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PRELIMINARY DESIGN AND
D.D.C. OF AN
EXTRACTIVE DISTILLATION COLUMN

by

W. D. JACKSON, B. ENG.

PRELIMINARY DESIGN AND
DIRECT DIGITAL CONTROL
OF AN
EXTRACTIVE DISTILLATION COLUMN

by

W. D. JACKSON, B. ENG.

A Thesis
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July, 1974

Abstract

As the first stage in a long term process control and optimization study, a fourteen stage extractive distillation column was re-designed, re-built and evaluated. Under the operating conditions studied, the overall column efficiency was found to be approximately 60%. Recommendations for design modifications to improve column capacity and operation were presented.

A control computer was interfaced to the process and a computer monitor/control software package was developed. The computer was found to be very useful as an aid to the manual operation of the extractive distillation column. The next stages in this study will involve the continuous computer control of solvent and feed stream flowrates and temperatures, and reboiler liquid level. The executive program and parameter change routine developed during this project were designed to be very flexible in format. so that as more programs are written for the next stages of this study, they may be easily added to the existing software package.

Acknowledgements

I would like to thank Dr. J.D. Wright and Dr. T.W. Hoffman for their many suggestions and discussions throughout this project. I would also like to thank several fellow graduate students, especially Jean-Pierre Tremblay, for their help and encouragement.

Words cannot express my thanks to my parents for their support throughout the years leading to this degree, nor my appreciation to my wife, Pat, for her endurance.

Lastly, I would like to thank the School of Graduate Studies at McMaster University and the National Research Council for their financial assistance.

To "Pepper"

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1. Introduction

For the safe and economic operation of most chemical processes, automatic control is a necessity. For many years, this has been accomplished with analog control devices and computers, but this type of computer has limited uses for more complex control schemes and on-line optimization techniques. Early digital computers could handle these more complex systems, but were too expensive, when compared to the improvements in process operation, to install for normal applications. With the development of small, relatively inexpensive digital computers, the advantages of direct digital control are rapidly increasing. These small computers can be installed in the plant for direct control of the process or simply set-point control, and can be linked with a larger supervisory computer for implementation of the on-line process adjustments needed for optimization of the total plant production. This project represents the first stage in a long-term, process control and optimization study of a complex process.

1.1 Choosing a Process

The process to be chosen for this project had to be reasonably complex so that several projects in the fields of process control, optimization and simulation could be supported by the equipment. The research projects would be especially useful if the process was common in industry, but not in research laboratories, so that new technology could be developed and applied to industrial systems.

A constraint on the choice of a process was that the equipment had to be used for an undergraduate teaching laboratory, to make better use of the project expenditures. As such, the process had to operate on fairly basic principles, the raw materials used by the process had to be safe, inexpensive and readily obtainable, and the process had to be contructed of glass, as much as possible.

Based on these requirements, the process chosen for this project was extractive distillation, using water as the solvent to separate an acetone-methanol feed mixture. This process does operate on basic vapour-liquid equilibrium relationships, although they are complicated by the ternary system, and the equipment can be constructed mainly of glass, to aid the teaching aspect. The process is quite complicated, so it will present a challenge as a graduate research project for several different projects. The ternary system, acetone-methanol-water, was chosen because the materials are fairly safe (non-explosive), inexpensive and readily obtainable. The literature contains an extensive amount of physical property data in the forms of tables and graphs [5,9,10,11], which are necessary for hand calculations, and several analytical methods and equations for determining equilibrium compositions [3,14, 26], which are needed for computer simulation studies.

1.2 Distillation Column Control

The literature contains an extensive amount of information on distillation column control, including several recent review papers such as that by WILLIAMS [33]. Because of these reviews and the volume of material, this survey gives only a brief outline of the general control approach, some problems in distillation column control, and previous azeotropic and extractive distillation control studies.

General Control Approach

The early methods of distillation column control used a feedback control loop on one product stream, with relatively no control on the other product stream. This type of control would maintain the controlled variable about its setpoint, but because of the size and complexity of the column, large disturbances in the feed rate or composition would cause long periods of poor quality product, as the controller tried to correct for the disturbance as its effects showed up at the measured variable location. Feedforward control was thus tried in attempts to correct for these disturbances as they occurred.

One of the earliest attempts at feedforward control of a binary distillation column was that by LUPFER and PARSONS [16].The feed flow measurement was used to determine the heat input to the feed heater and the setpoint of the reflux controller.Feed flow and composition measurements, along with product stream composition specifications,were used to determine the bottom product flow setpoint.Manual zero correction of this setpoint was implemented after periodic laboratory analysis of the product streams,to compensate for inaccuracies in measurements and computer calculations.The boil-up rate was determined by a reboiler level controller.

LUYBEN and GERSTER [17],undertook pilot column and analog simulation studies for the feedforward control of a binary distillation system.Using energy and mass balances,the boil-up rate and reflux rate were adjusted relative to feed flow and composition measurements,to maintain a desired bottom product composition.They found that feedforward control alone did not work well,because the control model was not exact,no adjustments were made for unmeasurable disturbances, and physical and process limitations inhibited full control action.They concluded that feedforward-feedback control would result in better column operation.

BROSILOW and HANDLEY [2],examined control of the overhead product from a rectifying column,using control tray temperature measurements to adjust the product flowrate.Their pilot column represented the rectifying section of a distillation column, using the vapour from a kettle boiler as a feed stream.

LUYBEN [18], continued his control studies by combining feedback and feedforward control, using an analog simulation of an ideal binary distillation column. Addition of feedforward control to the feedback control scheme reduced the offset in product stream composition resulting from changes in feed composition or flow. The measured variables for the feedback control loop were the temperatures of intermediate trays near the controlled end of the column.

SHINSKY [28], looked at the proper pairing of variables to obtain stable column control. He suggested that material balance control, rather than energy balance control, would lead to the best product stream control. He recommended that, for two-product-composition control, a "relative gain" term should be evaluated to determine the interaction in the control loops.

SHUNTA and LUYBEN [30], looked at sampled data control of a binary distillation column for setpoint and load disturbances, using a simulation model of the column. They found that the controller designed for a particular disturbance worked well for that disturbance, but not for the other. They examined the method of switching control schemes on-line, when the type of disturbance changed, but found that a dual controller, which combined the two control schemes in one package, gave better control, especially since the switching period was often critical.

WOOD and PACEY [34], also evaluated feedforward, feedback, and combined feedforward-feedback control of a binary distillation column. They examined overhead product composition control obtained by adjusting the reflux rate and boil-up rate in response to changes in feed flow only. The feedback variable was overhead composition instead of tray temperature. Addition of the feedforward control loop resulted in improved product composition. They found, however, that the best way to determine the feedforward controller gains was by experiment, and recommended that further research be done to improve the reliability of the prediction of these gains.

HU and RAMIRIZ [15], looked at multi-variable proportional-integral control of linear and non-linear models of a binary distillation column, while DUYFJES and VAN DER GRINTEN [8], used mathematical models to examine the problems and advantages of various control techniques which could be used for any distillation column control problem.

DAVISON [7], presented a method for the systematic design of control systems, and used a binary distillation column as an example application. Comparison of the controllers developed by this technique and the conventional approach, by applying them to a simulation model of the column, showed that the systematic design controller maintained better control over the controlled variables, overhead and bottom product compositions and column pressure. The design controller also had the advantage of being simple to implement.

Most of the literature to date has dealt with distillation column control studies using simulation models and/or pilot columns. NISENFELD and MIYASAKI [24], however, documented three plant applications of simple feedforward control loops applied to distillation columns. All three examples showed large returns and high reliability for small investments, verifying the value of this type of control research.

Example Problems in Distillation Column Control

One of the major problems in feedback control of both product streams in a distillation column is the control instabilities brought about by control loop interactions. DAVISON [6], has mathematically examined the interactions between several pairs of control loops in a binary distillation column. He predicted that severe interactions exist between several of these pairs, and that moderate interactions existed between most of the other pairs. LUYBEN [20], used a linear model of a binary distillation column to design the decoupling elements required for feedback control of the product streams, and tested their effectiveness by digital simulation of the non-linear model. He found that ideal decoupling lead to unstable control, while simplified decoupling gave very good, stable control. NIEDERLINSKI [23], however, found that by not decoupling the control loops, the resulting interacting control loops gave better disturbance attenuation than the non-interacting design. In addition to improved response, the interacting design was less expensive to implement. CHANGLAI and WARD [4], did use decoupling to control the compositions of the product streams of a distillation column,and found that the feedback controllers maintained the product compositions at their set-points.The controllers manipulated the reflux rate and boil-up rate in response to changes in product compositions,brought about by the unmeasured disturbances,feed rate and composition. It appears from the contradictions in these publications that more research in this area is needed,in addition to research in the general field of distillation control.

Another problem in distillation column control is that of inverse response of bottom product composition to vapour boil-up rate.LUYBEN [19],examined this problem and found that lead/lag elements improved the feedback control of the bottom product composition,while dynamically perfect feedforward control was impossible because the controller was open-loop unstable.In a later publication,LUYBEN and SHUNTA [21],compared sampled data control and continuous proportional-integral-derivative (PID) control methods for the feedback control of a distillation column with this same problem.They concluded that the sampled data controller worked better than the continuous PID controller,using a digital simulation model of the process.

Azeotropic and Extractive Distillation Control Studies

Only one publication on extractive distillation column control was found,and this was the patent by MCNEILL and SACKS [22],for a general control technique which could be applied to any distillation process. The control stategy was to adjust and control the feed rate to maintain the desired overhead or bottom product stream composition.

Two other publications dealt with the control of an azeotropic distillation column. NISENFELD and STRAVINSKI [25], looked at feedforward control of the overhead product in the azeotropic distillation of an acetic acid/water feed, but neglected control of the bottom product since it was essentially pure acetic acid under most operating conditions. They found that feedforward control was adequate for feed flow changes, but that feedback control was needed to handle feed composition changes. This feedback controller adjusted the reflux-to-feed ratio, based on a control tray temperature. WENKOFF [32], examined multivariable control of an azeotropic distillation column, but in this case, the primary objective was control of the bottom product composition, since the overhead product was purified in a second column. His multivariable controller combined integral output feedback action with feedforward control based on material and energy balances. His extensive literature survey revealed that there has been very little experimental work in multi-variable control to date, and that attempts to extend the usual single loop techniques to multi-variable control have been rather poor. He suggested that any meaningful development of optimal control theory must be directly coupled to real physical processes.

Comments on Extractive Distillation Process Control

The complete control package for the extractive distillation process will be very large and complex, since control routines are required for the solvent recovery column, the extractive distillation column, and several stream flowrates and temperatures. As well as controllers, a handler must be included to obtain and interpret stream composition measurements from a process gas chromatograph, and the computer must operate in real time, allowing on-line changes in program parameters.

The solvent recovery column controller will be based on the binary distillation column control schemes documented in the literature, but the controller must maintain tight control over both product streams and must be able to handle variations in acetone concentration in the feed stream and in the column. Tight control is necessary because the overhead product is the finished methanol product, while the bottom product is recycled to the solvent stream of the extractive distillation column. Variations in the composition of this stream will have a detrimental effect on the control of this column. Variations in acetone concentration in the solvent recovery column will affect the column controller, if the controller uses column temperatures as input variables, since composition has a marked effect on boiling point, and thus column temperature. This could be avoided if overhead product composition is used as the feedback variable.

The extractive distillation column controller will be more complex than a binary distillation column controller because;

1. it is a special case of multi-component distillation complicated by the variation in key component volatilities through the column, brought about by the varying effect of solvent concentration
2. the column has two extra control variables to determine, solvent flowrate and temperature, both of which have a large affect on column operation
3. very good control over both product streams is required, unlike [25] and [32], because the overhead is the finished acetone product, while any acetone in the bottom product stream will contaminate the finished methanol product stream from the solvent recovery column

It is obvious from this discussion that control of the extractive distillation process will be a very complex problem which may be approached by a variety of methods.

