilibrium relationships (K_n) are called from subroutine KVALUE and enthalpy balance provided by either subroutine ENBAL 1 or ENBAL 2. Convergence of tray temperatures and vapour rates are promoted by subroutine CVGP. Program TOWER checks the difference between the calculated total liquid flows and required liquid flows and if they are within a specified tolerance the calculations are terminated. Final column conditions are printed and punched onto cards. However, if the error in liquid flows are outside the tolerance, mass and energy balance calculations are continued.

In the flow diagrams, each logic step has been associated with numbered lines in the program listings. The listings have been spiced with comment cards describing the program. Required input data and its format is described at the beginning of program TOWER.

FIGURE B-1 Program TOWER

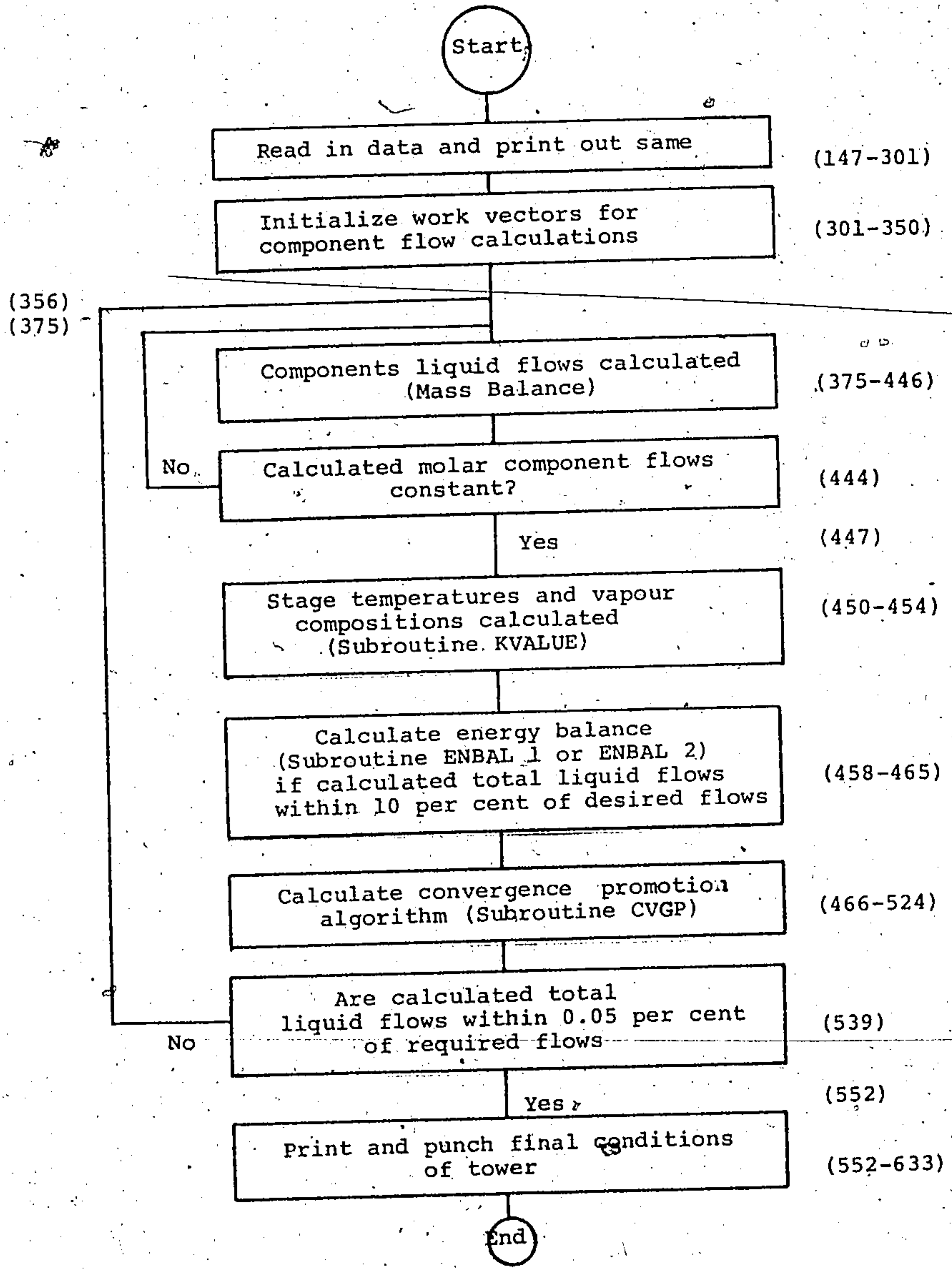


FIGURE B-2

SUBROUTINE KVALUE

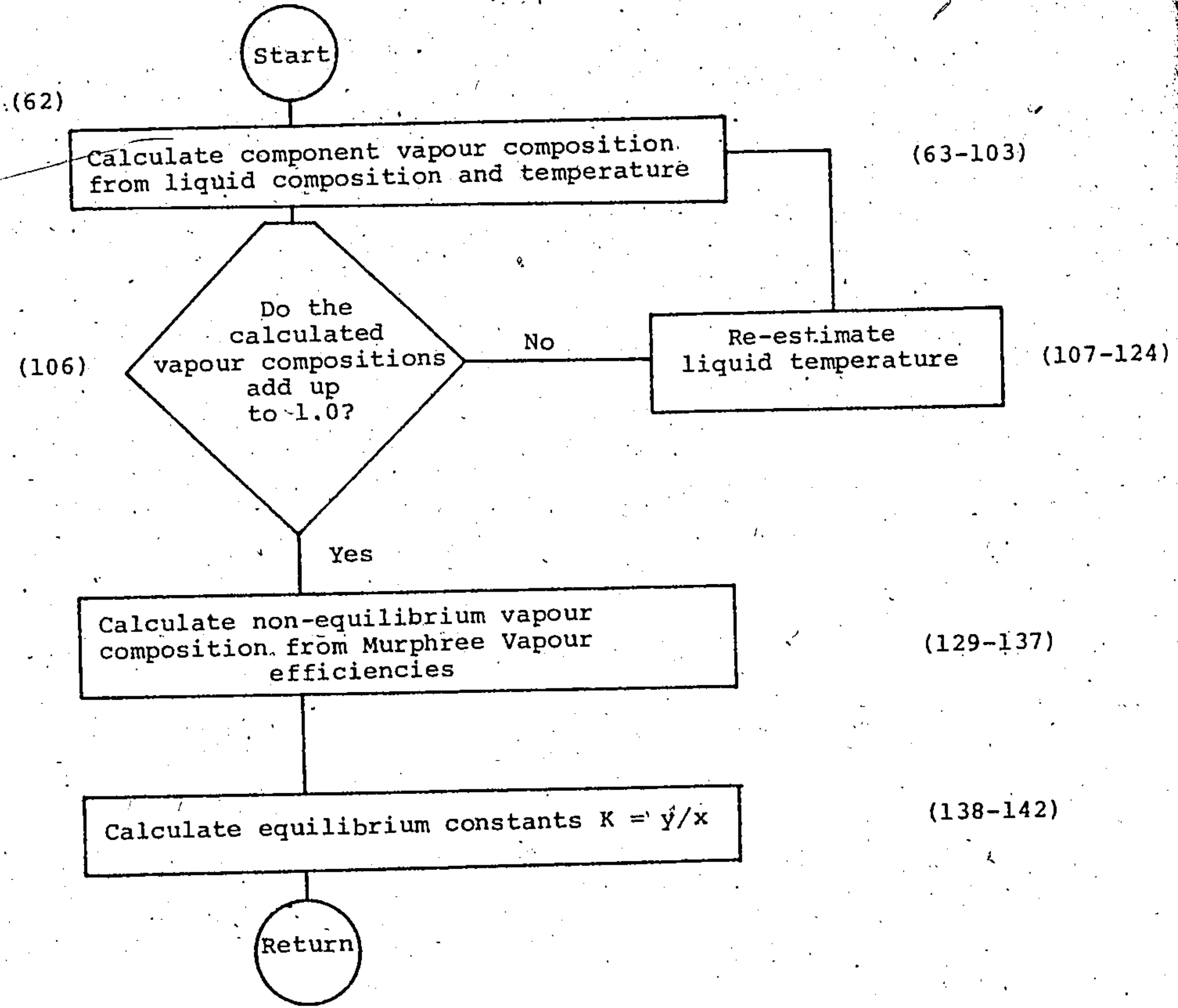
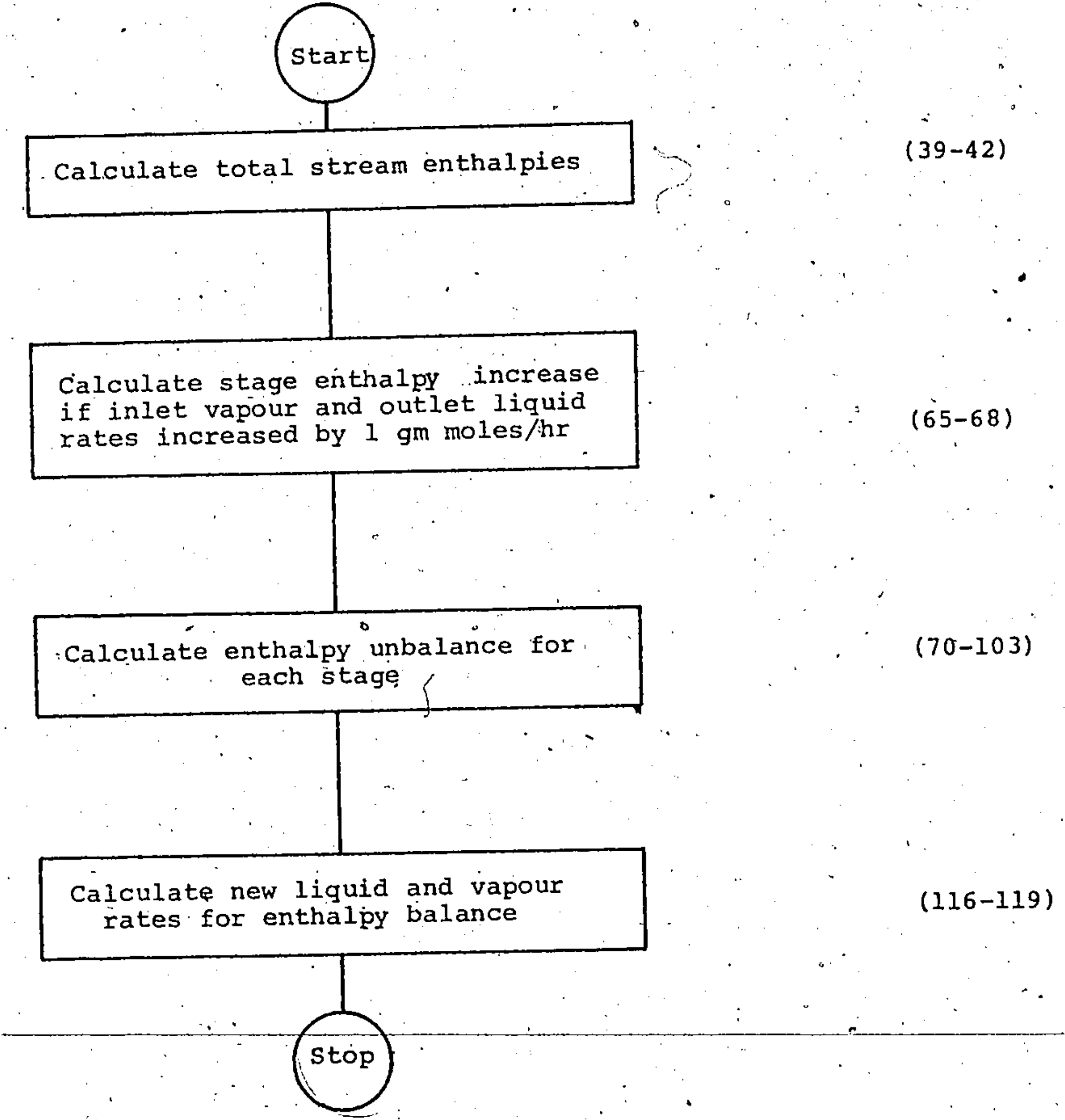


FIGURE B-3

SUBROUTINE ENBAL 1



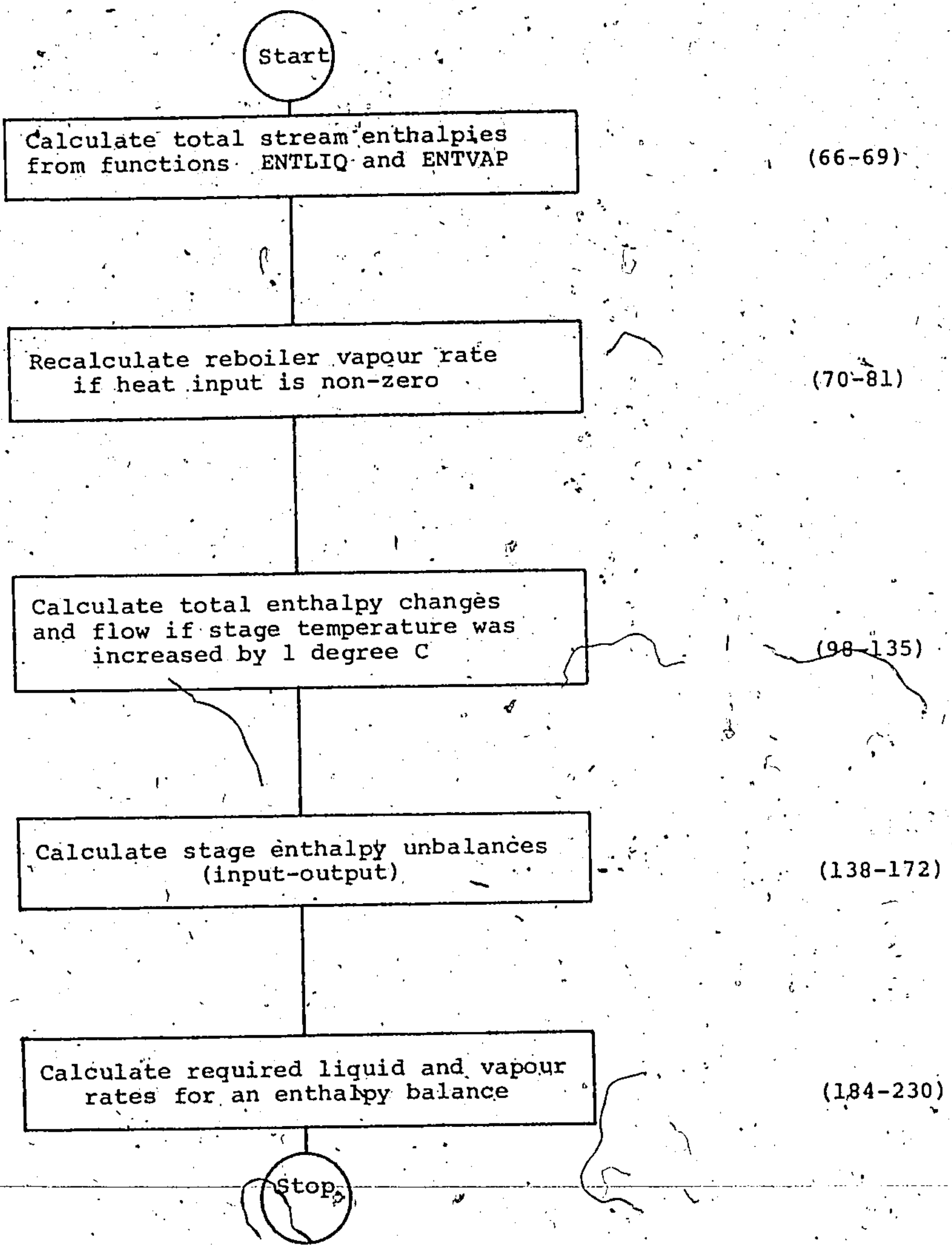


FIGURE B-4 Subroutine ENBAL 2

```
      PROGRAM TOWER(INPUT,OUTPUT,PUNCH)
C
C***********************************************************************
C
C          STEADY STATE SIMULATION OF AN EXTRACTIVE DISTILLATION TOWER
C          SEPERATING ACETONE-METHANOL USING WATER
C
C          WRITTEN MARCH-AUGUST 1974 BY D.L. ONEILL.
C
C          REQUIRE EQUIPMENT PARAMETERS, FEED CONDITIONS, REFLUX RATIO
C          AND EITHER (1) OVERHEAD PRODUCT RATE OR
C          (2) REBOILER HEAT INPUT (BOILUP RATE)
C
C          GENERAL METHOD - SUCCESSIVE APPROXIMATION AS DESCRIBED BY
C          C.J. KING, *SEPARATION PROCESSES*, CHPT 11
C          MASS BALANCE BY SOLUTION OF TRIDIAGONAL MATRIX
C          AS DESCRIBED BY J.F. BOSTON AND S.L. SULLIVAN, C.J.C.E.
C          VOL 50, OCT. 1972 PG 663
C          ENTHALPY BALANCE AS DESCRIBED BY C.J. KING FOR CONSTANT PRODUCT
C          ENTHALPY BALANCE AS DESCRIBED BY D.N. HANSON,METHOD III
C          *COMUTATION OF MULTISTAGE SEPERATION PROCESSES*
C          FOR REBOILER HEAT INPUT CASE
C          CONVERGENCE IS PROMOTED BY DOMINANT EIGENVALUE METHOD AS
C          MODIFIED BY MASATOSHI NISHIO
C***********************************************************************
C
C      DATA INPUT
C
C***********************************************************************
C
C   CARD NUMBER                     VARIABLES                         FORMAT
C
C  1    FLAG FOR CONSTANT OVERHEAD RATE ( CONSTANT PRODUCTS )          5A4
C       OR REBOILER HEAT INPUT (FLOATING PRODUCT)
C  2                          TITLE1                                   18A4
C  3                          TITLE2                                   18A4
C  4                          TITLE3                                   18A4
C  5    NO. OF COMPONENTS, SOLVENT STAGE NO., FEED STAGE NO.
C       TOTAL NO. OF STAGES ( STAGES NO. FROM CONDENSER TO REBOILER  4I3
C
C  6    SOLVENT RATE,TEMP,FRACTION VAPOUR,ACETONE MOLE FRACTION 5G15.4
C       ,SOLVENT METHANOL MOL FRACTION
C  7    SOLVENT WATER MOLE FRACTION                                    1G15.4
C
C  8    FEED RATE,TEMP,FRACTION VAPOUR,ACETONE MOL FRACT,              5G15.4
C       FEED METHANOL MOLE FRACTION
C  9    FEED WATER MOLE FRACTION                                       1G15.4
C
C 10    *ISEF* FLAG,CONDENSER TEMP, REBOILER TEMP           I2,G13.4,G15.4
C       IF *ISEF* FLAG IS 0 TOWER STAGE TEMP TAKEN AS LINEAR BETWEEN
C       CONDENSER AND REBOILER
C       IF *ISEE* FLAG NOT 0 INDIVIDUAL STAGE TEMP,ARE READ NEXT
C 10A,B,C..  STAGE TEMPERATURES FROM TOP TO BOTTOM                   5G15.4
C
C 11    *ISEF* FLAG,CONDENSER PRESSURE,REBOILER PRESSURE  I2,G13.4,G15.4
C       IF *ISEF* IS 0 TOWER STAGE PRESSURE TAKEN AS LINEAR BETWEEN
C       CONDENSER AND REBOILER
C       IF *ISEF* IS NOT 0 INDIVIDUAL STAGE PRESSURES ARE READ NEXT
C 11A,B,C..      STAGE PRESSURES FROM TOP TO BOTTOM                  5G15.4
```

```
C
C 12    *ISEE* FLAG,REFLUX RATIO,REBOILER VAPOUR RATE,
C       REBOILER HEAT INPUT                                  I2,G12.4,G15.4
C       IF *ISEE* FLAG IS 0 REBOILER VAPOUR RATE IS USED FOR ALL STAGES
C       AND LIQUID RATES ARE CALCULATED BY OVERALL MASS BALANCE
C       IF *ISEE* FLAG IS NOT 0 INDIVIDUAL STAGE VAPOUR RATES AND LIQUID
C       RATES ARE READ NEXT
C 12A,B,C..     STAGE VAPOUR RATES FROM TOP TO BOTTOM                5G15.4
C 12E,F,G..     STAGE LIQUID RATES FROM TOP TO BOTTOM                5G15.4
C       NOTE- IF INDIVIDUAL VAPOUR AND LIQUID RATES ARE READ IN THEY
C       MUST KEEP EACH STAGE IN TOTAL MASS BALANCE + OR - 0.005 GM MOL/HR
C       IF THERE IS NOT A MASS BALANCE THE SIMULATION WILL NOT CONVERGE
C       TO A CASE WITH A MASS BALANCE
C
C 13    MURPHREE VAPOUR EFFICIENCIES FOR SECTIONS TOP,MIDDLE,BOTTOM AND
C       REBOILER
C 13A   METHANOL EFFICIENCY                                          5G15.4
C 13B   WATER EFFICIENCY                                             5G15.4
C         ACETONE EFFICIENCY IS NOT REQUIRED BECAUSE ONLY TWO
C         EFFICIENCIES ARE INDEPENDANT
C
C 14A,B,C..  LIQUID COMPOSITIONS (ACETONE,METHANOL,WATER) FOR EACH STAGE
C       FROM CONDENSER TO REBOILER                                   3G15.4
C
C**************************************************************************
C
C       DEFINITION OF IMPORTANT VARIABLES
C
C**************************************************************************
C
C  BL(I)  TOTAL LIQUID FLOW FROM STAGE(I) (GM MOLES/HR)
C  BOILUP HEAT INPUT TO REBOILER  (KCAL/HR)
C  BV(I)  TOTAL VAPOUR FLOW FROM STAGE(I)  (GM MOLES/HR)
C  CONDUT CONDENSER DUTY  (KCAL/HR)
C  E(I,J) MURPHREE VAPOUR EFFICIENCY FOR STAGE(I),COMPONENT(J)
C  EK(I,J) EQUILIBRIUM CONSTANT K=Y/X FOR STAGE(I),COMPONENT(J)
C  ERROR  LARGEST FRACTION ERROR BETWEEN CALCULATED AND DESIRED TOTAL
C         LIQUID STAGE FLOW
C  ERROR2 LARGEST FRACTION DIFFERENCE BETWEEN TOTAL LIQUID STAGE FLOWS I
C         SUCCESIVE COMPONENT MOLAR FLOW CALCULATIONS
C  FEDC(J) FEED STREAM COMPOSITION FOR COMPONENT(J) (MOLE FRAC)
C  FEDFRV FEED STREAM FLOW FRACTION VAPOUR
C  FEDR   FEED STREAM FLOW RATE (GM MOLE/HR)
C  FEDT   FEED STREAM TEMPERATURE  (DEG. C)
C  ICFLAG CONVERGENCE CONDITION FLAG
C         =0 IF ERROR GREATER THAN 0.005 (CONVERGENCE CALCULATION CONTINU
C         =1 IF ERROR LESS THAN 0.005 CASE IS TERMINATED
C  IEFLAG ENERGY CONDITION FLAG
C         =0 IF ERROR GREATER THAN 0.1 NO ENERGY BALANCE PERFORMED
C         =1 IF ERROR LESS THAN 0.1 ENERGY BALANCE PERFORMED
C  IF     FEED STAGE NO. (NUMBERED FROM TOP OF COLUMN INCLUDING CONDENSE
C  ILOOP  MAIN ITERATION LOOP NO.(AROUND MASS AND ENERGY BALANCE CALCUL)
C  ILOOP2 LOOP NO. FOR MASS BALANCE CONVERGENCE CALCULATION
C  ILOOP3 NO. OF ITERATIONS COMPLETED WITH ENERGY BALANCE CALCULATED
C  INDVAR NO. OF INDEPENDENT VARIABLES THAT VARY,VAPOUR RATE AND TEMP
C         FOR EACH STAGE EXCEPT TOTAL CONDENSER
C  IS     SOLVENT FEED STAGE NO.,STAGES NO. FROM TOP INCLUDING CONDEN
C  ISEE   FLAG TO INDICATE IF MORE INFORMATION IS TO BE READ IN
```

```
C   IT      TOTAL NO. OF STAGES (INCLUDES TRAYS,CONDENSER AND REBOILER)
C   NC      TOTAL NO. OF COMPONENTS- COMPONENT(1)ACETONE (2)METHANOL (3)WA
C   P(I)    PRESSURE STAGE(I)          (MM HG)
C   REBDUT  REBOILER DUTY  (KCAL/HR)
C   RR      REFLUX RATIO
C   SL(I,J) COMPONENT MOLAR FLOWS FOR STAGE(I),COMPONENT(J)  (GM MOL/HR)
C   SOVC(J) SOLVENT FEED COMPOSITION FOR COMPONENT(J)   (MOLE FRACTION)
C   SOVFRV  SOLVENT FRACTION VAPOUR
C   SOVR    SOLVENT FEED RATE (GM MOLES/HR)
C   SOVT    SOLVENT FEED TEMP  (DEG. C)
C   T(I)    STAGE(I) TEMPERATURE  (DEG. C)
C   VAR     INDEPENT VARIABLES, VAPOUR RATE AND TEMPERATURE
C   X(I,J)  LIQUID COMPOSITION STAGE(I),COMPONENT(J) (MOLE FRACTION)
C   Y(I,J)  VAPOUR COMPOSITION STAGE(I),COMPONENT(J) (MOLE FRACTION)
C
C
C
C*********************************************************************
      COMMON /BLK1/ BV(20),BL(20),T(20),P(20),SL(20,3),IT,NC,IS,IF,RR
      COMMON /BLK2/ SB(20,3),SC(20,3),SPP(20,3),SFP(20,3),SCP(20,3)
      COMMON /BLK3/ IEFLAG,ICFLAG,ILOOP
      COMMON /BLK4/ X(20,3),Y(20,3),EK(3),E(20,3)
      COMMON /BLK5/ WL(20),WV(20),SF(20,3),BOILUP,SOVRV,SOVRL,FEDRV,FEDR
     1L,FEDC(3),SOVC(3),SOVT,FEDT,CONDUT,REBDUT
      DIMENSION TITLE1(18), TITLE2(18), TITLE3(18), TBL2(20), SEPDUT(3),
     1 RL(20), RV(20), QL(20), QV(20), S(20,3), SPDP(20,3), TBL(20), VAR