1-3 Project Objectives and Course of Action

This project was the first stage in a long term process control and optimization study of the extractive distillation process, and, as such, the objectives were to;

1. design and build the extractive distillation column
2. evaluate the performance and overall efficiency of the column

3. interface the process with the control computer
4. develop and implement a computer software package for process monitoring and simple control

Since this project dealt with the preliminary design of the extractive distillation column for evaluation purposes, the column design used inexpensive or on-hand equipment whenever possible, to keep expenditures to a minimum. Following construction of the column, the equipment had to be operated manually to find and correct, if possible, design problems, before proceeding with the evaluation step.

To evaluate column efficiency, an analytical technique had to be developed to measure product stream compositions. Evaluation of column efficiency would be made by measuring input and output stream compositions and flowrates at steady-state and calculating the number of theoretical stages in the column. To aid in the steady-state operation of the column, the control computer had to be interfaced to the process and the monitor/control package developed and implemented.

2. Extractive and Azeotropic Distillation

This chapter begins with a brief discussion on basic vapour-liquid equilibrium relationships,with emphasis on the application to extractive and azeotropic distillations.This is followed by a general comparison of the two distillation techniques,and a description of the graphical method for designing an extractive distillation process.

2.1 Basic Equilibrium Theory

For an ideal system,the composition of the vapour in equilibrium with the liquid is a function of temperature and pressure only.This equilibrium relation may be represented by equation 2.1.a ,where x_i and y_i are the mole fractions of component i in the liquid and vapour phases respectively,and K_i^o is the equilibrium constant. K_i^o is a function of pressure and temperature,independent of solution composition.

$$y_i = K_i^o x_i \qquad 2.1.a$$

The process of distillation separates components by virtue of inequalities in volatilities.Thus,the ratio of the volatilities is of more importance for separation than the actual volatilities of the components.This ratio can be represented by the ratio of equilibrium constants and is called the relative volatility.Equation 2.1.b expresses the relative volatility of component i to component j.

$$\alpha_{ij} = K_i^o/K_j^o = (y_i/y_j)/(x_i/x_j) \qquad 2.1.b$$

For non-ideal systems, the component volatilities are functions of temperature and pressure, plus composition. To account for this dependence on composition, a correction factor, the activity coefficient (γ_i), is applied to the ideal equilibrium relation to give the non-ideal equilibrium relation, equation 2.1.c.

$$y_i = \gamma_i K_i^o x_i \qquad 2.1.c$$

The relative volatility of non-ideal systems is also a function of composition, and is defined as

$$\alpha_{ij} = \frac{\gamma_i K_i^o}{\gamma_j K_j^o} = \frac{y_i/y_j}{x_i/x_j} \qquad 2.1.d$$

The distillation process is simply a series of equilibrium stages connected by their input/output vapour and liquid streams. By equation 2.1.d, the ratio of component i to component j will be greater in the vapour phase at equilibrium than in the liquid phase if $\alpha_{ij} > 1$. Thus, to separate components i and j by simple distillation, the relative volatility α_{ij} must be greater than unity for all ratios x_i/x_j. If $\alpha_{ij}=1$ for some ratio, the component ratios in the liquid and vapour phases will be the same (an azeotropic composition) and the components cannot be separated by simple distillation. However, the relative volatility is actually a function of component activity coefficients rather than the composition. Therefore, if another component, not already present in the system, can be found which, by its addition, changes γ_i and γ_j by differing amounts, then α_{ij} will also be changed. By the proper choice of this extra component, α_{ij} may be greater than unity for all x_i/x_j ratios, and the i-j azeotrope will be broken. This change in the relative volatility due to the addition of an extraneous compound is the basis for azeotropic and extractive distillation processes.

The acetone-methanol system is a simple example of this type of problem. At atmospheric pressure, this system has an azeotropic composition of 80 mole % acetone. By adding water, the activity coefficients of the key components are changed by differing amounts, as shown in Figure 2.1.A. The effect on the equilibrium compositions is shown in Figure 2.1.B, where the equilibrium concentrations of acetone, on a water-free basis, are plotted for several water concentrations. No azeotrope exists when the water concentration is greater than approximately 30%.

2.2. Comparison of Extractive and Azeotropic Distillation

Because the extraneous component added to a distillation system can be more volatile or less voltaile than the feed, two distinct types of processes exist which use the same basic phenomenon; the differing deviations from ideality caused by the extraneous compound. Representative flow diagrams for these two types of distillation, extractive and azeotropic, are shown in Figures 2.2.A and 2.2.B respectively.

The azeotropic distillation process shown is the

Figure 2.1.A Binary Activity Coefficients

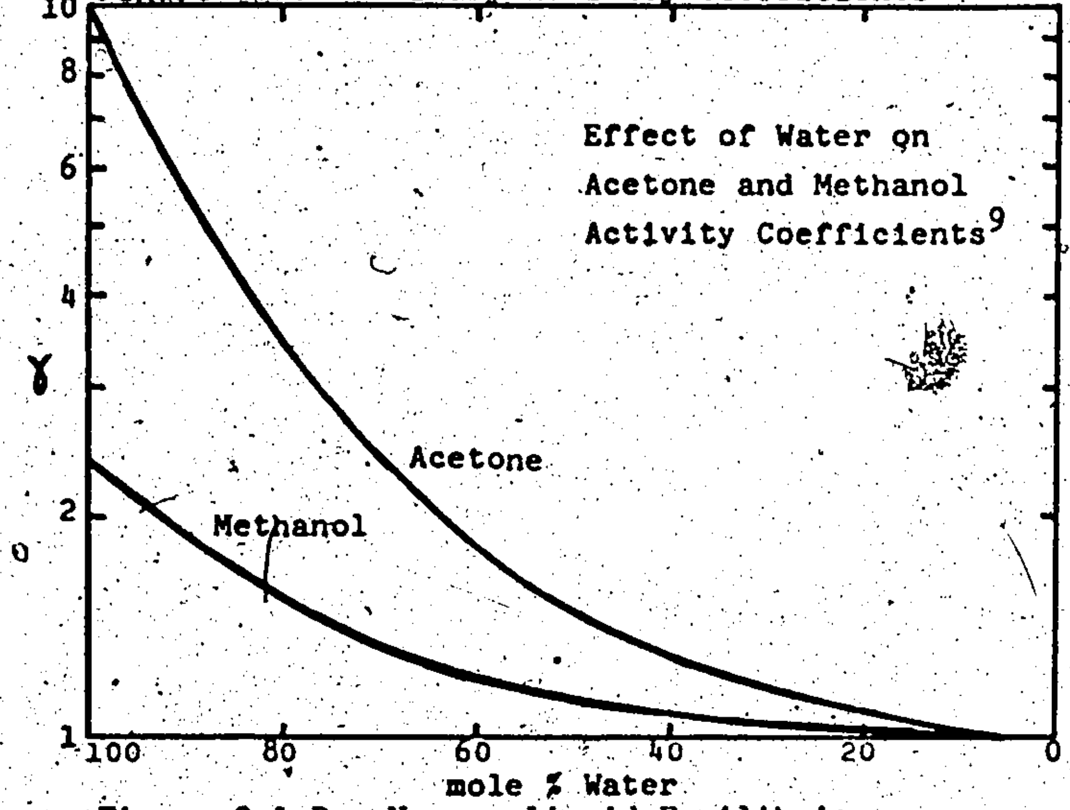


Figure 2.1.B Vapour-Liquid Equilibria

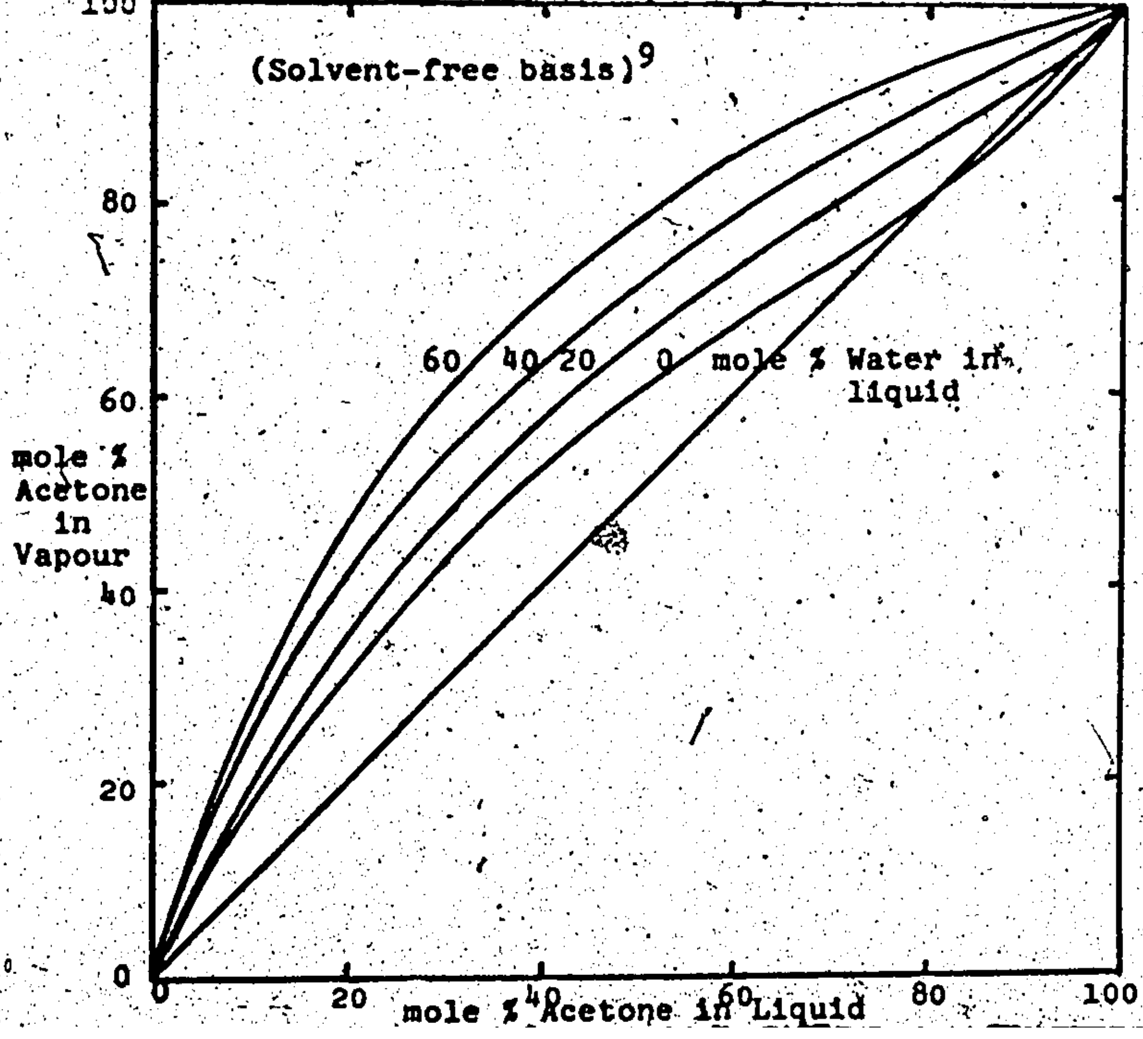


Figure 2.2.A Extractive Distillation

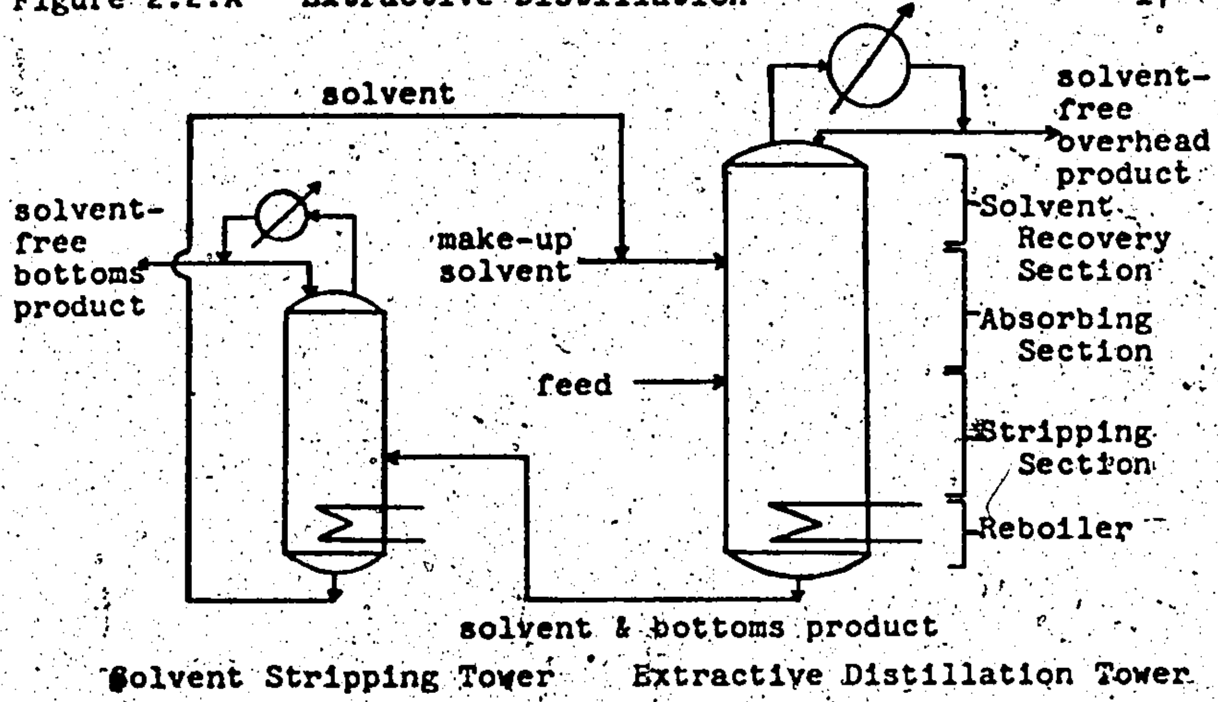
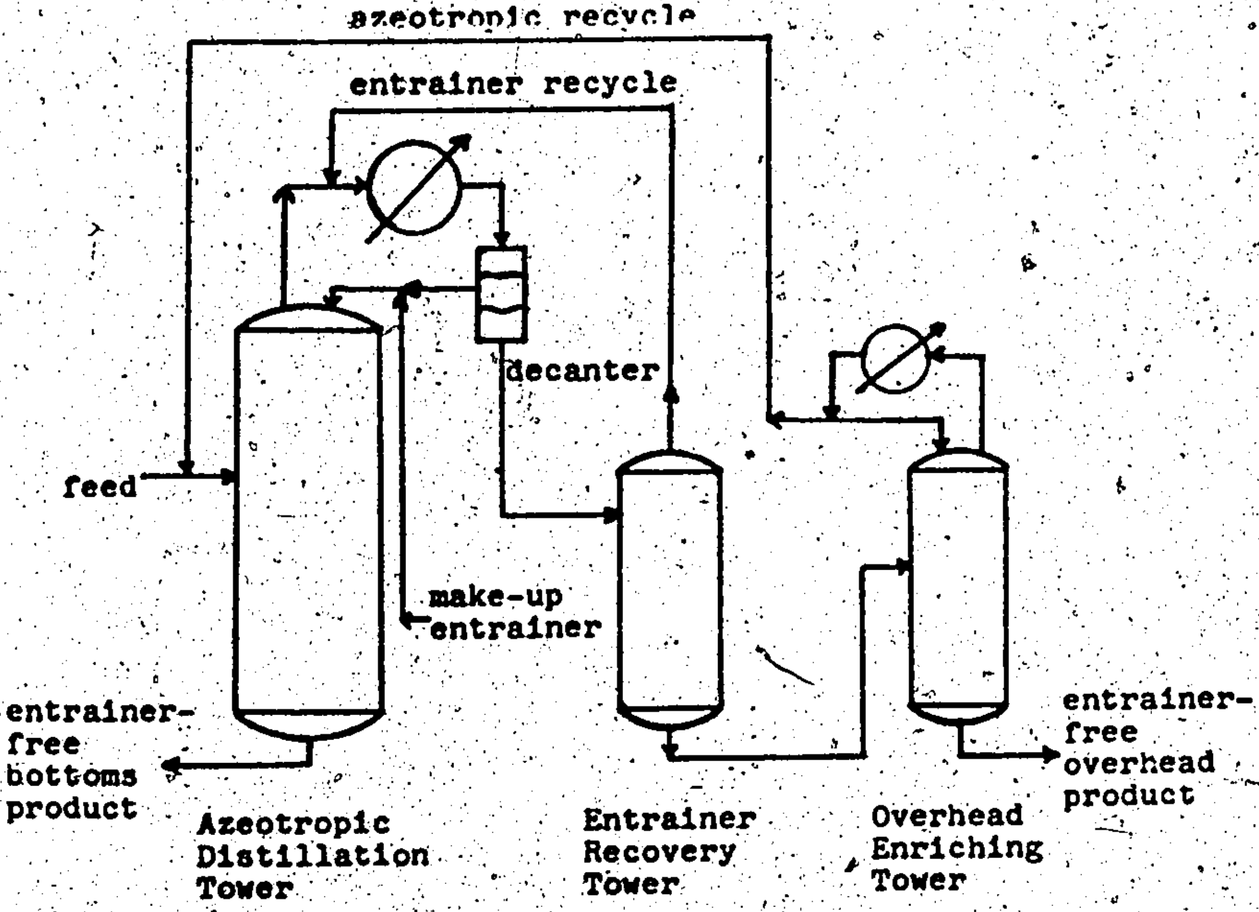


Figure 2.2.B Azeotropic Distillation



separation of water from ethanol,using benzene as the entrainer.As in all azeotropic distillations,the entrainer is as volatile or more volatile than the feed,therefore it also forms an azeotrope with the key components.This azeotrope passes overhead,while the bottoms product is essentially pure ethanol.Recovery of the entrainer from the overhead is complicated by the existence of the azeotrope,and,in this case,requires one distillation tower to recover the benzene, and a second distillation tower to remove the water,recycling the ethanol carried overhead back to the first tower.

Extractive distillation is usually much simpler,as far as solvent recovery is concerned,than azeotropic distillation because the solvent used to effect the separation is less volatile than the feed and does not form an azeotrope with the key components.Because of the low volatility,the net flow of solvent is down the tower,therefore it must be charged near the top.Most of the solvent in the vapour stream leaving the top stage of the absorbing section can be recovered by adding a simple rectifying section to the top of the column (solvent recovery section),while a simple stripping tower serves to recover the solvent from the bottoms product for recycle to the extractive distillation tower.

2.3 Stage Calculations for Extractive Distillation Column Design

To design an extractive distillation column for a ternary system, stage calculations may be carried out on triangular co-ordinate graph paper representing component concentrations. Desired flowrates and compositions of the two input streams and two product streams are found analytically and the streams, designated F (feed), S (solvent), B (bottom product), and D(distillate) are plotted on the graph, as shown in Figure 2.3.B.

In carrying out the stage calculations, it is assumed that the column operates at constant molal vapour flow and each section operates at constant molal overflow. It is also assumed that the liquid and vapour streams leaving a stage are in equilibrium.

The non-key component in extractive distillation is less volatile than the feed and is found in the highest concentration in the reboiler. Stage calculations are thus begun at this stage, and the stages are numbered starting at the bottom. Figure 2.3.A shows the general solution to an extractive distillation column design problem. A total of c stages, excluding the reboiler, are required, with feed and solvent charged to stages a and b, respectively.

By the assumptions of constant molal vapour flow, the molal flowrate of vapour leaving each stage may be determined from the overhead rate and the reflux ratio, $R=L_{c+1}/D$. The composition of the vapour leaving the reboiler may be determined, by the assumption of perfect equilibrium, from ternary equilibrium data, and plotted on the graph as V_0.