     22(40), SEPDU1(3), ISET(5)
C
C*********************************************************************
C
C     READ IN DATA AND PRINT OUT SAME
C
      PRINT 1
1     FORMAT (*1*,//,5X,*EXTRACTIVE DISTILLATION SIMULATION OF ACETONE,
     1METHANOL, WATER SYSTEM*,//)
C
C     READ FLAG FOR EITHER CONSTANT OVERHEAD RATE (*CONSTANT PRODUCTS*)
C     OR REBOILER DUTY CONSTANT ( OVERHEAD RATE FLOATS
C     FLOATING PRODUCTS* )
      READ 2, ISET
2     FORMAT (5A4)
      PRINT 3, ISET
3     FORMAT (* *,5X,5A4,//)
C
C         3 TITLE CARDS
      READ 4, TITLE1,TITLE2,TITLE3
4     FORMAT (18A4)
      PRINT 5, TITLE1,TITLE2,TITLE3
5     FORMAT (* *,18A4,2(//,1X,18A4))
C
C         NUMBER OF COMPONENTS, SOLVENT STAGE NO., FEED STAGE NO.,
C         AND TOTAL NO. OF STAGES
      READ 6, NC,IS,IF,IT
6     FORMAT (4I3)
      PRINT 7, NC,IS,IF,IT
7     FORMAT (* *,/,5X,*COMPONENTS*,I11,/,5X,*SOLVENT TRAY NO.*,I5,/,5X,
     1*FEED TRAY NO.*,I8,/,5X,*TOTAL NO. OF TRAYS*,I3)
      ISM1=IS-1
```

```
      ISP1=IS+1
      IFM1=IF-1
      IFP1=IF+1
      ITM1=IT-1
C
C        SOLVENT RATE TEMP, FRACTION VAPOUR AND COMPOSITIONS
      READ 8, SOVR,SOVT,SOVFRV,(SOVC(I),I=1,NC)
C        FEED RATE TEMP, FRACTION VAPOUR AND COMPOSITIONS
      READ 8, FEDR,FEDT,FEDFRV,(FEDC(I),I=1,NC)
 8    FORMAT (5G15.4)
      SOVRV=SOVR*SOVFRV
      SOVRL=SOVR-SOVRV
      FEDRV=FEDR*FEDFRV
      FEDRL=FEDR-FEDRV
      PRINT 9
 9    FORMAT (* *,//,5X,*SOLVENT STREAM CONDITIONS (FLOW,TEMP,FRACTION V
     1APOUR,COMP)*)
      PRINT 11, SOVR,SOVT,SOVFRV,(SOVC(I),I=1,NC)
      PRINT 10
 10   FORMAT (* *,//,5X,*FEED STREAM CONDITIONS (FLOW,TEMP,FRACTION VAPO
     1UR,COMP)*)
      PRINT 11, FEDR,FEDT,FEDFRV,(FEDC(I),I=1,NC)
 11   FORMAT (* *,/,10(5X,5G16.5,/))
C
C        READ TEMPERATURES
      READ 12, ISEF,T(1),T(IT)
 12   FORMAT (I2,G13.4,2G15.4)
      IF (ISEF.EQ.0) GO TO 13
C        READ ALL TEMPERATURES IF ISEF IS 1
      READ 8, (T(I),I=1,IT)
      GO TO 15
C     SET TEMP TO LINEAR INTERPOLATION IF *ISFE* IS 0
 13   DO 14 I=1,IT
      T(I)=T(1)+(T(IT)-T(1))/FLOAT(IT-1)*FLOAT(I-1)
 14   CONTINUE
 15   PRINT 16
 16   FORMAT (* *,//,5X,*TRAY TEMPERATURES (DEG. C)*)
      PRINT 11, (T(I),I=1,IT)
C
C        READ PRESSURES
      READ 12, ISEF,P(1),P(IT)
      IF (ISEE.EQ.0) GO TO 17
C        READ ALL PRESSURES IF ISEE IS 1
      READ 8, (P(I),I=1,IT)
      GO TO 19
C     SET PRESSURE TO LINEAR INTERPOLATION IF *ISEF* IS 0
 17   DO 18 I=1,IT
      P(I)=P(1)+(P(IT)-P(1))/FLOAT(IT-1)*FLOAT(I-1)
 18   CONTINUE
 19   PRINT 20
 20   FORMAT (* *,//,5X,*TRAY PRESSURES (MM HG)*)
      PRINT 11, (P(I),I=1,IT)
C
C        READ ISEE , REFLUX RATIO, BOILUP RATE AND REBOILER HEAT INPUT
      READ 12, ISEF,RR,BV(IT),BOILUP
      IF (ISEF.EQ.0) GO TO 21
C        IF ISEF IS 1 READ STAGE VAPOUR AND LIQUID RATES
      READ 8, (BV(I),I=1,IT)
```

```
      READ 8, (BL(I),I=1,IT)
      RR=BL(1)/(BV(2)-BL(1))
      GO TO 28
C        IF ISEE IS [illegible] ALL VAPOUR RATES EQUAL AND TRAY MASS BALANCE
C          CALCULATED
21    DO 22 I=1,IT
      BV(I)=BV(IT)
22    CONTINUE
      DO 23 I=1,IFM1
      BV(I)=BV(I)+FEDBV
23    CONTINUE
      DO 24 I=1,ISM1
      BL(1)=RR*BV(2)/(RR+1.0)
      BV(I)=BV(I)+SOVBV
24    CONTINUE
C    TOTAL CONDENSER
      BV(1)=0.0
      DO 25 I=2,ISM1
      BL(I)=BL(1)
25    CONTINUE
      DO 26 I=IS,IFM1
      BL(I)=BL(1)+SOVBL
26    CONTINUE
      DO 27 I=IF,ITM1
      BL(I)=BL(1)+SOVBL+FEDBL
27    CONTINUE
      BL(IT)=BL(ITM1)-BV(IT)
28    PRINT 29
29    FORMAT (* *,//,5X,*REFLUX RATIO , BOILUP RATE , REBOILER DUTY*)
      PRINT 11, RR,BV(IT),BOILUP
      PRINT 30
30    FORMAT (* *,//,5X,*MOLAR VAPOUR FLOWS*)
      PRINT 11, (BV(I),I=1,IT)
      PRINT 31
31    FORMAT (* *,//,5X,*MOLAR LIQUID FLOWS*)
      PRINT 11, (BL(I),I=1,IT)
C
C          STAGE/ MURPHREE VAPOUR EFFICIENCES
      DO 32 J=2,NC
      READ 8, E(1,J),E(IS,J),E(IF,J),E(IT,J)
32    CONTINUE
      DO 33 I=2,ISM1
      DO 33 J=2,NC
33    E(I,J)=E(1,J)
      DO 34 I=ISP1,IFM1
      DO 34 J=2,NC
34    E(I,J)=E(IS,J)
      DO 35 I=IFP1,ITM1
      DO 35 J=2,NC
35    E(I,J)=E(IF,J)
      PRINT 36
36    FORMAT (* *,//,5X,*MURPHEE PLATE LIQUID EFFICIENCIES*)
      DO 37 J=1,NC
      EK(J)=0.0
37    CONTINUE
      DO 38 J=2,NC
      PRINT 11, (E(I,J),I=1,IT)
38    CONTINUE
```

```
C
C          INITIAL APPROXIMATION OF LIQUID COMPOSITIONS
      DO 39 I=1,IT
      READ 8, (X(I,J),J=1,NC)
39    CONTINUE
      PRINT 40
40    FORMAT (* *,//,5X,*INITIAL ESTIMATE OF LIQUID COMPOSITIONS*)
      DO 41 I=1,IT
      PRINT 11, (X(I,J),J=1,NC)
41    CONTINUE
C
C*************************************************************************
C
C   MASS BALANCE SECTION FROM J. BOSTONS 'AN IMPROVED ALOGRITHM FOR
C     SOLVING MASS BALANCE EQ'N' C.J.CH.E. VOL50, OCT,1972
C*************************************************************************
C
C     INITIALIZATION OF INPUT AND OUTPUT STREAMS
C
C          ZERO FEED STREAM VECTOR (SF) , LIQUID DRAWOFF (VL)
C          AND VAPOUR DRAWOFF (WV) -NOMINCATURE FOLLOWS THAT OF ARTICLE
C          BY BOSTON
      DO 43 I=1,IT
      DO 42 J=1,NC
      SF(I,J)=0.0
42    CONTINUE
      WL(I)=WV(I)=0.0
43    CONTINUE
C   LIQUID DRAWOFF WL
C   NO VAPOUR DRAWOFF, NO INTERMEDIATE DRAWOFF
      WL(1)=BV(2)-BL(1)-FV(1)
      DO 44 I=1,IT
      QL(I)=WL(I)/BL(I)
      QV(I)=WV(I)/BV(I)
44    CONTINUE
C          FEED STREAM VECTORS SET FOR FEED AND SOLVENT FEED
      DO 45 I=1,NC
      SF(IF,I)=FEDC(I)*FEDR*(1.0-FEDFRV)
      SF(IS,I)=SOVC(I)*SOVR*(1.0-SOVFRV)
      SF(IF-1,I)=FEDC(I)*FEDR*FEDFRV
      SF(IS-1,I)=SOVC(I)*SOVR*SOVFRV
45    CONTINUE
      DO 46 I=2,ITM1
      RL(I)=1.0+WL(I)/BL(I)
      RV(I)=1.0+WV(I)/BV(I)
46    CONTINUE
      RL(1)=1.0+WL(1)/BL(1)
      RV(IT)=1.0+WV(IT)/BV(IT)
      DO 47 I=1,IT
      TBL(I)=BL(I)
47    CONTINUE
      IEFLAG=ICFLAG=0
      PRINT 48
48    FORMAT (*1*)
C
C          INDVAR - NO. OF INDEPENDANT VARIABLES
      INDVAR=2*(IT-1)
      IF (BOILUP.EQ.0.0) INDVAR=INDVAR-1
```

```
      ILOOP3=1
C
C*****************************************************************************
C
C         MAIN ITERATION LOOP FOR MASS AND ENTHALPY BALANCE
C
      DO 80 ILOOP=1,30
      PRINT 49, ILOOP
49    FORMAT (*1*,//,1X,*XXX  LOOP NUMBER*,I4,*  XXXXXXXXXXXXXXXXXXXXXX
     1 XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX*,///)
C  LIQUID OVERHEAD PRODUCT CHANGES DUE TO ENERGY BALANCE
      WL(1)=BV(2)-BL(1)-BV(1)
      QL(1)=WL(1)/BL(1)
      RL(1)=1.0+WL(1)/BL(1)
C        ITERATION LOOP FOR CALCULATION OF NEW LIQUID COMPONENT FLOWS
C        THIS IS REQUIRED BECAUSE EQUILIBRIUM IS A FUNCTION OF LIQUID
C        COMPOSITION AS WELL AS TEMPERATURE
C     IF HIGH ACETONE CONCENTRATIONS IN OVERHEAD PRODUCT GREATER THAN
C     94 PER CENT, THE UPPER LIMIT FOR ILOOP2 SHOULD BE INCREASED
C     FROM 5
C     UPPER LIMIT OF 5 WORKS WITH THE PARTICULAR TOWER CONDITIONS AT
C     PRESENT BUT WITH CASES WITH OVERHEAD PRODUCT GREATER THAN 94 PER
C     CENT THE CASE WOULD NOT CONVERGE BECAUSE CALCULATED MOLAR
C     COMPONENT FLOWS WOULD STILL CHANGE AFTER 5 CALCULATIONS IN
C     THIS LOOP
      DO 66 ILOOP2=1,5
      DO 50 I=1,IT
      TBL2(I)=TBL(I)
50    CONTINUE
C        CALCULATE VALUE OF K (Y/X)
      DO 51 J1=1,IT
      I=IT+1-J1
      CALL KVALUE (X,Y,EK,E,T(I),P(I),I,1)
      DO 51 J=1,NC
51    S(I,J)=EK(J)*BV(I)/BL(I)
C    MAIN LOOP TO GET INDIVIDUAL COMPONENT FLOWS
      PRINT 52
52    FORMAT (* *,//,5X,*COMPONENT MOLAR FLOWS*)
      DO 58 KJ=1,NC
      SB(1,KJ)=RL(1)+S(1,KJ)
      DO 53 I=2,ITM1
      SB(I,KJ)=RL(I)+RV(I)*S(I,KJ)
53    CONTINUE
      SB(IT,KJ)=1.0+RV(IT)*S(IT,KJ)
      DO 54 I=1,ITM1
      SC(I,KJ)=S(I+1,KJ)
54    CONTINUE
      SBDP(1,KJ)=S(1,KJ)+QL(1)
      SBP(1,KJ)=1.0+SBDP(1,KJ)
      DO 55 I=2,IT
      SBDP(I,KJ)=S(I,KJ)*(RV(I)*SBDP(I-1,KJ)+QV(I))/SBP(I-1,KJ)+QL(I)
      SBP(I,KJ)=1.0+SBDP(I,KJ)
55    CONTINUE
      SFP(1,KJ)=SF(1,KJ)/SBP(1,KJ)
      DO 56 I=2,IT
      SCP(I-1,KJ)=SC(I-1,KJ)/SBP(I-1,KJ)
      SFP(I,KJ)=(SF(I,KJ)+SFP(I-1,KJ))/SBP(I,KJ)
56    CONTINUE
```

```
C          SL CALCULATED - COMPONENT MOLAR FLOWS'
        SL(IT,KJ)=SFP(IT,KJ)
        DO 57 J=1,ITM1
        I=IT-J
        SL(I,KJ)=SFP(I,KJ)+SCP(I,KJ)*SL(I+1,KJ)
 57     CONTINUE
C      PRINT COMPONENT MOLAR FLOWS
        PRINT 11, (SL(I,KJ),I=1,IT)
 58     CONTINUE
C          TOTAL LIQUID FLOW CALCULATED
        DO 61 I=1,IT
        TBL(I)=0.0
        DO 59 KJ=1,NC
        TBL(I)=TBL(I)+SL(I,KJ)
 59     CONTINUE
C          NEW LIQUID COMPOSITIONS CALCULATED
        DO 60 KJ=1,NC
        X(I,KJ)=SL(I,KJ)/TBL(I)
 60     CONTINUE
 61     CONTINUE
        PRINT 62
 62     FORMAT (* *,//,5X,*TOTAL LIQUID FLOW CALCULATED*)
        PRINT 11, (TBL(I),I=1,IT)
        PRINT 63
 63     FORMAT (* *,//,5X,*LIQUID MOLE FRACTIONS*)
        DO 64 KJ=1,NC
        PRINT 11, (X(I,KJ),I=1,IT)
 64     CONTINUE
        ERROR2=0.0
        DO 65 I=1,IT
        ERROR3=ABS((TBL(I)-TBL2(I))/TBL(I))
        IF (ERROR3.GT.ERROR2) ERROR2=ERROR3
 65     CONTINUE
C          CHECK IF LIQUID FLOW ON ANY PLATE AS CHANGED MORE THAN
C         0.0025 PER CENT
C          IF CHANGE LESS THAN 0.0025 PER CENT GET OUT OF LOOP
        IF (ERROR2.LT.0.00025) GO TO 67
C          CONTINUE IN THIS LOOP  IF ILOOP2 LESS THAN 6
 66     CONTINUE
 67     CONTINUE
C          CALCULATE STAGE TEMPERATURES AND VAPOUR COMPOSITIONS FROM
C          SUBROUTINE KVALUE
        DO 68 J=1,IT
C       HAVE TO CALCULATE FROM BOTTOM TO TOP BECAUSE OF STAGE EFFICIENCES
        I=IT+1-J
        CALL KVALUE (X,Y,EK,E,T(I),P(I),I,30)
 68     CONTINUE
C
C          IF LIQUID FLOW CALCULATED WITHIN 10 PER CENT OF DESIRED
C          AND THIS IS NOT THE FIRST LOOR , CALCULATE ENTHALPY BALANCE
        IF (IEFLAG.EQ.2.OR.ILOOP.EQ.1) GO TO 86
        IF (ISET.EQ.20HFLOATING PRODUCTS    ) GO TO 69
C       ENTHALPY BALANCE FOR CONSTANT PRODUCT RATE
        CALL ENBAL1
        GO TO 70
C       ENTHALPY BALANCE FOR FLOATING PRODUCTS
 69     CALL ENBAL2
 70     CONTINUE
```

```
C
C          SET UP VECTOR OF INDEPENDENT VARIABLES *VAR*
      DO 71 I=1,ITM1
      VAR(I)=T(I+1)
 71   CONTINUE
      DO 72 I=IT,INDVAR
      VAR(I)=BV(I-IT+2)
 72   CONTINUE
C          CALL SUBROUTINE CVGP -CONVERGENCE PROMOTION
      CALL CVGP (ILOOP3,INDVAR,VAR,30,1.0E-40,1.0E-40)
C          SET NEW CALCULATED INDEPENDENT VARIABLES TEMPERATURE AND
C          VAPOUR FLOW
      DO 73 I[illegible]ITM1
      T(I+1)=VAR(I)
 73   CONTINUE
C
      IF (ISET.EQ.20HFLOATING PRODUCTS    ) GO TO 75
C     FOR CONSTANT OVERHEAD RATE VAPOUR RATE TO CONDENSER MUST BE CONST
      BVZ=BV(2)
      DO 74 I=IT,INDVAR
      BV(I-IT+2)=VAR(I)-VAR(IT)+BVZ
 74   CONTINUE
      GO TO 77
C     VAPOUR RATE TO CONDENSER MAY CHANGE FOR FLOATING OVERHEAD
 75   DO 76 I=IT,INDVAR
      BV(I-IT+2)=VAR(I)
 76   CONTINUE
 77   CONTINUE
C          RECALCULATE LIQUID FLOWS FOR MASS BALANCE
      BL(1)=RR*BV(2)/(1.0+RR)-BV(1)
      DO 78 I=2,IT
      IF (I.EQ.ISM1) GO TO 79
      BL(I)=BV(I+1)+BL(I-1)-BV(I)
 78   CONTINUE
 79   BL(I)=BV(I+1)+SOVRV+BL(I-1)-BV(I)
      I=I+1
      BL(I)=BV(I+1)+BL(I-1)+SOVRL-BV(I)
      J=I+1
      DO 80 I=J,IT
      IF (I.EQ.IFM1) GO TO 81
      BL(I)=BV(I+1)+BL(I-1)-BV(I)
 80   CONTINUE
 81   BL(I)=BV(I+1)+BL(I-1)+FEDRV-BV(I)
      I=I+1
      IF (I.EQ.IT) GO TO 84
      BL(I)=BV(I+1)+BL(I-1)+FEDRL-BV(I)
      J=I+1
      DO 82 I=J,IT
      IF (I.EQ.IT) GO TO 83
      BL(I)=BV(I+1)+BL(I-1)-BV(I)
 82   CONTINUE
C
 83   BV(I)=BL(I-1)-BL(I)
      GO TO 85
 84   BL(I)=BL(I-1)+FEDRL-BV(I)
 85   CONTINUE
      ILOOP3=ILOOP3+1
 86   CONTINUE
```

```
C        PRINT NEW TEMPERATURE, VAPOUR RATES AND LIQUID RATES
      PRINT 16
      PRINT 11, (T(I),I=1,IT)
      PRINT 30
      PRINT 11, (BV(I),I=1,IT)
      PRINT 31
      PRINT 11, (BL(I),I=1,IT)
C    *ERROR* IS LARGEST FRACTION ERROR OF THE SUM OF LIQUID RATES
C    COMPARED TO REQUIRED LIQUID RATE
      ERROR=0.0
      DO 87 I=1,IT
      ERROR1=ABS((BL(I)-TBL(I))/BL(I))
      IF (ERROR1.GT.ERROR) ERROR=ERROR1
87    CONTINUE
C    IF LIQUID RATE CHANGE LESS THAN 0.0005 CASE HAS CONVERGED
      IF (ICFLAG.EQ.1.AND.ERROR.LT.0.005) GO TO 90
      IF (ERROR.LT.0.0005) ICFLAG=1
C     SET IEFLAG=1 IF ERROR LESS THAN 0.1
C     WITH IEFLAG=1 ENERGY BALANCE IS CALCULATED DURING NEXT LOOP
      IF (ERROR.LE.0.1) IEFLAG=1
      PRINT 88, IEFLAG,ICFLAG
88    FORMAT (* *,//,5X,2I5)
89    CONTINUE
C
C***********************************************************************
C
C     FINAL CONDITION PRINT OUT
C
90    PRINT 91
91    FORMAT (*1*,//,5X,*FINAL CONDITIONS*,//)
      PRINT 3, ISET
      PRINT 5, TITLE1,TITLE2,TITLE3
      PRINT 7, NC,IS,IF,IT
      PRINT 9
      PRINT 11, SOVP,SOVT,SOVFRV,(SOVC(I),I=1,NC)
      PRINT 10
      PRINT 11, FEDP,FEDT,FEDFRV,(FEDC(I),I=1,NC)
      PRINT 16
      PRINT 11, (T(I),I=1,IT)
      PRINT 20
      PRINT 11, (P(I),I=1,IT)
      PRINT 29
      PRINT 11, RR,BV(IT),BOILUP
      PRINT 36
      DO 92 J=1,NC
      PRINT 11, (E(I,J),I=1,IT)
92    CONTINUE
      PRINT 30
      PRINT 11, (BV(I),I=1,IT)
      PRINT 93
93    FORMAT (* *,//,5X,*VAPOUR MOLE FRACTIONS*)
      DO 94 KJ=1,NC
      PRINT 11, (Y(I,KJ),I=1,IT)
94    CONTINUE
      PRINT 31
      PRINT 11, (BL(I),I=1,IT)
      PRINT 63
      DO 95 KJ=1,NC
```

```
      PRINT 11, (X(I,KJ),I=1,IT)
95    CONTINUE
      PRINT 96
96    FORMAT (* *,//,5X,*OVERHEAD PRODUCT CONDITIONS (FLOW,TEMP,COMP)*)
      PRINT 11, WL(1),T(1),(X(1,I),I=1,NC)
      PRINT 97
97    FORMAT (* *,//,5X,*BOTTOM PRODUCT CONDITIONS (FLOW,TEMP,COMP)*)
      PRINT 11, BL(IT),T(IT),(X(IT,I),I=1,NC)
      PRINT 98
98    FORMAT (* *,//,5X,*SEPARATIVE DUTIES*)
      SEPDUT(1)=WL(1)*(2.0*X(1,1)-1.0)*ALOG(X(1,1)/(1.0-X(1,1)))+BL(IT)*
     1(2.0*X(IT,1)-1.0)*ALOG(X(IT,1)/(1.0-X(IT,1)))-FEDR*(2.0*FEDC(1)-1.
     20)*ALOG(FEDC(1)/(1.0-FEDC(1)))
      SEPDUT(2)=WL(1)*(2.0*X(1,2)-1.0)*ALOG(X(1,2)/(1.0-X(1,2)))+BL(IT)*
     1(2.0*X(IT,2)-1.0)*ALOG(X(IT,2)/(1.0-X(IT,2)))-FEDR*(2.0*FEDC(2)-1.