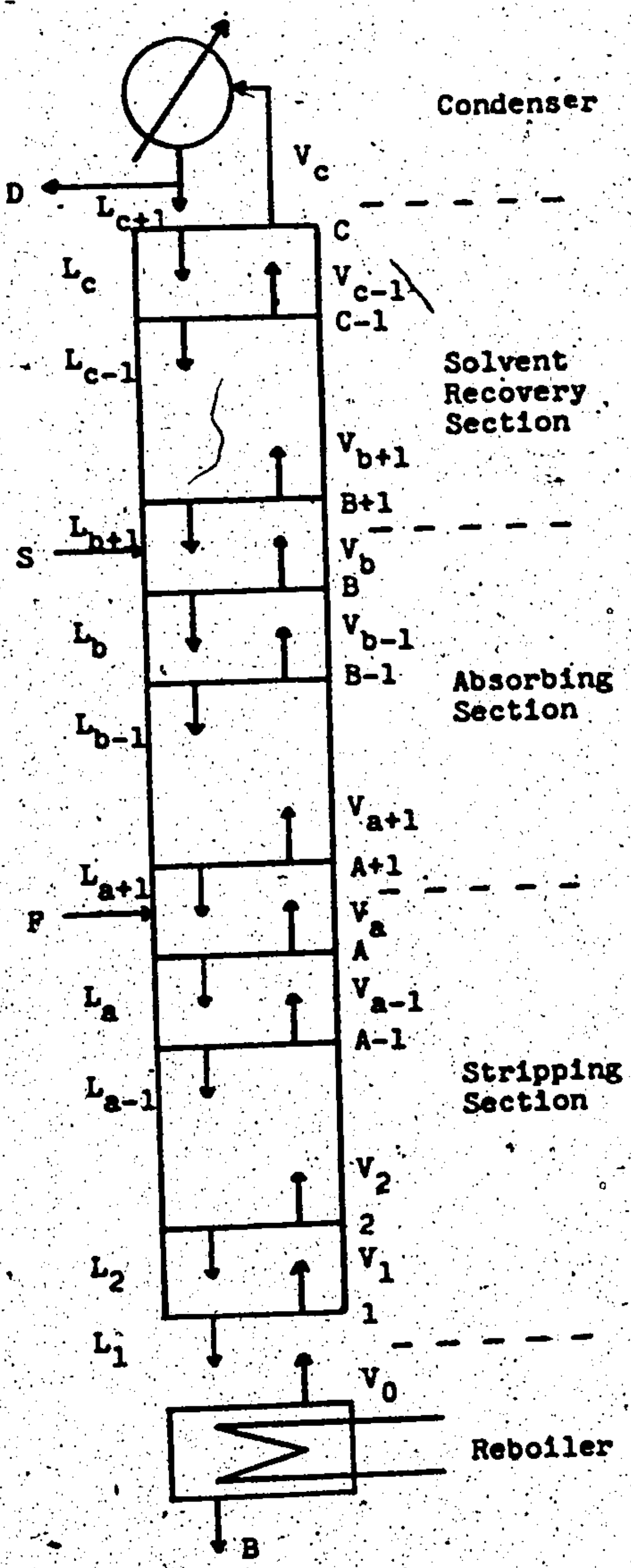
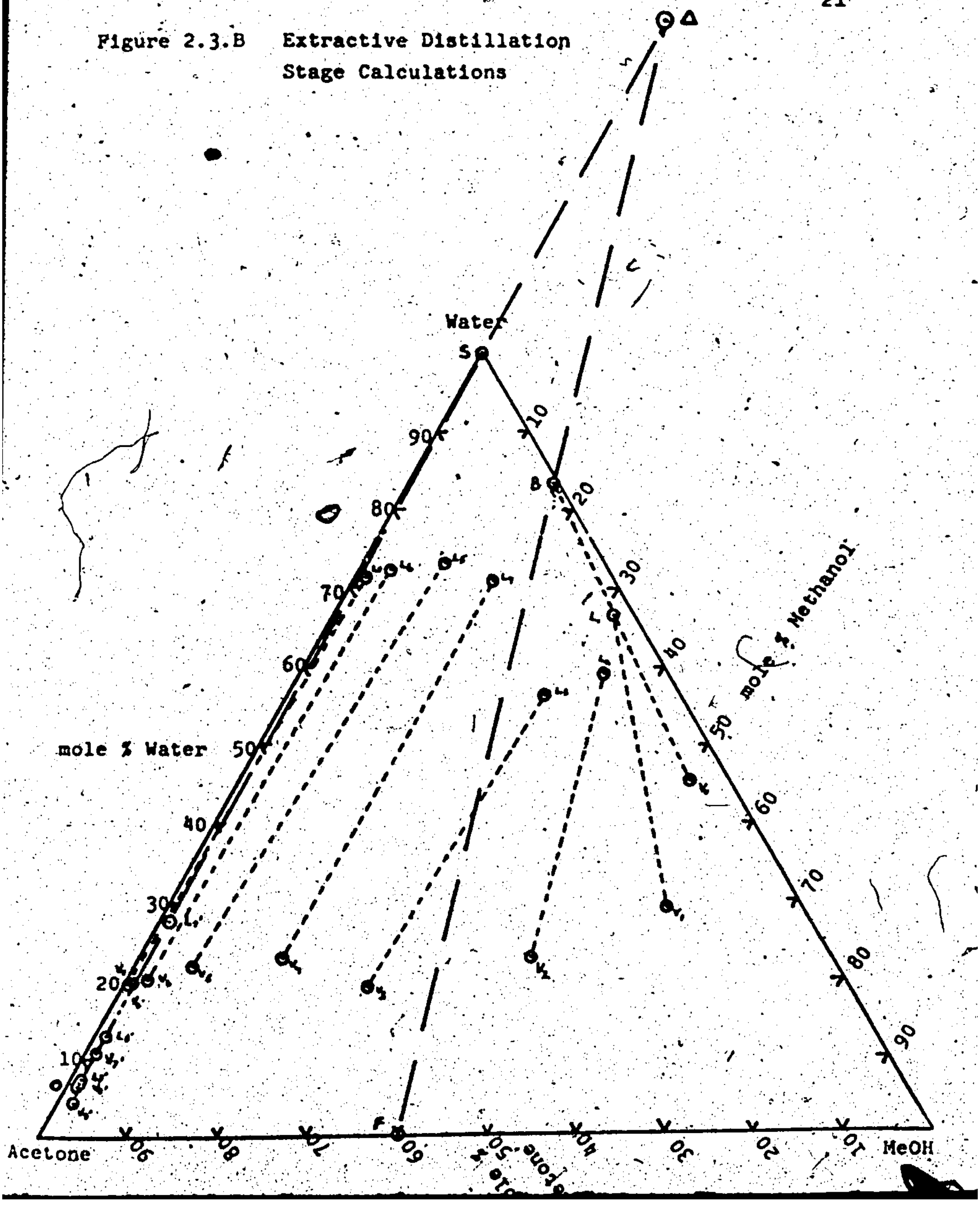


Figure 2.3.A Extractive Distillation Tower Flow Diagram

Figure 2.3.B Extractive Distillation Stage Calculations



By mole balance around the reboiler, the molar flowrate of the liquid stream entering the reboiler, L_1, may be determined graphically, using the Lever Rule, so that L_1 is located on the line joining B and V_0 such that

$$\overline{BL_1} = \frac{V_0}{V_0+B}\,\overline{BV_0} \qquad 2.3.a$$

where V_0 and B are molar flowrates, and $\overline{BL_1}$ and $\overline{BV_0}$ are distances on the graph.

Now that the composition of L_1 is known, the composition of the vapour leaving the first stage, V_1, may be determined from equilibrium data. L_2 is again found graphically, using the same procedure as was used to find L_1, and the procedure repeated until the equilibrium tie-line, $L_a V_a$, crosses the line BF, thus locating the feed stage.

A mole balance around the lower third of the column, from the feed stage down, yields the equation,

$$L_{a+1} = (B-F) + V_a \qquad 2.3.b$$

while a mole balance around the rest of the column yields

$$L_{a+1} = (S-D) + V_a \qquad 2.3.c$$

To satisfy both of these equations, (B-F) and (S-D) must represent the same point on the graph. This point is located by the intersection of the the lines FB and DS at Δ.

Since V_a is known, L_{a+1} may be located on the line joining Δ and V_a, by the Lever Rule.

$$\overline{\Delta V_{a+1}} = \frac{V_a}{B-F+V_a}\,\overline{\Delta V_a} \qquad 2.3.d$$

As in the first set of calculations, V_{a+1} may be determined from the equilibrium data, and the stage calculations carried out across the graph until the equilibrium tie-line, L_bV_b, crosses the line DS, thus locating the solvent stage.

A mole balance around the upper third of the column yields the equation,

$$V_b = D + L_{b+1} \qquad 2.3.e$$

therefore L_{b+1} may be located on the line extending from D through V_b such that

$$\overline{DV}_b = \frac{L_{b+1}}{V_b}\overline{DL}_{b+1} \qquad 2.3.f$$

By substitution and assumption of equimolal flows, this may be reduced to

$$\overline{DL}_{b+1} = \frac{R+1}{R}\overline{DV}_b \qquad 2.3.g$$

Calculations are carried on until the vapour from stage c is approximately the same as the desired overhead product composition.

In some design cases, V_b is located very close to a binary composition on the graph, as it is in Figure 2.3.B. When DV_b is extended to locate L_{b+1}, the extension crosses the axis, giving a negative concentration of one component. To approximate the overhead stage calculations, the intersection of $V_{b-1}V_b$ with DS can be used as the input vapour stream, V_b', to the solvent recovery section.

The graphical analysis performed in Figure 2.3.B was the design calculation for the equipment constructed in the laboratory.Table 2.3.A lists the flows and compositions of the four streams,based on a reflux ratio of 2.Table 2.3.B summarizes the theoretical stage calculations and lists the actual number of stages used in each section of the extractive distillation column.

To complete the equipment design,the number of stages in the solvent recovery column must be calculated.This was approximated by assuming a binary system of methanol/water, and carrying out stage calculations on an x-y diagram,as in Figure 2.3.C,Table 2.3.C summarizes the results of this analysis.

Table 2.3.A

Stream Data for Extractive Distillation Stage Calculations

Stream	Flow g-mole/hr	Acetone mole %	Methanol mole %	Water mole %
F	10.0	60.0	40.0	0.0
S	20.5	0.0	0.0	100.0
D	6.4	92.0	1.7	6.3
B	24.1	0.35	16.15	83.5

Table 2.3.B

Results of Extractive Distillation Stage Calculations

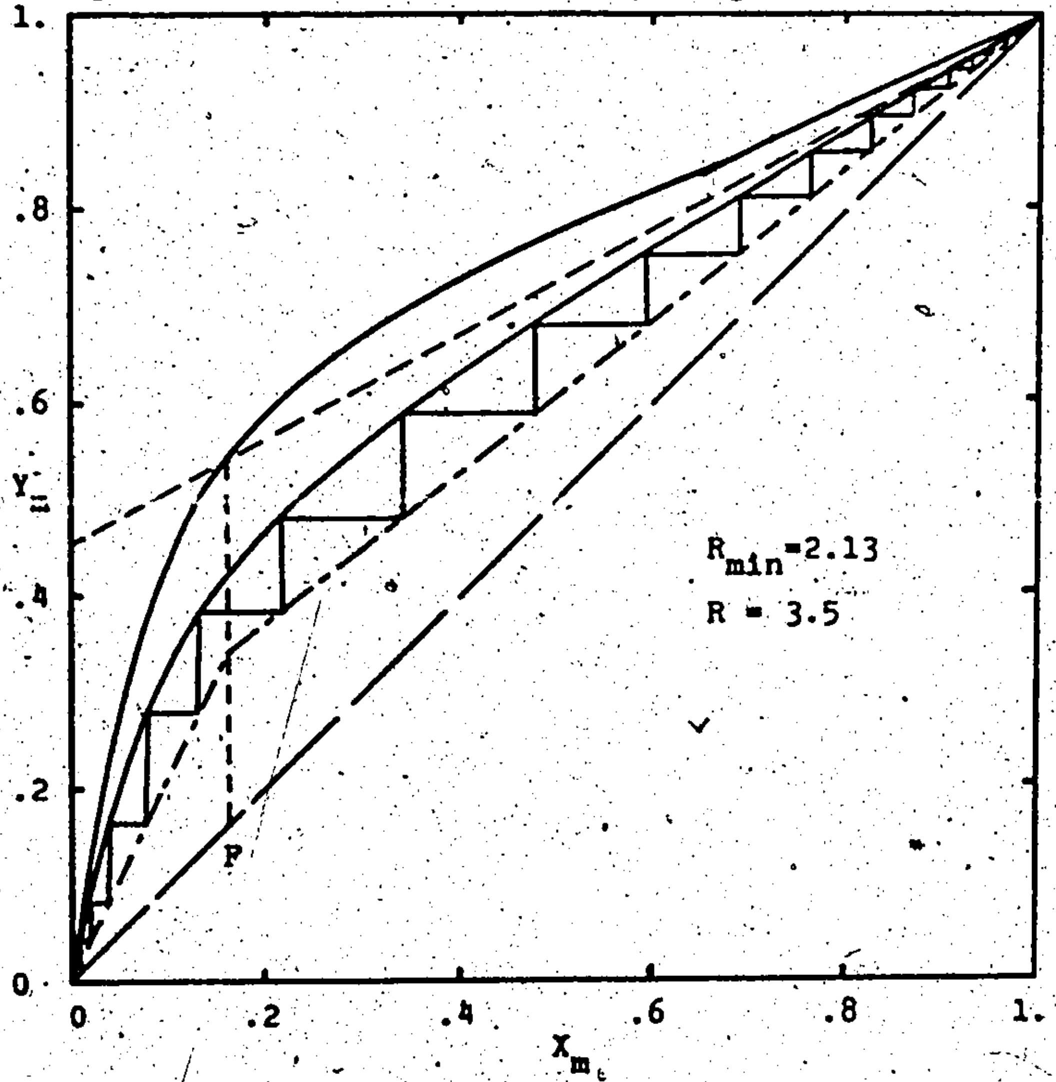
	Theoretical	Design
Total # Stages	10+reboiler	13+reb
Feed Stage	3	3
Solvent Stage	7	8

Table 2.3.C

Results of Solvent Recovery Stage Calculations

	Theoretical
Total # Stages	17+reboiler
Feed Stage	5
Reflux Ratio	$3.5 = 1.7 \times R_{min}$

Figure 2.3.C Solvent Recovery Stage Calculations



3. Process Equipment

The first stage of this project dealt with the development of the extractive distillation column for manual operation.Many refinements to column design were made to improve column operation,and an analytical technique for stream analysis was developed.

Figure 3.A outlines the overall flow diagram for the entire extractive distillation process.This thesis deals with the extractive distillation column only,so details of the solvent recovery distillation column are not included.