     20)*ALOG(FEDC(2)/(1.0-FEDC(2)))
      SEPDUT(3)=WL(1)*(2.0*X(1,3)-1.0)*ALOG(X(1,3)/(1.0-X(1,3)))+BL(IT)*
     1(2.0*X(IT,3)-1.0)*ALOG(X(IT,3)/(1.0-X(IT,3)))
      PRINT 11, (SEPDUT(I),I=1,NC)
      PRINT 99
99    FORMAT (* *,//,5X,*SEPARATIVE DUTIES PER MOLE OF COMPONENT FEEDS*)
      DO 100 I=1,NC
      SEPDU1(I)=SEPDUT(I)/(SOVR*SOVC(I)+FEDR*FEDC(I))
100   CONTINUE
      PRINT 11, (SEPDU1(I),I=1,NC)
C        FINAL CONDITION PUNCH OUT IN SAME FORMAT AS INPUT
      PUNCH 2, ISET
      PUNCH 4, TITLE1,TITLE2,TITLE3
      PUNCH 6, NC,IS,IF,IT
      PUNCH 8, SOVR,SOVT,SOVFRV,(SOVC(I),I=1,NC)
      PUNCH 8, FEDR,FEDT,FEDFRV,(FEDC(I),I=1,NC)
      ISEE=1
      PUNCH 12, ISEE,T(1),T(IT)
      PUNCH 8, (T(I),I=1,IT)
      PUNCH 12, ISEE,P(1),P(IT)
      PUNCH 8, (P(I),I=1,IT)
      PUNCH 12, ISEE,RR,BV(IT),BOILUP
      PUNCH 8, (BV(I),I=1,IT)
      PUNCH 8, (BL(I),I=1,IT)
      DO 101 J=2,NC
      PUNCH 8, E(1,J),E(IS,J),E(IF,J),E(IT,J)
101   CONTINUE
      DO 102 I=1,IT
      PUNCH 8, (X(I,J),J=1,NC)
102   CONTINUE
      PUNCH 8, (SEPDUT(I),I=1,NC)
      PUNCH 8, (SEPDU1(I),I=1,NC)
      CALL DATE (ADATE)
      CALL TIME (ATIME)
      PUNCH 103, ADATE,ATIME
103   FORMAT (2A15)
      STOP
      END
```

```
                                                                        EN
      SUBROUTINE ENBAL1                                                 EN
C***********************************************************************EN
C                                                                       EN
C         THIS ROUTINE CHANGES TOTAL LIQUID AND VAPOUR FLOWS TO         EN
C         PRODUCE AN ENTHALPY BALANCE AROUND EACH STAGE                 EN
C         THIS ENTHALPY BALANCE ROUTINE REQUIRES A FIXED OVERHEAD PRODUCTEN
C         AND REFLUX RATIO                                              EN
C                                                                       EN
C         AN ENTHALPY BALANCE IS CALCULATED DOWN THE TOWER FROM THE TOP EN
C         VAPOUR RATE ON TRAY(J) AND LIQUID RATE ON TRAY (J-1) RECALCULATEN
C         USING ENTHALPY BALANCE                                        EN
C         METHOD AS DESCRIBED BY C.J. KING  *SEPARATION PROCESSES*      EN
C         PG 534                                                        EN
C         WRITTEN BY D.L. ONEILL JULY. 1974                             EN
C                                                                       EN
C     DEFINITION OF MAJOR VARIABLES                                     EN
C    -THIS LIST DOES NOT INCLUDE VARIABLES DEFINED IN THE MAIN          EN
C    PROGRAM *TOWER*                                                    EN
C                                                                       EN
C    DEDV    STAGE ENTHALPY BALANCE (INPUT-OUTPUT) CHANGE IF INLET VAPOUR EN
C            RATE AND OUTLET LIQUID RATES ARE INCREASED BY 1 GM MOLE/HR EN
C    DEL     STAGE LIQUID RATE CHANGE REQUIRED TO PUT STAGE INTO ENTHALPY, EN
C    ENTHBL(I) TOTAL ENTHALPY (KCAL/HR) OF LIQUID STREAM FROM STAGE(I)  EN
C    ENTHBV(I) TOTAL ENTHALPY (KCAL/HR) OF VAPOUR STREAM FROM STAGE(I)  EN
C    FEL     TOTAL ENTHALPY (KCAL/HR) OF LIQUID FRACTION OF FEED STREAM EN
C    FEV     TOTAL ENTHALPY (KCAL/HR) OF VAPOUR FRACTION OF FEED STREAM EN
C    QUNBAL(I) STAGE(I) ENTHALPY BALANCE (INPUT-OUTPUT) (KCAL/HR)       EN
C    SEL      TOTAL ENTHALPY (KCAL/HR) OF LIQUID FRACTION OF SOLVENT STREAMEN
C    SEV      TOTAL ENTHALPY (KCAL/HR) OF VAPOUR FRACTION OF SOLVENT STREAMEN
C    TOTE   TOTAL ENTHALPY OF FEED AND SOLVENT STREAMS                  EN
C                                                                       EN
C***********************************************************************EN
      COMMON /BLK1/ BV(20),BL(20),T(20),P(20),SL(20,3),IT,NC,IS,IF,PR   EN
      COMMON /BLK4/ X(20,3),Y(20,3),EK(3),E(20,3)                       EN
      COMMON /BLK5/ WL(20),WV(20),SF(20,3),BOILUP,SOVRV,SOVPL,FEDRV,FEDREN
     1L,FEDC(3),SOVC(3),SOVT,FEDT,CONDUT,REBDUT                         EN
      DIMENSION QUNBAL(20), ENTHBL(20), ENTHBV(20), DEDV(20), DEL(20)   EN
C       CALCULATE TOTAL STREAM ENTHALPIES                               EN
      DO 1 I=1,IT                                                       EN
      ENTHBL(I)=ENTLIQ(T(I),BL(I),X(I,1),X(I,2))                        EN
      ENTHBV(I)=ENTVAP(T(I),BV(I),Y(I,1),Y(I,2))                        EN
    1 CONTINUE                                                          EN
      PRINT 2                                                           EN
    2 FORMAT (* *,//,5X,*LIQUID STREAM ENTHALPIES*)                     EN
      PRINT 4, (ENTHBL(I),I=1,IT)                                       EN
      PRINT 3                                                           EN
    3 FORMAT (* *,//,5X,*VAPOUR STREAM ENTHALPIES*)                     EN
      PRINT 4, (ENTHBV(I),I=1,IT)                                       EN
    4 FORMAT (* *,/,10(5X,5G16.5,/))                                    EN
      PRINT 5                                                           EN
    5 FORMAT (* *,//,5X,*FEED STREAM ENTHALPIES*)                       EN
      PRINT 4, ENTLIQ(SOVT,SOVPL,SOVC(1),SOVC(2)),ENTVAP(SOVT,SOVRV,SOVCEN
     1(1),SOVC(2)),ENTLIQ(FEDT,FEDRL,FEDC(1),FEDC(2)),ENTVAP(FEDT,FEDRV,EN
     2FEDC(1),FEDC(2))                                                  EN
      ISM1=IS-1                                                         EN
      ISM2=IS-2                                                         EN
      IFM1=IF-1                                                         EN
      IFM2=IF-2                                                         EN
      ITM1=IT-1
```

```
      QUNBAL(1)=ENTHBV(2)-ENTHBL(1)-ENTLIQ(T(1),WL(1),X(1,1),X(1,2))
      CONDUT=QUNBAL(1)
C        CALCULATE CHANGE IN TRAY ENTHALPY BALANCE IF INLET VAPOUR RATE
C        TO STAGE AND OUTLET LIQUID RATE FROM STAGE (NOTE. STILL TOTAL
C        MASS BALANCE) INCREASED BY 1 GM MOLE / HR
      DO 6 J=2,ITM1
      DEDV(J)=ENTVAP(T(J+1),1.0,Y(J+1,1),Y(J+1,2))-ENTLIQ(T(J),1.0,X(J,1
     1),X(J,2))
6     CONTINUE
      PRINT 4, (DEDV(J),J=1,IT)
C        CALCULATE TRAY HEAT UNBALANCE
      SEV=ENTVAP(SOVT,SOVRV,SOVC(1),SOVC(2))
      IF (ISM2.LE.1) GO TO 8
      DO 7 J=2,ISM2
      QUNBAL(J)=ENTHBV(J+1)+ENTHBL(1)-ENTHBV(2)-ENTHBL(J)
7     CONTINUE
8     IF (ISM1.GT.1) GO TO 9
      QUNBAL(1)=QUNBAL(1)+ENTVAP(SOVT,SOVRV,SOVC(1),SOVC(2))
      GO TO 10
9     QUNBAL(ISM1)=ENTHBV(IS)+ENTHBL(1)-ENTHBV(2)-ENTHBL(ISM1)+SEV
10    SEL=ENTLIQ(SOVT,SOVRL,SOVC(1),SOVC(2))
      QUNBAL(IS)=ENTHBV(IS+1)+ENTHBL(1)+SEL+SEV-ENTHBV(2)-ENTHBL(IS)
      ISP1=IS+1
      IF (IFM2.LT.ISP1) GO TO 12
      DO 11 J=ISP1,IFM2
      QUNBAL(J)=ENTHBV(J+1)+ENTHBL(1)-ENTHBV(2)-ENTHBL(J)+SEL+SEV
11    CONTINUE
12    FEV=ENTVAP(FEDT,FEDRV,FEDC(1),FEDC(2))
      FEL=ENTLIQ(FEDT,FEDRL,FEDC(1),FEDC(2))
      TOTE=FEL+FEV+SEL+SEV
      IF (IFM1.LE.IS) GO TO 13
      QUNBAL(IFM1)=ENTHBV(IF)+ENTHBL(1)-ENTHBV(2)-ENTHBL(IFM1)+FEV+SEL+S
     1EV
13    IF (IF.LT.IT) GO TO 14
      QUNBAL(IT)=ENTHBL(1)+TOTE-ENTHBV(2)-ENTHBL(IT)
      GO TO 16
14    QUNBAL(IF)=ENTHBV(IF+1)+ENTHBL(1)-ENTHBV(2)-ENTHBL(IF)+TOTE
      IFP1=IF+1
      DO 15 J=IFP1,ITM1
      QUNBAL(J)=ENTHBV(J+1)+ENTHBL(1)-ENTHBV(2)-ENTHBL(J)+TOTE
15    CONTINUE
      QUNBAL(IT)=ENTHBL(1)+TOTE-ENTHBV(2)-ENTHBL(IT)
16    CONTINUE
      REBDUT=-QUNBAL(IT)
      PRINT 17
17    FORMAT (* *,//,5X,*HEAT UNBALANCE , INPUT-OUTPUT*)
      PRINT 4, (QUNBAL(J),J=1,IT)
C        CALCULATE LIQUID CHANGE TO EACH STAGE REQUIRED TO OBTAIN
C        ENTHALPY BALANCE
C        LIQUID RATE CHANGE=- HEAT UNBALANCE(KCAL/HR) / INCREASE IN HEAT
C        PER UNIT INCREASE IN INLET VAPOUR RATE
      DO 18 J=2,ITM1
      DEL(J)=-QUNBAL(J)/DEDV(J)
18    CONTINUE
C        CALCULATE NEW LIQUID AND VAPOUR RATES
C        ( ALL LIQUID RATE CHANGES ABOVE TRAY(J) AFFECT TRAY(J) LIQUID R
      DO 19 J=2,ITM1
      BV(J+1)=BV(J+1)+DEL(J)
```

```
      BL(J)=BL(J)+DEL(J)
19    CONTINUE
      PRINT 20
20    FORMAT (* *,//,5X,*MOLAR VAPOUR FLOWS*)
      PRINT 4, (BV(J),J=1,IT)
      PRINT 21
21    FORMAT (* *,//,5X,*MOLAR LIQUID FLOWS*)
      PRINT 4, (BL(J),J=1,IT)
      RETURN
      END
```

```
      SUBROUTINE ENBAL2                                                 EN
C***********************************************************************EN
C                                                                       EN
C          THIS ROUTINE CHANGES TOTAL LIQUID AND VAPOUR FLOWS TO        EN
C          PRODUCE AN ENTHALPY BALANCE AROUND EACH STAGE                EN
C          THIS ENTHALPY BALANCE ROUTINE REQUIRES FLOATING PRODUCT RATES EN
C          AND CONSTANT REFLUX RATIO AND GIVEN BOILUP RATE              EN
C          OR REBOILER HEAT INPUT                                       EN
C                                                                       EN
C          THE ENTHALPY BALANCE IS ADJUSTED AROUND                      EN
C          EACH STAGE BY CHANGING THE VAPOUR AND LIQUID RATES           EN
C          FROM THAT STAGE                                              EN
C          METHOD AS DESCRIBED BY HANSON ET AL. (1962), METHOD II       EN
C          WRITTEN BY D.L. ONEILL  JUNE,1974                            EN
C                                                                       EN
C    DEFINITION OF MAJOR VARIABLES                                      EN
C   -THIS LIST DOES NOT INCLUDE VARIABLES DEFINED IN THE MAIN           EN