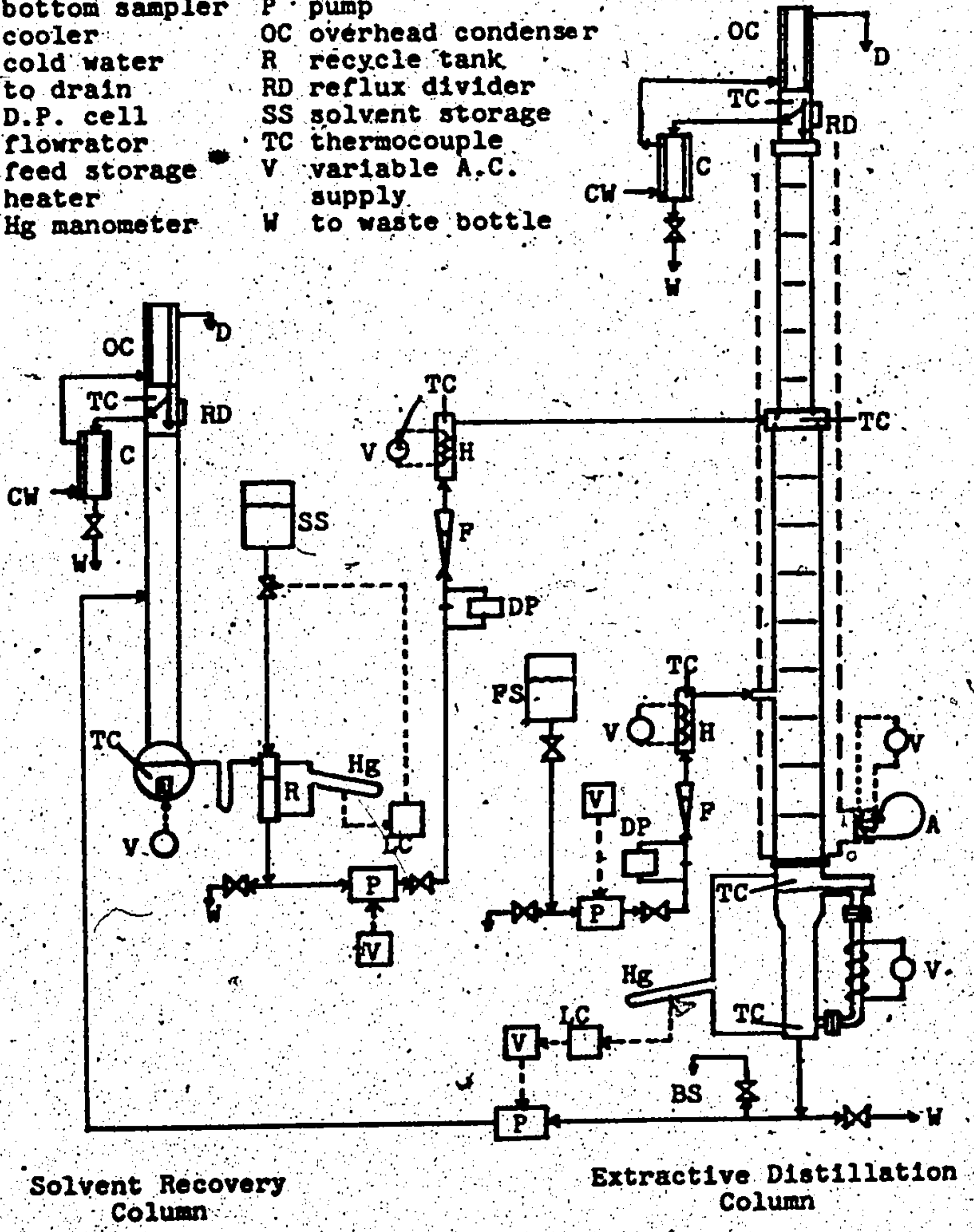
Discussion of the process equipment is presented under the following categories,

1. Reboiler
2. Absorbing and Stripping Sections
3. Solvent Recovery Section
4. Hot Air Jacket
5. Overhead Condenser and Reflux Divider
6. Solvent and Feed Stream Hardware
7. Instrumentation and Stream Analysis

Figure 3.A Flow Diagram - Extractive Distillation Process

Abbreviations

A	air gun	LC	level controller
BS	bottom sampler	P	pump
C	cooler	OC	overhead condenser
CW	cold water	R	recycle tank
D	to drain	RD	reflux divider
DP	D.P. cell	SS	solvent storage
F	flowrator	TC	thermocouple
FS	feed storage	V	variable A.C. supply
H	heater		
Hg	Hg manometer	W	to waste bottle

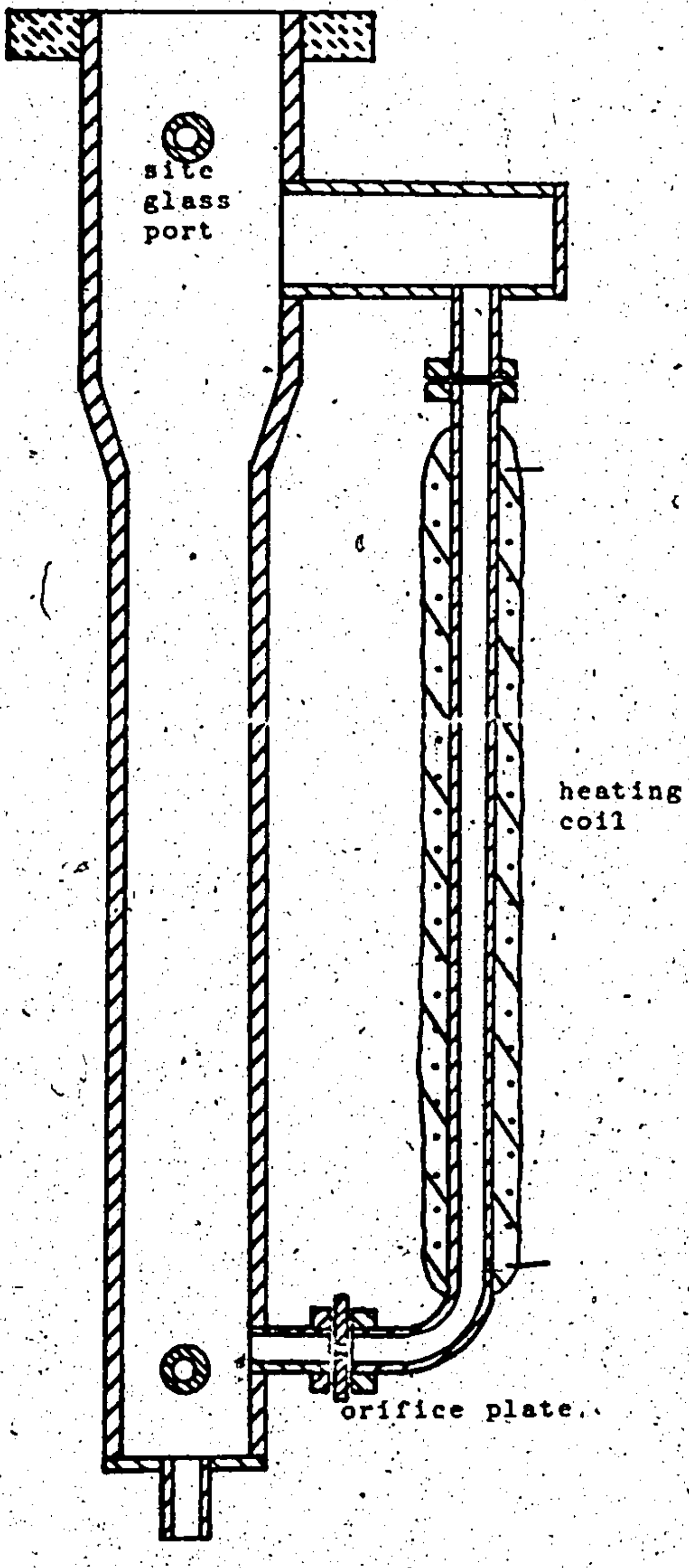


3.1 Reboiler

Figure 3.1.A is a cross-section of the stainless steel reboiler built for the extractive distillation column.The upper section of the reboiler was machined from stainless steel to convert from the 2" I.D. glass column,mounted to the steel support flange,to the 1 1/4 " diameter reservoir. The electrically heated sidearm,which has a range of 0 to 620 watts provided by a Powerstat transformer,is removable to permit replacement of the orifice plate (7/64" orifice) required to prevent surging in the reboiler.The bottoms product is withdrawn through the 1/2 " diameter tube at the bottom,to which is attached the 1/4 " stainless steel tube waste line with brass Swagelok fittings. An inclined mercury manometer measures the liquid level in the reboiler, which may also be observed via a sight glass mounted to the side of the reboiler.Contact points in the manometer activate a Warrick Controls level controller which alternately turns the waste pump on and off when the level reaches the upper and lower limits,respectively.The reboiler is insulated with fibreglass and is supported by a pair of angle-irons mounted to the laboratory framework.

The original reboiler was steam heated because it was felt that this method would be most easily interfaced with the control computer.However,trial runs showed that the

Figure 3.1.A Reboiler



column vapour flow capacity was considerably less than expected. The subsequent reduction in heat requirements would necessitate a steam jacket 2" long, which would be difficult to construct and use. For this reason, the new heater was constructed using resistance wire wound around a 1/2 " stainless steel tube.

3.2 Absorbing and Stripping Sections

The absorbing/stripping column consists of an eight-stage sieve plate-and-downcomer assembly supported in the 3' X 2" I.D. Q.V.F. glass pipe by wall friction. A glass side-arm, mounted 15" from the bottom end, is the feed inlet to the third plate from the bottom. Figure 3.2.A is a cross-sectional diagram of the bottom end of this column, illustrating the various pieces forming one stage. The stainless steel split ring beneath the bottom plate was inserted as a safety precaution for plate assembly support.

The 1/16" X 1 15/16" stainless steel sieve plates (Figure 3.2.B) are strung on a 1/8" X 30" stainless steel rod and separated by 3 1/2" sections of 1/4" stainless steel tube. Teflon collars were machined to fit the plates and provide a tight-as-possible fit against the glass wall. The 1/2" O.D. stainless steel downcomers extend 1/2" above the plates to provide overflow weirs. The slightly angled bottom end of each downcomer almost touches the plate below to use the liquid level on that plate as a seal to prevent vapour flow up the downcomer. The downcomer for the bottom plate was