C   PROGRAM *TOWER*                                                     EN
C                                                                       EN
C   BLT       TEMPORARY LIQUID RATE FROM HYPOTHETICAL FLASH PROCESS     EN
C   BVT       TEMPORARY VAPOUR RATE FROM HYPOTHETICAL FLASH PROCESS     EN
C   CL(J)     CHANGE IN LIQUID ENTHALPY WITH A 1 DEG INCREASE IN        EN
C             STAGE(J) TEMPERATURE                                      EN
C   CV(J)     CHANGE IN VAPOUR ENTHALPY WITH A 1 DEG INCREASE IN        EN
C             STAGE(J) TEMPERATURE                                      EN
C   DELL(J)   CHANGE IN STAGE(J) LIQUID RATE TO PRODUCE AN ENTHALPY     EN
C             BALANCE                                                   EN
C   DELT(J)   CHANGE IN STAGE(J) TEMPERATURE TO PRODUCE AN ENTHALPY     EN
C             BALANCE                                                   EN
C   DELV(J)   CHANGE IN STAGE(J) VAPOUR RATE TO PRODUCE AN ENTHALPY     EN
C             BALANCE ( DELV(J)=-DELL(J) ).                             EN
C   DVDT(J)   DERIVATIVE DV/DT - CHANGE IN VAPOUR RATE PER UNIT         EN
C             CHANGE IN STAGE(J) TEMPERATURE                            EN
C   ENEW      TOTAL ENTHALPY OF REBOILER VAPOUR                         EN
C   ENTHBL(J) TOTAL LIQUID ENTHALPY FROM STAGE(J)                       EN
C   ENTHBV(J) TOTAL VAPOUR ENTHALPY FROM STAGE(J)                       EN
C   ENTLT     TOTAL LIQUID STREAM ENTHALPY FROM HYPOTHETICAL FLASH PROC EN
C   ENTVT     TOTAL VAPOUR STREAM ENTHALPY FROM HYPOTHETICAL FLASH PROC EN
C   FLFEED(I)  TOTAL COMPONENT(I) FEED TO HYPOTHETICAL FLASH            EN
C   FLLIQ(I)  COMPONENT(I) LIQUID RATE FROM HYPOTHETICAL FLASH          EN
C   FLVAP(I)  COMPONENT(I) VAPOUR RATE FROM HYPOTHETICAL FLASH          EN
C   QUNBAL(J) STAGE(J) ENTHALPY BALANCE (KCAL/HR )  ,INPUT-OUTPUT       EN
C   TEMP      HYPOTHETICAL FLASH PROCESS TEMPERATURE                    EN
C   VNEW      CALCULATED REBOILER VAPOUR RATE                           EN
C                                                                       EN
C***********************************************************************EN
      COMMON /BLK1/ BV(20),BL(20),T(20),P(20),SL(20,3),IT,NC,IS,IF,PP   EN
      COMMON /BLK3/ IEFLAG,ICFLAG,ILOOP                                 EN
      COMMON /BLK4/ X(20,3),Y(20,3),EK(3),E(20,3)                       EN
      COMMON /BLK5/ WL(20),WV(20),SF(20,3),BOILUP,SOVRV,SOVRL,FEDRV,FEDREN
     1L,FEDC(3),SOVC(3),SOVT,FEDT,CONDUT,REBDUT                         EN
      DIMENSION DVDT(20), FLFEED(3), FLVAP(3), FLLIQ(3), CV(20), CL(20),EN
     1 QUNBAL(20), ENTHBL(20), ENTHBV(20), DELT(20), DELV(20), DELL(20) EN
C        LEAST SQUARE FIT OF VALUES OF K (Y/X) FOR ACETONE,METHANOL     EN
C        AND WATER                                                      EN
      ACK2(TC,X1,X2)=EXP(6.839-196.8/(TC-45.0)+2296./(TC-45.0)**2+2.966*EN
     1X1+9.15*X1**2-5.932*X2+2.967*X2**2-221.5*X1/(TC-45.0)+66.11*X2/(TCEN
     2-45.0))                                                           EN
      RMEK2(TC,X1,X2)=EXP(5.462-211.1/(TC-45.0)+2318.0/(TC-45.0)**2-4.27EN
```

```
     12*X2+2.06*X2**2+5.671*X1+9.416*X1**2+65.51*X2/(TC-45.0)-222.4*X1/(
     2TC-45.0))
      WTK2(TC,X1,X2)=EXP(3.855-132.5/(TC-45.0)+864.6/(TC-45.0)**2-4.188*
     1X2+1.652*X2**2+2.006*X1+0.1719*X1**2+34.25*X2/(TC-45.0)-14.82*X1/(
     2TC-45.0))
C        CALCULATE STREAM ENTHALPIES
      DO 10 I=1,IT
      ENTHBL(I)=ENTLIQ(T(I),BL(I),X(I,1),X(I,2))
      ENTHBV(I)=ENTVAP(T(I),BV(I),Y(I,1),Y(I,2))
   10 CONTINUE
C        IF BOILUP (REBOILER HEAT INPUT) IS ZERO USE OLD REBOILER
C        VAPOUR RATE
      IF (BOILUP.EQ.0.0) GO TO 2
C        CALCULATE NEW REBOILER VAPOUR RATE BY ENTHALPY BALANCE
      ENEW=ENTHBL(IT-1)+BOILUP-ENTHBL(IT)
      IF (IT.EQ.IF) ENEW=ENEW+ENTLIQ(FEDT,FEDRL,FEDC(1),FEDC(2))
      VNEW=BV(IT)/ENTHBV(IT)*ENEW
      ENTHBV(IT)=VNEW/BV(IT)*ENTHBV(IT)
C        RESTRICT VAPOUR CHANGE TO 10 PER CENT
      IF ((BV(IT)-VNEW)/BV(IT).GT.0.10) VNEW=0.9*BV(IT)
      IF ((BV(IT)-VNEW)/BV(IT).LT.-0.10) VNEW=1.1*BV(IT)
      BV(IT)=VNEW
    2 CONTINUE
      PRINT 3
    3 FORMAT (* *,//,5X,*LIQUID STREAM ENTHALPIES*)
      PRINT 5, (ENTHBL(I),I=1,IT)
      PRINT 4
    4 FORMAT (* *,//,5X,*VAPOUR STREAM ENTHALPIES*)
      PRINT 5, (ENTHBV(I),I=1,IT)
    5 FORMAT (* *,/,10(5X,5G16.5,/))
      ISM1=IS-1
      ISM2=IS-2
      IFM1=IF-1
      IFM2=IF-2
      ITM1=IT-1
      DVDT1=1.0
C
C        CALCULATING VAPOUR RATE CHANGE PER DEGREE OF TEMPERATURE (DVDT)
      DO 8 J=2,ITM1
      TEMP=T(J)+1.0
      DVDT(J)=BLT=BVT=0.0
      EK(1)=ACK2(TEMP,X(J,1),X(J,2))
      EK(2)=RMEK2(TEMP,X(J,1),X(J,2))
      EK(3)=WTK2(TEMP,X(J,1),X(J,3))
C     TAKE OUTPUT STREAMS FROM STAGE(J) AND CALCULATE ENTHALPY
C     CHANGES IF THEY WERE PUT THROUGH A FLASH PROCESS AT A
C     TEMPERATURE 1 DEGREE HIGHER THAN THE STAGE TEMPERATURE
      DO 6 I=1,NC
C    MOLAR COMPONENT I FLOW OF STREAMS LEAVING STAGE J
      FLFEED(I)=BL(J)*X(J,I)+BV(J)*Y(J,I)
C    MOLAR COMPONENT FLOW OF VAPOUR LEAVING FLASH PROCESS AT *TEMP*
      FLVAP(I)=FLFEED(I)/(BL(J)/(BV(J)*EK(I))+1.0)
      DVDT(J)=DVDT(J)+FLVAP(I)
      FLLIQ(I)=FLFEED(I)-FLVAP(I)
      BLT=BLT+FLLIQ(I)
      BVT=BVT+FLVAP(I)
    6 CONTINUE
C     CALCULATE CHANGE IN TOTAL VAPOUR RATE WITH A 1 DEG. INCREASE IN
```

```
C     STAGE TEMP
      DVDT(J)=DVDT(J)-BV(J)
C     IF *DVDT* IS NOT POSITIVE THERE IS SOMETHING WRONG
C     THEREFORE USE LATEST DVDT
      IF (DVDT(J).GT.0.0) GO TO 7
      BVT=BV(J)+DVDT1
      BLT=BL(J)-DVDT1
      DVDT(J)=DVDT1
7     DVDT1=DVDT(J)
      ENTLT=ENTLIQ(TEMP,BLT,X(J,1),X(J,2))
      ENTVT=ENTVAP(TEMP,BVT,Y(J,1),Y(J,2))
C        CALCULATE ENTHALPY CHANGE FOR LIQUID AND VAPOUR STREAMS
C        PER 1 DEGREE CHANGE OF TEMPERATURE
      CV(J)=ENTVT-ENTHBV(J)
      CL(J)=ENTLT-ENTHBL(J)
8     CONTINUE
      CV(1)=CL(1)=CV(IT)=CL(IT)=1.0
      DVDT(1)=DVDT(IT)=0.0
C
C        CALCULATE HEAT UNBALANCE (QUNBAL) FOR EACH TRAY
C     INPUT - OUTPUT
      QUNBAL(1)=ENTHBV(2)-ENTHBL(1)-ENTLIQ(T(1),WL(1),X(1,1),X(1,2))
      CONDUT=QUNBAL(1)
      IF (ISM2.LE.1) GO TO 10
      DO 9 J=2,ISM2
      QUNBAL(J)=ENTHBV(J+1)+ENTHBL(J-1)-ENTHBV(J)-ENTHBL(J)
9     CONTINUE
10    IF (ISM1.GT.1) GO TO 11
      QUNBAL(1)=QUNBAL(1)+ENTVAP(SOVT,SOVRV,SOVC(1),SOVC(2))
      GO TO 12
11    QUNBAL(ISM1)=ENTHBV(IS)+ENTHBL(ISM2)-ENTHBV(ISM1)-ENTHBL(ISM1)+ENT
     1VAP(SOVT,SOVRV,SOVC(1),SOVC(2))
12    QUNBAL(IS)=ENTHBV(IS+1)+ENTHBL(ISM1)-ENTHBV(IS)-ENTHBL(IS)+ENTLIQ(
     1SOVT,SOVRL,SOVC(1),SOVC(2))
      ISP1=IS+1
      IF (IFM2.LT.ISP1) GO TO 14
      DO 13 J=ISP1,IFM2
      QUNBAL(J)=ENTHBV(J+1)+ENTHBL(J-1)-ENTHBV(J)-ENTHBL(J)
13    CONTINUE
14    IF (IFM1.LE.IS) GO TO 15
      QUNBAL(IFM1)=ENTHBV(IFM1+1)+ENTHBL(IFM2)-ENTHBL(IFM1)-ENTHBV(IFM1)
     1+ENTVAP(FEDT,FEDRV,FEDC(1),FEDC(2))
15    IF (IF.LT.IT) GO TO 16
      QUNBAL(IT)=ENTHBL(IFM1)-ENTHBV(IF)-ENTHBL(IF)+ENTLIQ(FEDT,FEDRL,FE
     1DC(1),FEDC(2))
      GO TO 18
16    QUNBAL(IF)=ENTHBV(IF+1)+ENTHBL(IFM1)-ENTHBV(IF)-ENTHBL(IF)+ENTLIQ(
     1FEDT,FEDRL,FEDC(1),FEDC(2))
      IFP1=IF+1
      DO 17 J=IFP1,ITM1
      QUNBAL(J)=ENTHBV(J+1)+ENTHBL(J-1)-ENTHBV(J)-ENTHBL(J)
17    CONTINUE
      QUNBAL(IT)=ENTHBL(ITM1)-ENTHBV(IT)-ENTHBL(IT)
18    CONTINUE
      REBDUT=-QUNBAL(IT)
      PRINT 19
19    FORMAT (* *,//,5X,*FEED STREAM ENTHALPIES*)
      PRINT 5, ENTLIQ(SOVT,SOVRL,SOVC(1),SOVC(2)),ENTVAP(SOVT,SOVRV,SOVC
```

```
     1(1),SOVC(2)),ENTLIQ(FEDT,FEDRL,FEDC(1),FEDC(2)),ENTVAP(FEDT,FEDPV,
     2FEDC(1),FEDC(2))
      PRINT 20
   20 FORMAT (* *,//,5X,*HEAT UNBALANCE , INPUT-OUTPUT*)
      PRINT 5, (QUNBAL(I),I=1,IT)
C     CALCULATE REQUIRED CHANGE IN STAGE TEMPERATURE, LIQUID AND
C     VAPOUR RATES FOR STAGE ENTHALPY BALANCE
C     NOTE STAGE STILL IN TOTAL MASS BALANCE
      DO 21 J=2,ITM1
      DELT(J)=QUNBAL(J)/(CV(J)+CL(J))
      DELV(J)=DVDT(J)*DELT(J)
      DELL(J)=-DELV(J)
   21 CONTINUE
      DELV(1)=DELL(1)=DELT(1)=0.0
      DELV(IT)=DELL(IT)=DELT(IT)=0.0
C     REDUCE VAPOUR (AND LIQUID ) RATE CHANGE BY A FACTOR OF 2
C     IF REQUIRED VAPOUR RATE IS GREATER THAN 10 PER CENT
      DO 25 IJ=1,10
      DO 22 J=2,ITM1
      IF (ABS(DELV(J)/BV(J)).GT.0.10) GO TO 23
   22 CONTINUE
C     VAPOUR RATE CHANGE IS WITHIN LIMITS
      GO TO 27
C        IF VAPOUR RATE CHANGES GREATER THAN 10 PER CENT REDUCE CHANGE
C        BY A FACTOR OF 2
   23 DO 24 J=2,ITM1
      DELV(J)=DELV(J)*0.5
      DELL(J)=DELL(J)*0.5
      DELT(J)=DELT(J)*0.5
   24 CONTINUE
   25 CONTINUE
      PRINT 26
   26 FORMAT (* *,//,5X,*VAPOUR CHANGE IN ENBAL TOO HIGH*)
C        VAPOUR RATE CHANGES ARE ADDITIVE FROM THE BOTTOM UP
   27 DO 28 J=2,ITM1
      K=IT+1-J
      DELV(K)=DELV(K)+DELV(K+1)
   28 CONTINUE
C        NEW VAPOUR RATES
C     CALCULATE NEW VAPOUR RATES
      DO 29 J=2,ITM1
      BV(J)=BV(J)+DELV(J)
   29 CONTINUE
C        CHANGE LIQUID RATES FOR MASS BALANCE
      DELL(1)=RR*BV(2)/(1.0+RR)-BV(1)-BL(1)
      BL(1)=BL(1)+DELL(1)
C     CALCULATE NEW LIQUID RATES
      DO 30 J=2,ITM1
      BL(J)=BL(J)+DELL(1)+DELL(J)
C     ADD LIQUID RATE CHANGES FROM STAGES ABOVE STAGE(J)
      DELL(1)=DELL(1)+DELL(J)
   30 CONTINUE
C     CALCULATE NEW BOTTOM PRODUCT RATE
      BL(IT)=BL(ITM1)-BV(IT)
      IF (IF.EQ.IT) BL(IT)=FEDRL+BL(IT)
C     NEW OVERHEAD RATE IS CALCULATED IN PROGRAM *TOWER*
      PRINT 31
   31 FORMAT (* *,//,5X,*MOLAR VAPOUR FLOWS*)
```

```
      PRINT 5, (BV(I),I=1,IT)                                          EN.