Figure 3.2.A Cross-Section of Base of Absorbing/Stripping Column

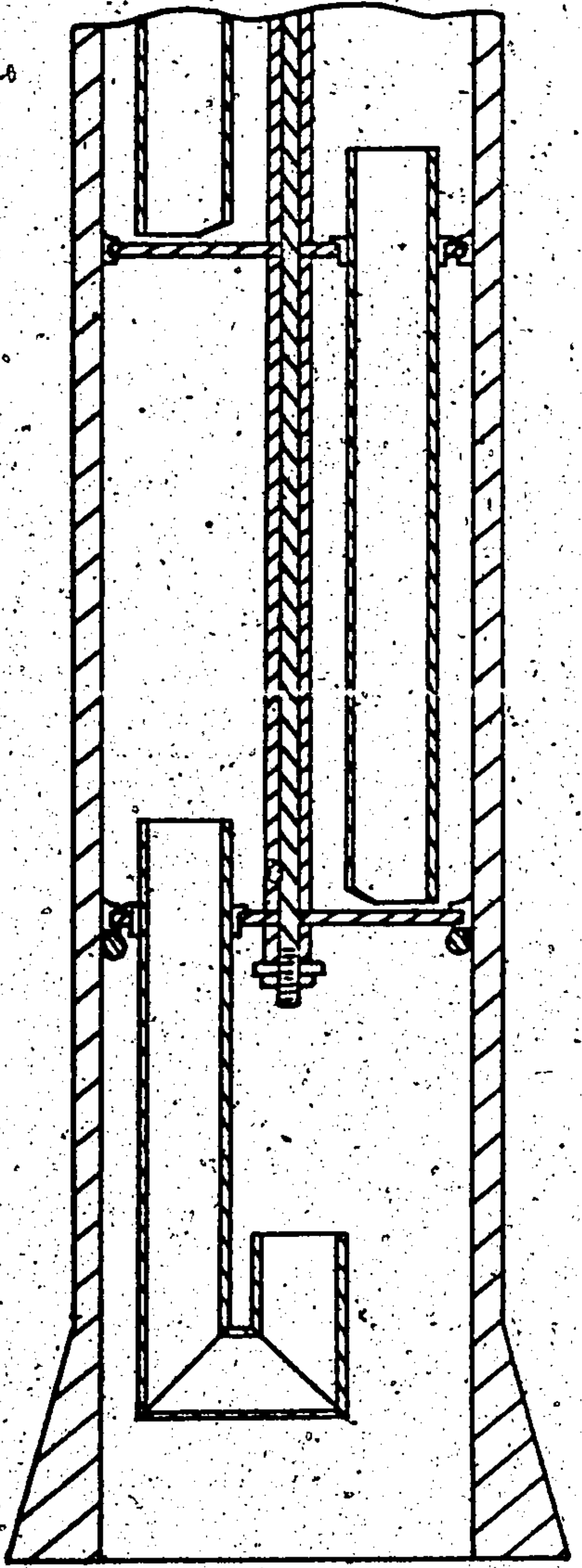


Figure 3.2.B Sieve Plate Pattern

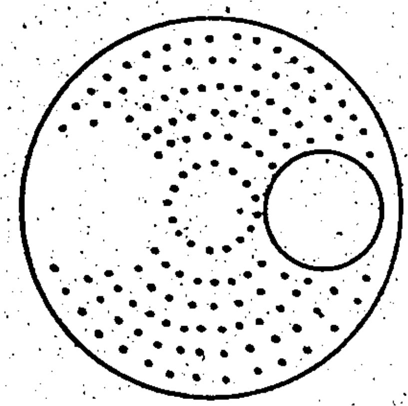
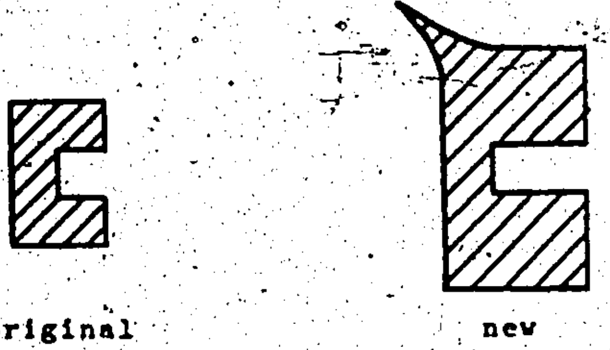


Figure 3.2.C Teflon Plate Collar Designs



constructed with a U-bend to provide its own liquid seal.All downcomers were supported by teflon collars machined to fit a 9/16" diameter hole in the plates.

Several problems in the original plate assembly necessitated its complete replacement or modification.Excessive weepage through the plate,and leakage between the plate and wall,caused low liquid hold-up on the plates.This allowed vapour flow up the downcomers,which compounded the hold-up problem because of reduced plate pressure drop.To reduce the liquid losses,new plates were machined with fewer,smaller, more evenly spaced holes,and the teflon collars were redesigned,as shown in Figure 3.2.C,to achieve a better wall seal.A good wall seal was difficult to achieve because of the variations in glass pipe diameter,especially at the flange locations,and the semi-fluid characteristics of the teflon material.The new collar design incorporated a feathered edge, which could spring back after passing through the pipe neck constriction,and a greater plate overlap,to hold the collar more securely on the plate.The downcomers were modified,as shown in Figure 3.2.D,to reduce the possibility of vapour flow up them at low liquid hold-up.

3.3 Solvent Recovery Section

The original extractive distillation column was built with a five sieve tray,1" diameter solvent recovery section. Because this section severely limited the operating capacity of the column,a new section was built,based on the design of

Figure 3.2.D Modification to Downcomer Design

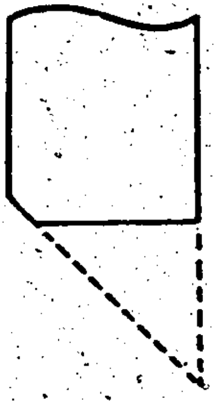
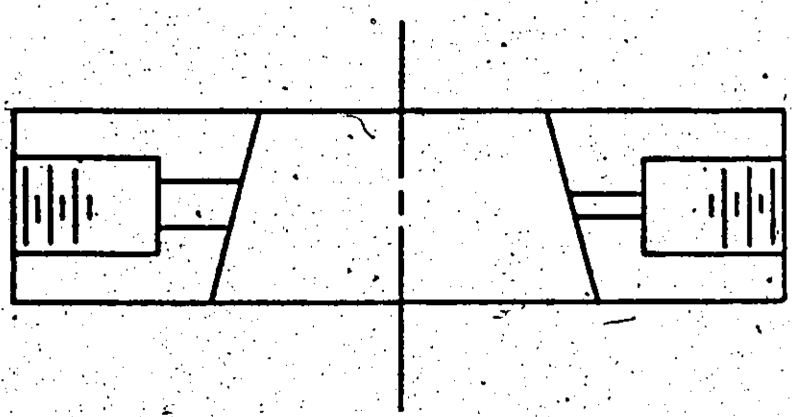


Figure 3.3.A Column Transition Piece



the absorbing/stripping section.The present solvent recovery section consists of a 2' X 1 1/2" I.D. Q.V.F. glass pipe supporting five sieve plates with 3/8" O.D. downcomers.A stainless steel transition piece,as shown in Figure 3.3.A, was machined to connect the glass pipes and provide solvent stream and thermocouple ports.Another similar piece,without ports,was machined to attach the 2" I.D. to 24/40 standard taper glass joint connector to the top of the solvent recovery section.

3.4 Hot Air Jacket

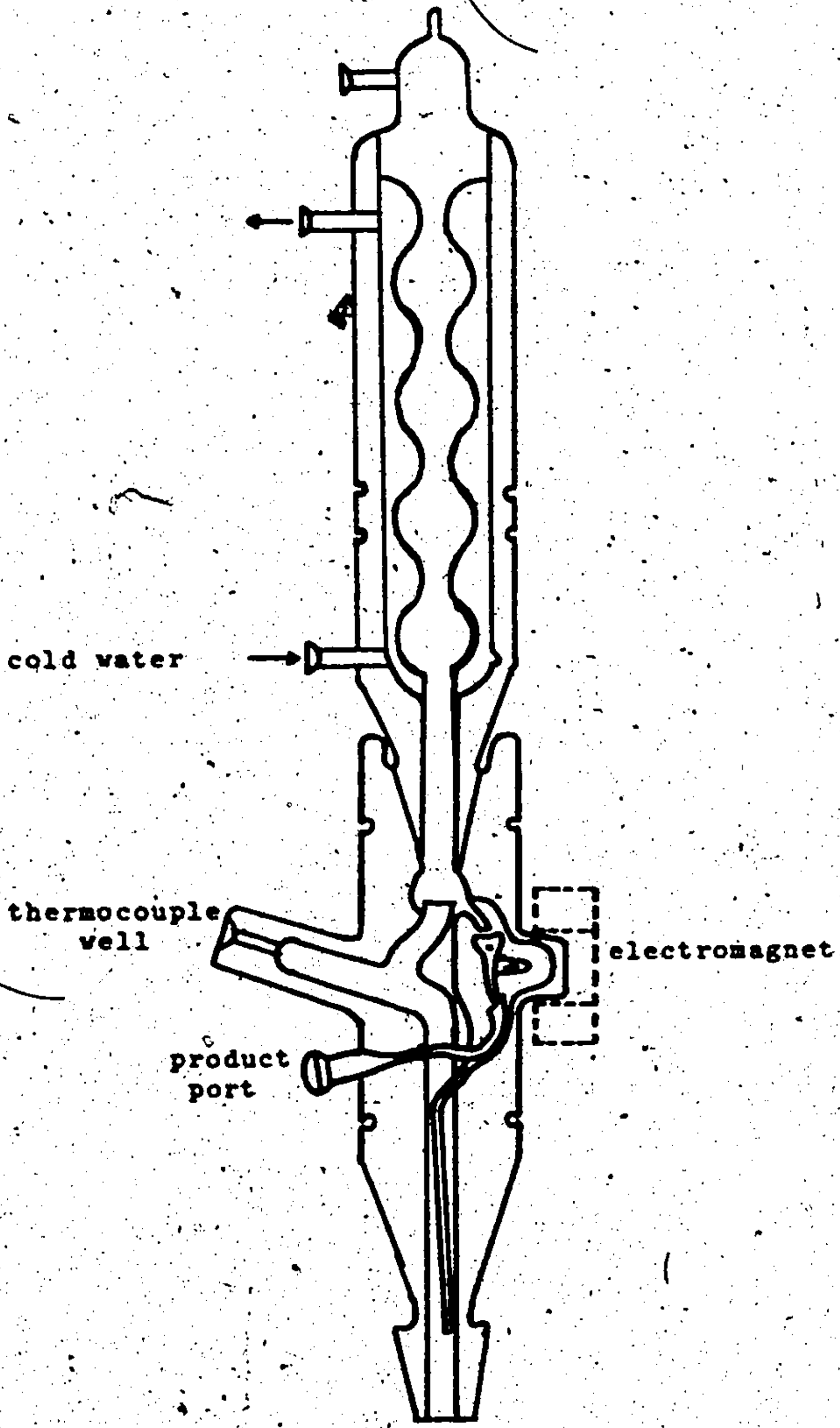
Because this equipment was to be used as an undergraduate teaching project,a hot air jacket was used for column insulation to allow observation of the plate action. The jacket was constructed of two sections of 4 1/2" I.D. Perspex plastic pipe connected by a locating ring constructed of the same material.Hot air from a Master Appliance Corporation heat gun,re-wired to allow variable temperature control, was directed into the jacket by a 4" X 2" I.D. Perspex pipe attached to the side of the jacket base.A Perspex plate sealed the bottom of the jacket and centred it about the column.The air temperature,limited to the softening temperature of the Perspex (80°C),could be adjusted by a Variac power transformer.Under normal operating conditions,the temperature at the top of the jacket was approximately 20°-25°C lower than the base temperature.

3.5 Overhead Condenser and Reflux Divider

The overhead condenser and reflux divider,as shown in Figure 3.5.A,are glass,double-walled,vacuum-insulated devices manufactured by Standard Glassblowing Laboratories.The reflux divider is activated by an electromagnet positioned beside a funnel in which is imbedded a piece of soft iron.The on and off periods of the electromagnet are set by an adjustable electrical timing device manufactured by Eagle Signal Corporation.The condensate from the total condenser drips into the funnel,which directs it back into the column or out the side port.The product stream is cooled in a water jacketed graduated cylinder before it is discharged to a waste bottle. A stopcock at the bottom of the cylinder allows sample collection for composition analysis and flowrate measurement. The overhead vapour temperature is measured with a thermocouple inserted into the vapour stream via the thermocouple well in the reflux divider unit.

A problem with this arrangement of condenser/divider is that the condensate is cooled several degrees below its boiling point when it is returned to the column.This cooled recycle condenses more of the vapour entering the top plate than was assumed in the stage calculations,therefore the vapour and liquid flows in the column are greater than those which were determined from overhead product flow measurements.

Figure 3.5.A Overhead Condenser and Reflux Divider



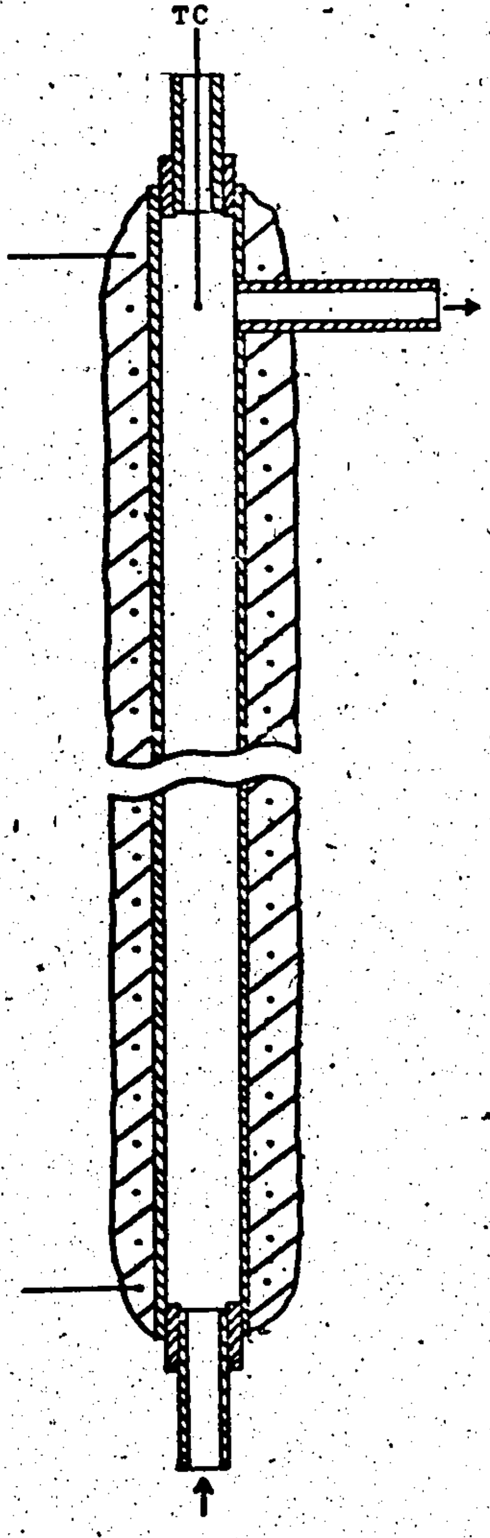
3.6 Solvent and Feed Stream Hardware

The arrangement of the hardware in the solvent and feed streams may be seen in Figure 3.A. Feed from the 10 litre glass storage tank is pumped to the third from bottom plate,while solvent from the solvent reservoir is pumped to the eighth from bottom plate.Both streams use only glass or 1/4" stainless steel tube except for the surgical tubing used by the Chemical Rubber Company Vibrostaltic pumps.A glass orifice in each stream reduces the pump capacity to the desired range and provides a pressure drop for a differential pressure cell flow measurement.Visual flow measurements are made with F&P Co. Precision Bore Flowrators which are mounted with ground glass ball and socket joints to reduce maintenance breakage.

Each stream heater,Figure 3.6.A,is constructed of 1/2" stainless steel tube wound with resistance wire.The thermocouple port allows measurement of the outlet stream temperature for adjustment of the power input (0 to 920 watts) from a Powerstat transformer.The feed line is attached to the column with a ground glass ball and socket joint,while the solvent line is attached to the stainless steel transition piece with brass Swagelok fittings.

The solvent reservoir collects the overflow from the solvent recovery column reboiler for recycle to the extractive

Figure 3.6.A Stream Heater



distillation column. A Warrick Controls level controller, activated by contact points in an inclined mercury manometer measuring the reservoir liquid level, operates the solenoid valve in the make-up solvent line to maintain the reservoir level above a desired minimum level.

Flow adjustment was designed to be made by the C.R.C. Speed Control units providing power to the Vibrostaltic pumps. This adjustment was found to be too delicate at the low flows required, so the pumps are operated at a higher rate than required and flow adjustment is made with a Hoffman clamp on the surgical tubing downstream from the pump.

3.7 Instrumentation and Stream Analysis

Copper-Constantan thermocouples, insulated with RTV silicone-rubber, provide temperature measurements at the ten locations shown in Figure 3.A. The leads from the thermocouples terminate in a patch panel, from which the desired readings may be directed to a 12-point Honeywell Electronik 15 recorder for manual operation, or to Acromag transducers for transmission to the control computer.

The solvent and feed flows are measured visually with flowrators and electrically with Dynascience Corporation differential pressure cells. The millivolt signal generated by the D.P. cell is converted to a 0 to 10 volt D.C. signal by a Dynascience Corporation Carrier-Demodulator, for transmission to the control computer.

Samples of the overhead and bottom streams are analysed with a Varian 90-P3 gas chromatograph.Helium carries the vaporized sample through a 6' X 1/4" O.D. stainless steel column packed with 80 mesh Porapak Q material.The chromatograph signal is sent to a Hewlett-Packard 3370B Integrator which calculates the area beneath the peaks,printing out the peak time and area for each peak.A calibration curve relating peak area to moles of compound was developed,using several calibration-solutions.Stream composition is determined by converting the component peak area to moles and then calculating mole fractions.The calibration curve is independent of sample size,for samples from 0.4 to 0.7 μ-litres.Details of chromatograph and integrator settings and the calibration curve are found in Appendix A.Analysis of a sample takes approximately 2.5 to 3 minutes,from time of injection to print out of the last peak area.This limits the sampling frequency to a maximum of three per hour,if two streams are sampled and two analysis done on each sample.

4. Application of the Control Computer

Since future work on this project will deal with direct digital control studies as well as process dynamics studies, the second phase of this project dealt with incorporation of a preliminary computer monitor/control package. After interfacing the process equipment with the control computer, the monitor/control package was used as an aid in operating the extractive distillation column.

4.1 The Control Computer

The control computer used for this project was a 16 K Supernova digital computer manufactured by Data General Corporation. Analog signals (0 to 10 V D.C.) from the process, such as thermocouple readings, are handled by 16 input channels multiplexed into a single 10-bit analog-to-digital converter (ADC). Analog signals from the computer to the process are handled by six 10-bit digital-to-analog converters (DAC). Also interfaced with the computer are sixteen relay outputs which are used as on/off switches, and 16 contact sense inputs which can be used as process-to-computer switches. Two teletypes and a line printer are used for printed input and/or output, while two high speed devices, a reader and a punch, handle paper tape input/output. Extra memory storage is provided with a 256 K fixed head disk and a 160 K cassette tape recorder.[31]

4.2 Computer Interfacing

Signals between the computer, located on the third floor of the Chemical Engineering Department, and the process equipment, located on the first floor, are transmitted a distance of 120' by a pair of Belden cables. These cables, types 8775 and 8778, contain 6 and 11, respectively, individually twisted, shielded pairs of wires, and are carried in a 6" square electrical conduit used solely for computer application projects. With these cables, there are two interfaces, one at the process site and one at the computer site.

The interface at the computer site consists of a patch panel network which currently handles inputs from four different locations in the Chemical Engineering Department. This panel was installed to allow more flexible and efficient use of the computer by simplifying the cable/computer interface procedure. This interfacing is accomplished by connecting the various cable terminals to the appropriate computer hardware terminals. Voltage signals from the process are input directly to the ADC terminals, while current signals are dropped across appropriate resistors (200 Ω for thermocouple transducer signals) and the resulting voltage drops are input to the ADC terminals. Details of the patch panel wiring are given in Appendix B, while proper connections of terminals for this project are given in Appendix C.

The interface at the process site consists of a mobile metal box containing eight Acromag transducers and the wiring for the interface panel to which the transmission cables, relay box cable, thermocouple leads and other process equipment leads are connected. The eight transducers, six for Copper-Constantan thermocouples and two for Chromel-Alumel thermocouples, are hard-wired to eight pairs of wires in the 11-pair cable. The other three pairs in this cable, plus the six in the other cable may be used for other signals transmitted to or from the computer.

A relay box has been constructed which contains eight 6 Volt, single pole, double throw relay switches wired to the mobile box with a Belden cable. These relays are activated by the computer relays and are used as on/off switches in the process equipment. By using a set of relays at the process site, high voltages are confined to the process site and kept safely away from the computer interface panel. For this project, the relays were used to operate five signal lights, two heaters and a reflux divider.

4.3 Computer Software Package

This preliminary software package was developed to aid in the manual operation of the extractive distillation column. To accomplish this and yet avoid great expenditures for control equipment, the necessary requirements of this package were determined to be,

1. operate on a real time basis to allow on-line initiation and/or termination of tasks and on-line parameter changes
2. measure and record process temperatures and input stream flowrates
3. control input stream temperatures
4. signal for manual correction of stream flowrates
5. operate the reflux divider at any desired ratio

To partially satisfy requirement 1.,the software package was programmed to operate within the real time operating system (RTOS) supplied by the computer manufacturer. With this system,input/output hardware devices are serviced according to a device priority scheme designed to minimize the average time devices must wait for servicing.User-written programs operating under this system are also given priority ratings by the user.The RTOS package then schedules all the tasks for execution according to these priorities under a task priority scheme.Thus,the important programs will be executed when required,temporarily interrupting lower priority programs if necessary.

To allow on-line initiation and/or termination of tasks,an executive program,XTRAC,was written to initiate four service routines upon start-up and then to respond to operator requests for service.This program accepts the operator input and,if the correct code word was entered,initiates or terminates the desired program.

The service routines initiated by XTRAC were written to obtain process measurements (ADRDR),operate the computer relays (RELAY),function as a clock (CLOCK),and indicate to the operator at the process site that the computer is functioning (PRUN).

By having one program,ADRDR,read all the process measurements at one time,the number of device service requests is reduced,thus reducing computer software overhead time.The program digitally filters the input data and stores it in a table for other programs to use.

For similar reasons,one program,RELAY,is used to operate all the computer relays.Control and monitor programs which use relays simply set a code word within RELAY instead of obtaining the current relay pattern,changing the desired relay bit to the appropriate contents and then installing the new relay pattern.

The service routine PRUN uses a relay to turn a light at the process site on and off at 5 second intervals to indicate to the operator that the computer is still functioning.If the computer stops,the light will remain on or off and appropriate action may be taken by the operator immediately.

CLOCK is a simple routine to count seconds,minutes and hours.This provides a time measurement from program initiation,enabling comparison of real-time events with computer-time process measurements.

The software package contains several program parameters which determine program periods,digital to engineering units conversion constants and control action calculations, etc.To be able to change these parameters without stopping the computer,a parameter change routine,EXPAR,was developed. With this program,the current value of a variety of parameters may be checked and changed if necessary.

Requirement 2 is partially satisfied by the program ADRDR,but the recording of process measurements is performed by a data logging routine,DALOG. At regular intervals,this routine obtains the current process measurements,converts them to engineering units,if so desired.and outputs the data to the second teletype.Each data set is numbered,and the output is in the format of 11" pages for convenience.To correlate the data sets with the time of day,each page is headed by the computer time at which the page was started.

Another routine,SSAMP,also outputs data to the second teletype.This routine checks the contact sense inputs every second and,if a change has occurred,outputs the current computer time and contact sense pattern.The contact sense inputs are activated by switches at the process site,which are normally used to indicate when an overhead or bottom stream sample is being collected.