      PRINT 32                                                         EN.
   32 FORMAT (* *,//,5X,*MOLAR LIQUID FLOWS*)                          EN.
      PRINT 5, (BL(I),I=1,IT)                                          EN.
      RETURN                                                           EN.
      END                                                              EN.
```

142(a)

```
      SUBROUTINE KVALUE (X,Y,EK,E,T1,PT,IM,ITYPE)
C***********************************************************************
C
C     ORIGINAL PROGRAM WRITTEN BY T.W.HOFFMAN,APRIL,1973.
C     METHOD OF ITERATION REVISED BY W.D.JACKSON,APRIL,1973.
C     CHANGED FROM WILSON TO ENTHALPIC WILSON BY DL ONEILL,MARCH,1974.
C
C
C         MURPHREE EFFICIENCY INCLUDED JUNE,1974.
C         EFFICIENCIES FOR DIFFERENT COMPONENTS CHANGED JAN,1975
C
C         EFFICIENCIES DISCUSSION PG. 537 *SEPARATION PROCESSES*
C         C.J. KING
C         FOR THE LIQUID TO BE AT SATURATION TEMPERATURE
C         ONLY (NC - 1 ) (NC=NO. OF COMPONENTS) EFFICIENCIES
C         CAN BE SET
C
C         BINARY DATA COEFFICIENTS FROM S. BRUIN IND. ENG. CHEM. FUND.
C         VOL. 9 , NO.3 AUGUST 1970, PG. 305
C         THIS SUBROUTINE CALCULATES THE EQUILIBRIUM VAPOUR COMPOSITION
C         OF AN ACETONE-METHANOL-WATER MIXTURE GIVEN THE PRESSURE
C         ( MM HG ), LIQUID COMPOSITION AND INITIAL ESTIMATE OF THE
C         EQUILIBRIUM TEMPERATURE ( DEG. C )
C         USING MURPHREE VAPOUR EFFICIENCIES E=(Y(J)-Y(J+1))/(Y(J)*
C         -Y(J+1))
C         THE NON-EQUILIBRIUM VAPOUR COMPOSITION IS DETERMINED
C         THE EQUILIBRIUM CONSTANT EK (K) =Y/X
C         IS THEN CALCULATED FOR EACH COMPONENT
C         THE RESULTS FROM THIS ROUTINE HAVE BEEN CHECKED WITH
C         EXPERIMENTAL EQUILIBRIUM DATA AND IS AT LEAST WITHIN
C         + OR - 0.05 MOLE FRACTION OF THE EXPERIMENTAL AND USUALLY
C         MUCH BETTER
C
C***********************************************************************
      COMMON /BLK1/ BV(20),BL(20),T(20),P(20),SL(20,3),IT,NC,IS,IF,PR
      COMMON /BLK3/ IEFLAG,ICFLAG,ILOOP
      DIMENSION A(3), B(3), C(3), AA(3), BB(3), CC(3), X(20,3), GAM(3,3)
     1, ACT(3), LAM(3,3), Y(20,3), V(3), VV(3), E(20,3), EK(3), TERM(3)
      DIMENSION ALP(3,3), ALP1(3,3,3), SUMGX(3)
      REAL LAM
      DATA AA/56.9622,21.795,22.8876/,BB/8.4555E-3,4.9507E-2,-3.642E-2/,
     1CC/1.65E-4,5.68E-5,6.856E-5/
      DATA A/7.02447,7.87863,7.96681/,B/1161.,1473.11,1668.21/,C/224.,23
     10.,228./
      DO 1 I=1,NC
      IF (IM.EQ.IT) TERM(I)=X(IM,I)
      IF (IM.NE.IT) TERM(I)=Y(IM+1,I)
    1 CONTINUE
      TK=T1+273.0
      LAM(1,2)=-216.966
      LAM(2,1)=566.093
      LAM(1,3)=-436.468
      LAM(3,1)=1185.426
      LAM(2,3)=-238.7681
      LAM(3,2)=571.3078
C     ENTHALPIC WILSON EQUATION TO PREDICT VAPOUR MOL FRACTION IN EQUIBR
C     WITH GIVEN LIQUID CONCENTRATION
C     BINARY COEFFICIENTS FOR WILSON EQUATION,VAPOUR PRESSURE ANTOINE
C     COEFFICIENTS AND LIQUID MOLAR VOLUMES FROM
```

```
C     ACETONE -1, METHANOL -2, WATER -3                                 KV
      TK1=TK2=0.0                                                       KV
      DO 11 IJK=1,ITYPE                                                 KV
      DO 3 J=1,3                                                        KV
      DO 2 I=1,3                                                        KV
      IF (I.EQ.J) GO TO 2                                               KV
      FC=EXP(-LAM(I,J)/(1.987*TK))                                      KV
      V(J)=AA(J)+(BB(J)+CC(J)*TK)*TK                                    KV
      VV(I)=AA(I)+(BB(I)+CC(I)*TK)*TK                                   KV
      GAM(I,J)=V(J)/VV(I)*FC                                            KV
    2 CONTINUE                                                          KV
    3 CONTINUE                                                          KV
      GAM(1,1)=GAM(2,2)=GAM(3,3)=1.                                     KV
      DO 4 I=1,3                                                        KV
      SUMGX(I)=0.0                                                      KV
      DO 4 IP=1,3                                                       KV
    4 SUMGX(I)=SUMGX(I)+GAM(I,IP)*X(IM,IP)                              KV
      DO 5 I=1,3                                                        KV
      DO 5 J=1,3                                                        KV
    5 ALP(I,J)=1.0+X(IM,I)*(1.0-1.0/SUMGX(I)-GAM(J,I)/SUMGX(J))         KV
      DO 6 I=1,3                                                        KV
      DO 6 J=1,3                                                        KV
      DO 6 M=1,3                                                        KV
    6 ALP1(I,J,M)=1.0-GAM(J,I)/SUMGX(J)-GAM(M,I)/SUMGX(M)               KV
      SUMY=0.0                                                          KV
      DO 9 I=1,3                                                        KV
      SUM1=0.0                                                          KV
      DO 7 J=1,3                                                        KV
      SUM1=SUM1+X(IM,J)*ALOG(GAM(I,J)*GAM(J,I))*ALP(I,J)/(SUMGX(I)*SUMGXKV
     1(J))                                                              KV
    7 CONTINUE                                                          KV
      SUM2=0.0                                                          KV
      DO 8 M=1,3                                                        KV
      DO 8 J=1,3                                                        KV
      IF (J.EQ.I.OR.M.EQ.I.OR.M.EQ.J) GO TO 8                           KV
      SUM2=SUM2+X(IM,J)*X(IM,M)*ALOG(GAM(J,M)*GAM(M,J))*ALP1(I,J,M)/(SUMKV
     1GX(J)*SUMGX(M))                                                   KV
    8 CONTINUE                                                          KV
      TC=TK-273.2                                                       KV
      PC=10.0**(A(I)-B(I)/(C(I)+TC))                                    KV
      ACT(I)=EXP(-SUM1-SUM2)*PC/PT                                      KV
      Y(IM,I)=ACT(I)*X(IM,I)                                            KV
      SUMY=SUMY+Y(IM,I)                                                 KV
    9 CONTINUE                                                          KV
      IF (ITYPE.EQ.1) GO TO 13                                          KV
      FAC=SUMY-1.0                                                      KV
      IF (ABS(FAC).LE.0.0005) GO TO 13                                  KV
C     CONVERGENCE ON TK TO MAKE SUMY=1.0                                KV
      TK2=TK                                                            KV
      F2=FAC                                                            KV
      IF (IJK.GE.2) GO TO 10                                            KV
      TK1=TK                                                            KV
      F1=FAC                                                            KV
      IF (FAC.GT.0.0) TK=TK-2.0                                         KV
      IF (FAC.LT.0.0) TK=TK+2.0                                         KV
      IF (TK.GT.373.0) TK=(TK1+373.0)/2.0                               KV
      IF (TK.LT.328.0) TK=(TK1+328.0)/2.0                               KV
      GO TO 11                                                          KV
```

```
                                                                        KV
C     ITERATE BY REGULI-FALSI METHOD                                    KV
10    TK=(F1*TK2-F2*TK1)/(F1-F2)                                        KV
      IF (TK.LT.329.0) TK=(TK2+329.0)/2.0                               KV
      IF (TK.GT.373.0) TK=(TK2+373.0)/2.0                               KV
      TK1=TK2                                                           KV
      F1=F2                                                             KV
11    CONTINUE                                                          KV
      PRINT 12, IM                                                      KV
12    FORMAT (1X,* CONVERGENCE ON SUMY ON THE*,I5,*PLATE WAS NOT ACHIEVEKV
     1D*)                                                               KV
C     NORMALIZE MOLE FRACTIONS.                                         KV
13    DO 14 I=1,3                                                       KV
      Y(IM,I)=Y(IM,I)/SUMY                                              KV
14    CONTINUE                                                          KV
      Y1EQ=Y(IM,1)                                                      KV
      Y(IM,2)=Y(IM,2)*E(IM,2)+TERM(2)*(1.0-E(IM,2))                     KV
      Y(IM,3)=Y(IM,3)*E(IM,3)+TERM(3)*(1.0-E(IM,3))                     KV
      Y(IM,1)=1.0-Y(IM,3)-Y(IM,2)                                       KV
C        CALCULATE MURPHREE VAPOUR EFFICIENCY FOR ACETONE               KV
      E(IM,1)=(Y(IM,1)-TERM(1))/(Y1EQ-TERM(1))                          KV
      DO 15 I=1,3                                                       KV
      FK(I)=Y(IM,I)/X(IM,I)                                             KV
      IF (X(IM,I).EQ.0.0) FK(I)=1.0E-20                                 KV
15    CONTINUE                                                          KV
      IF (ITYPE.NE.1) T1=TK-273.0                                       KV
      RETURN                                                            KV
      END
```

```
      FUNCTION ENTLIQ (TEMP,FLOWL,X1,X2)                               EL
C***********************************************************************EL
C                                                                       EL
C        CALCULATE TOTAL LIQUID STREAM ENTHALPY ( KCAL /HR )            EL
C        HEAT CAPACITY DATA TAKEN FROM * CRITICAL TABLES*               EL
C        HEAT OF MIXING DATA TAKEN FROM * CRITICAL TABLES*              EL
C        AND POLYNOMIAL LEAST SQUARES FITTED .                          EL
C                                                                       EL
C        X1 MOLE FRACTION ACETONE, X2 MOLE FRACTION METHANOL            EL
C                                                                       EL
C***********************************************************************EL
C        LIQUID ENTHALPY, 0 DEG REFERENCE ( KCAL/GM MOLE)               EL
C        ENTHALPY OF ACETONE                                            EL
      HACEL(T)=30.6E-3*T                                                EL
C        ENTHALPY OF METHANOL                                           EL
      HMETL(T)=19.74E-3*T                                               EL
C        ENTHALPY OF WATER                                              EL
      HWATL(T)=18.016E-3*T                                              EL
C        HEAT OF MIXING (KCAL/ GM. MOLE)                                EL
C        HEAT OF MIXING FOR METHANOL - ACETONE                          EL
      HMIXMA(XC)=2.01772E-3-0.753418*XC+0.94408*XC**2-0.192305*XC**3    EL
C        HEAT OF MIXING FOR WATER - METHANOL                            EL
      HMIXWM(XC)=6.05874E-3+0.171893*XC+0.646148*XC**2-0.812034*XC**3   EL
C        HEAT OF MIXING FOR WATER - ACETONE                             EL
      HMIXWA(XC)=-6.00506E-3+0.268503*XC-2.1499*XC**2+5.30655*XC**3-3.50EL
     1732*XC**4                                                         EL
      X3=1.0-X1-X2                                                      EL
      ENTLIQ=HACEL(TEMP)*X1+HMETL(TEMP)*X2+HWATL(TEMP)*X3               EL
      IF (X1.EQ.1.0.OR.X2.EQ.1.0.OR.X3.EQ.1.0) GO TO 1                  EL
      X1B=X1+X2                                                         EL
      X1A=X2/X1B                                                        EL
      X2B=X3+X2                                                         EL
      X2A=X3/X2B                                                        EL
      X3B=X3+X1                                                         EL
      X3A=X3/X3B                                                        EL
      ENTLIQ=ENTLIQ-X1B*HMIXMA(X1A)-X2B*HMIXWM(X2A)-X3B*HMIXWA(X3A)     EL
1     ENTLIQ=ENTLIQ*FLOWL                                               EL
      RETURN                                                            EL
      END                                                               EL
      FUNCTION ENTVAP (TEMP,FLOWV,Y1,Y2)                                EV
C***********************************************************************EV
C                                                                       EV
C        CALCULATE TOTAL VAPOUR STREAM ENTHALPY ( KCAL/HR )             EV
C                                                                       EV
C        DATA FOR HEAT OF VAPOURIZATION OBTAINED FROM CRITICAL TABLES   EV
C        MOLAR VAPOUR ENTHALPY CALCULATED AND LEAST SQUARES FITTED      EV
C        SPECIFIC HEAT ZERO REFERENCE FOR LIQUID AT 0 DEG. C            EV
C        Y1 MOLE FRACTION ACETONE, Y2 MOLE FRACTION METHANOL            EV
C                                                                       EV
C***********************************************************************EV
C        SPECIFIC HEAT OF WATER                                         EV
      HWATV(T)=10.7278+9.31074E-3*T-1.47991E-5*T**2                     EV
C        SPECIFIC HEAT OF ACETONE                                       EV
      HACEV(T)=7.83444+2.26068E-2*T-4.89657E-5*T**2                     EV
C        SPECIFIC HEAT OF METHANOL                                      EV
      HMETV(T)=9.10835+1.40099E-2*T-7.98048E-5*T**2                     EV
      ENTVAP=(HACEV(TEMP)*Y1+HMETV(TEMP)*Y2+HWATV(TEMP)*(1.0-Y1-Y2))*FLOEV
     1WV                                                                EV
      RETURN                                                            EV
```

```
      SUBROUTINE CVGP (II,NKT,X1,IMAX,XMPER,XMNRM)                      CV
C*********************************************************************CV
C                                                                      CV
C        CONVERGENCE PROMOTION ROUTINE                                 CV
C        DOMINANT EIGENVALUE METHOD AS MODIFIED BY MASATOSHI NISHIO    CV
C        JUNE,1974                                                     CV
C                                                                      CV
C*********************************************************************CV
      DIMENSION DX(11,100), X1(1), X2(100), XMU(10), XA(100), XX(100), XCV
     1O(100), ERR(100), XC(100), SCPD(55), YSP(55), XSP(10)            CV
      DATA EGT,EPS,TR/0.05,0.1,0.0/                                    CV
      IF (II.GT.1) GO TO 2                                             CV
      KO=1                                                             CV
      JSM=1                                                            CV
      LK=JSM+2                                                         CV
      JJ=1                                                             CV
      XP=EPS*0.01                                                      CV
      DO 1 K1=1,NKT                                                    CV
      XA(K1)=X1(K1)                                                    CV
      X2(K1)=X1(K1)                                                    CV
1     CONTINUE                                                         CV
      GO TO 43                                                         CV
C                                                                      CV
2     KO=KO+1                                                          CV
      IF (II.GT.(JSM+1)) GO TO 4                                       CV
      DO 3 K1=1,NKT                                                    CV
      K2=JSM-II+2                                                      CV
      DX(K2,K1)=X1(K1)-X2(K1)                                          CV
      X2(K1)=X1(K1)                                                    CV
3     CONTINUE                                                         CV
      GO TO 39                                                         CV
4     IF (KO.GT.(JSM+2).A.JJ.EQ.0) GO TO 6                             CV
C     CALCULATE INITIAL SET OF SCALAR PRODUCTS.                        CV
C     MATRIX OF SCALAR PRODUCTS IS STORED AS VECTOR SCPD(I) IN LOWER   CV
C     TRIANGULAR FORM.                                                 CV
      K4=0                                                             CV
      DO 5 K1=1,JSM                                                    CV
      DO 5 K2=1,K1                                                     CV
      K4=K4+1                                                          CV
      SCPD(K4)=0.                                                      CV
      DO 5 K3=1,NKT                                                    CV
      SCPD(K4)=DX(K1,K3)*DX(K2,K3)+SCPD(K4)                            CV
5     CONTINUE                                                         CV
C          START THE PROMOTION CALCULATIONS                            CV
C     COMPUTE NEW VALUES OF SCALAR PRODUCTS)                           CV
6     DO 7 K=1,JSM                                                     CV
      XSP(K)=0.                                                        CV
7     CONTINUE                                                         CV
      SUMDEL=0.                                                        CV
      SUMSQ=0.                                                         CV
      DO 10 K1=1,NKT                                                   CV
      SUMSQ=SUMSQ+X1(K1)**2                                            CV
      DEL=X1(K1)-X2(K1)                                                CV
      SUMDEL=SUMDEL+DEL*DEL                                            CV
      DO 8 K=1,JSM                                                     CV
      XSP(K)=XSP(K)+DEL*DX(K,K1)                                       CV
8     CONTINUE                                                         CV
      X2(K1)=X1(K1)                                                    CV
      DO 9 K=1,JSM                                                     CV
```

```
      K2=JSM-K+2                                                        CV
      DX(K2,K1)=DX(K2-1,K1)                                             CV
9     CONTINUE                                                          CV
      DX(1,K1)=DEL                                                      CV
10    CONTINUE                                                          CV
C                                                                       CV
      IF (KO.LT.LK) GO TO 30                                            CV
C                                                                       CV
C     CALCULATE APPARENT SOLUTION                                       CV
      IF (JSM-2) 11,12,13                                               CV
11    XMU(1)=-XSP(1)/SCPD(1)                                            CV
      GO TO 16                                                          CV
12    DEN=SCPD(3)*SCPD(1)-SCPD(2)**2                                    CV
      XMU(1)=(XSP(2)*SCPD(2)-XSP(1)*SCPD(3))/DEN                        CV
      XMU(2)=(XSP(1)*SCPD(2)-XSP(2)*SCPD(1))/DEN                        CV
      GO TO 16                                                          CV
13    NSP=(JSM*(JSM+1))/2                                               CV
      DO 14 K1=1,JSM                                                    CV
      XMU(K1)=-XSP(K1)                                                  CV
14    CONTINUE                                                          CV
      DO 15 I=1,NSP                                                     CV
      YSP(I)=SCPD(I)                                                    CV
15    CONTINUE                                                          CV
      CALL SYMSOL (YSP,JSM,IERR,XMU)                                    CV
16    CONTINUE                                                          CV
      SUM=1.                                                            CV
      DO 17 K1=1,JSM                                                    CV
      SUM=SUM+XMU(K1)                                                   CV
17    CONTINUE                                                          CV
C                                                                       CV
      DO 20 K1=1,NKT                                                    CV
      XNUM=0.                                                           CV
      DO 19 K=1,JSM                                                     CV
      XSUM=0.                                                           CV
      DO 18 K2=K,JSM                                                    CV
      XSUM=XSUM+XMU(K2)                                                 CV
18    CONTINUE                                                          CV
      XNUM=XNUM+XSUM*DX(K,K1)                                           CV
19    CONTINUE                                                          CV
      XX(K1)=X1(K1)-XNUM/SUM                                            CV
20    CONTINUE                                                          CV
      OO=JSM*100                                                        CV
      PRINT 21, OO,(XX(K1),K1=1,NKT)                                    CV
21    FORMAT (1H ,8F15.5)                                               CV
      JJ=0                                                              CV
C                                                                       CV
C     ERROR CALCULATION ... 0 ORDER                                     CV
      ERROR=0.                                                          CV
      DO 23 K1=1,NKT                                                    CV
      ERR(K1)=DX(1,K1)                                                  CV
      DO 22 K3=1,JSM                                                    CV
      K4=K3+1                                                           CV
      ERR(K1)=ERR(K1)+XMU(K3)*DX(K4,K1)                                 CV
22    CONTINUE                                                          CV
      ERROR=ERROR+ERR(K1)*ERR(K1)                                       CV
23    CONTINUE                                                          CV
      PRINT 24, ERROR,(XMU(I),I=1,JSM)                                  CV
24    FORMAT (1H ,8F15.5)                                               CV
```

```
      IF (ERROR.LT.ERS) GO TO 25
      JSM=JSM+1
      JJ=1
      LK=JSM+2
   25 IF (JSM.EQ.1.O.JJ.EQ.1) GO TO 28
      DO 26 K1=2,JSM
      K3=(JSM-K1+2)*(JSM-K1+3)/2
      DO 26 K2=K1,JSM
      K4=K3-K2+K1
      K5=K4-JSM+K1-2
   26 SCPD(K4)=SCPD(K5)
      DO 27 K1=2,JSM
      K2=(K1-1)*K1/2+1
      SCPD(K2)=XSP(K1-1)
   27 CONTINUE
   28 CONTINUE
      SCPD(1)=SUMDEL
      RMSERR=SQRT(SUMDEL/SUMSQ)*100.
      JK=JSM
      IF (JJ.EQ.1) JK=JSM-1
      PRINT 29, II,KO,RMSERR,(XMU(K),K=1,JK)
   29 FORMAT (1H ,/* ON NO.  *I4,*-AND KOUNT = *I4* RMS. ERROR = *E12.5*
     1 PERCENT. */1H ,*EIGENVALUES*/(1X,9F10.5))
      XRERR=0.1E-10
      DO 30 K1=1,NKT
      IF (XX(K1).LE.0.1E-10) GO TO 30
      XXX=ABS((XX(K1)-X1(K1))/XX(K1))*100.
      IF (XXX.GT.XRERR) XRERR=XXX
   30 CONTINUE
      IF (RMSERR.GT.XMNRM.O.XRERR.GT.XMRER) GO TO 31
      II=0
      RETURN
   31 IF (II.LE.IMAX) GO TO 33
      II=-1
      PRINT 32
   32 FORMAT (1H0,10X,*ITERATION WAS NOT CONVERGED*)
      RETURN
   33 IF (KO.LT.LK) GO TO 39
      IF (KO.EQ.LK.O.JJ.EQ.1) GO TO 37
      XSUM=0.
      DO 34 K1=1,NKT
      IF (ABS(XX(K1)).LE.0.1E-10) GO TO 34
      E=(XX(K1)-XO(K1))/XX(K1)
      XSUM=E*E+XSUM
   34 CONTINUE
      XSUM=SQRT(XSUM)
      IF (XSUM.GT.EGT) GO TO 37
      DO 35 K1=1,NKT
      IF (XX(K1).LT.0.1E-10) XX(K1)=0.1E-10
      X2(K1)=XX(K1)
      XA(K1)=XX(K1)
   35 CONTINUE
      KO=1
      PRINT 36, JSM,II
   36 FORMAT (/2X,*PROMOTION STEP TAKEN OF ORDER *I1,* AT LOOP NO. *I4)
      GO TO 41
   37 DO 38 K1=1,NKT
      XO(K1)=XX(K1)
```

```
38     CONTINUE                                                                 CV
39     DO 40 K1=1,NKT                                                           CV
       XA(K1)=X2(K1)*(1.+TR)-TR*XA(K1)                                          CV
       IF (XA(K1).LT.0.1E-10) XA(K1)=0.1E-10                                    CV
40     CONTINUE                                                                 CV
       IF (TR.EQ.0) GO TO 43                                                    CV
41     DO 42 K1=1,NKT                                                           CV
       X1(K1)=XA(K1)                                                            CV
42     CONTINUE                                                                 CV
43     RETURN                                                                   CV
       END                                                                      CV
```