Control of input stream temperatures is currently limited to turning the stream heaters on and off with relay switches. The two stream temperature control routines, one for the solvent stream, SOLTC, and one for the feed stream, FEETC, have been written to implement this control in two different ways; simple on/off control and heater-on period control. The routines function to maintain the heater exit stream temperature at a setpoint determined by the user. In the case of the solvent temperature controller, if the setpoint is zero, the routine uses the current column temperature at the solvent inlet stage as the setpoint.

The simple control method is on/off control whereby the stream temperature is compared with the setpoint temperature at regular intervals and the heater turned on or off, depending on the sign of the error.

The other control method is more complex in that it is based on heat requirements. At time t, the actual heat input to the stream may be calculated from the current flowrate, F_t, and outlet temperature, T_t, as

$$Q_t = F_t(T_t - T_{in})C_p \tag{4.3.a}$$

where C_p is the heat capacity of the liquid and T_{in} is the assumed inlet temperature to the heat exchanger.

For this flowrate, F_t, the heat setpoint may be determined, based on the outlet temperature setpoint, T_{set}, as

$$Q_{set} = F_t(T_{set} - T_{in})C_p \tag{4.3.b}$$

Therefore, the error in heat input is

$$\begin{aligned} Q_e &= Q_{set} - Q_t \\ &= F_t(T_{set} - T_t)C_p \end{aligned} \tag{4.3.c}$$

Control action computed on this heat error would be added to the mean heat requirement at time t, assumed to be

$$Q_m = F_t(T_{set}-T_{in})C_p \qquad 4.3.d$$

to yield the predicted heat requirement for the next period as

$$Q_{req} = Q_m + f(Q_e) \qquad 4.3.e$$

where $f(Q_e)$ denotes the control action computed on the heat error. Substitution of equations 4.3.c and 4.3.d into equation 4.3.e yields

$$Q_{req} = F_t[(T_{set}-T_{in}) + f(T_{set}-T_t)]C_p \qquad 4.3.f$$

This simplifies the control algorithm since control action is now computed on the outlet temperature error rather than the heat error.

To implement this control action, the rate of heat input must be known. If the voltage to the heater is such that, at a maximum flowrate, F_{max}, the outlet temperature is at a desired maximum temperature, T_{max}, when the heater is on continuously, the maximum heat input is

$$Q_{max} = F_{max}(T_{max}-T_{in})C_p \qquad 4.3.g$$

Thus, if control action is computed every P seconds, the length of time the heater must be on to deliver the required heat is

$$P_{on,t} = Q_{req}/Q_{max} X\ P$$

$$= \frac{F_t[(T_{set}-T_{in}) + f(T_{set}-T_t)]}{F_{max}(T_{max}-T_{in})} P \qquad 4.3.h$$

With this ratio, the heat capacity of the liquid is not required and the control algorithm may be easily applied to any stream heater.The control routines use a general proportional-integral-derivative control subroutine to calculate the control portion of equation 4.3.h and then turn the stream heater on for the calculated time, $P_{on,t}$.The period of the controller may be changed without affecting the calculation of the required time on.

Flow control devices were not purchased for this phase of the project because the operating range of flowrates were not known.Instead,a flow monitoring program was written to compare measured flowrates with flow setpoints.If the magnitude of the error is greater than a specified error limit,a light at the process sight is turned on to indicate the error to the operator,requesting manual flow adjustment. The software package contains a monitor program for each input stream (SOLFM and FEEFM),with one light activated by each program.

In future studies,the reflux ratio will be one of the controlled variables in the direct digital control of the column.For this reason,the routine to operate the reflux divider,RFLUX,was written to calculate the length of time the divider must be activated each period to simulate a desired reflux ratio,and then activate the divider,using a relay switch,for this length of time.The calculations are repeated each period to allow changes in the reflux ratio or reflux period at any time.

Details of each program are given in Appendix D.

5. Experimental Results and Discussion

The first series of experimental runs was made under manual operating conditions to find and correct, if possible, design problems. The next series of runs was made with the computer to obtain steady-state data for the column and to evaluate the computer monitor/control software package. The results of the first series were presented in Chapter 3, which describes the process equipment and the major modifications made during this trial-and-error period. This chapter presents an analysis of the steady-state data obtained during the second series of experimental runs, an evaluation of the computer software package, and a discussion on current design problems and recommendations. Details of process start-up and operation are given in Appendix E.

5.1 Analysis of Steady-State Data

Through previous experimental runs, it was found that the 1 1/2" diameter solvent recovery section of the extractive distillation column limited the vapour flowrate to a maximum of approximately 16 moles/hour. The design flowrates were scaled to achieve this vapour rate, which resulted in a feed rate of 8.0 moles/hour and a solvent rate of 16.4 moles/hour. The feed was 60 mole% acetone, 40 mole% methanol. The desired overhead product rate, with the reflux divider operating to simulate a reflux ratio of 2, was 5.14 moles/hour or approximately 6 ml/minute.

Three sets of samples were collected and analyzed, as listed in Table 5.1.A, during a six hour run of the equipment. The feed and solvent rates were checked by an overall mole balance, based on stream compositions and overhead rate. The results, Table 5.1.B, indicate that the feed rate was fairly constant, but that the solvent rate varied considerably. Each set of data was used to determine the theoretical number of stages in the column, based on the calculated flowrates. The results from all three sets of data agreed that the theoretical column consisted of eight stages (including the reboiler) with feed and solvent charged to the second and fifth stages, respectively, above the reboiler. Thus, the overall column efficiency, as well as each section's overall efficiency, is approximately 60% under these operating conditions. This efficiency could be increased if the column was re-designed to eliminate leakage at each stage and the solvent recovery section replaced with a 2" diameter column to increase the total column capacity.

5.2 Computer Monitor/Control Package Evaluation

Attempts to manually operate the extractive distillation column under steady-state conditions required constant attention to input stream flowrates and temperatures. Implementation of the computer monitor/control package relieved the operator of these tedious and time-consuming tasks, maintained the column at steadier operating conditions,

Table 5.1.A

Experimental Results

Sample	Overhead Rate	Overhead Composition mole %			Bottom Composition mole %		
#	ml/min	A	M	W	A	M	W
1	5.9	91.7	4.15	4.15	1.6	16.2	82.2
2	5.6	91.6	4.3	4.1	1.7	16.2	82.1
3	5.7	92.1	4.1	3.8	1.9	17.6	80.5

A=Acetone   M=Methanol   W=Water

Table 5.1.B

Molar Flowrates Based On Mole Balance

Sample #	Feed moles/hr	Solvent moles/hr	Overhead moles/hr	Bottom moles/hr
1	8.19	15.78	5.03	18.94
2	7.89	15.14	4.83	18.20
3	7.98	13.88	4.85	17.01

and allowed more frequent stream sampling and analysis.The data logging routine produced a record of temperatures,flows, and sample intervals which was much more useful than the recorder chart output because the data was presented in neat tables in engineering units,and a paper tape copy of the data can be produced which can then be used to produce a deck of computer cards for more detailed column analysis or graphical presentation,using the university computer.

Time did not permit the implementation of the period-on temperature controller routines,but the on/off control routines worked reasonably well.Figure 5.2.A represents the flowrates and temperatures of the feed and solvent streams, logged at one minute intervals over a forty minute period.The temperature controller routines checked stream temperatures and took control action every two seconds.Table 5.2.A lists the setpoints for each data set,plus the average and standard deviation of each data set.

The temperature control of each stream is reasonably good,but it is evident from a comparison of the two streams that temperature control is very dependent on flow control.To achieve better flow control,the proper hardware must be purchased to enable the direct digital control of the flowrates.It should be noted that this evaluation of the temperature control routines is only approximate,because the data was logged at one minute intervals while control action was taken at two second intervals.More frequent data collection

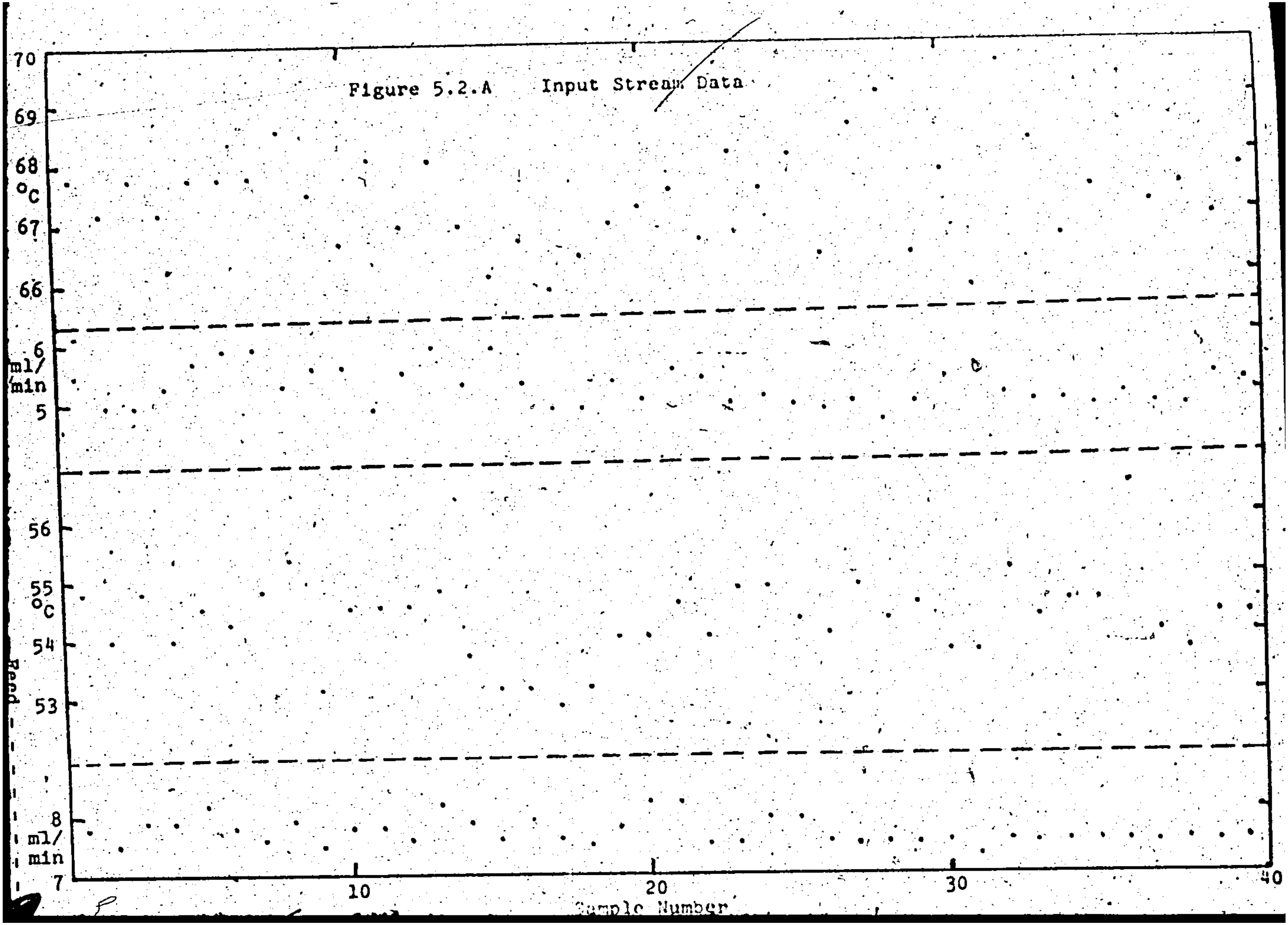


Figure 5.2.A Input Stream Data

Table 5.2.A

Input Stream Data Analysis

Data Set	Set Point	Average	Standard Deviation
Solvent F	5.0	5.2	.35
Solvent T	67.0	67.4	.87
Feed F	7.9	7.7	.24
Feed T	54.0	54.3	.68

F=flow,ml/min. T=temperature,°C.

is required to fully analyze the controllers.

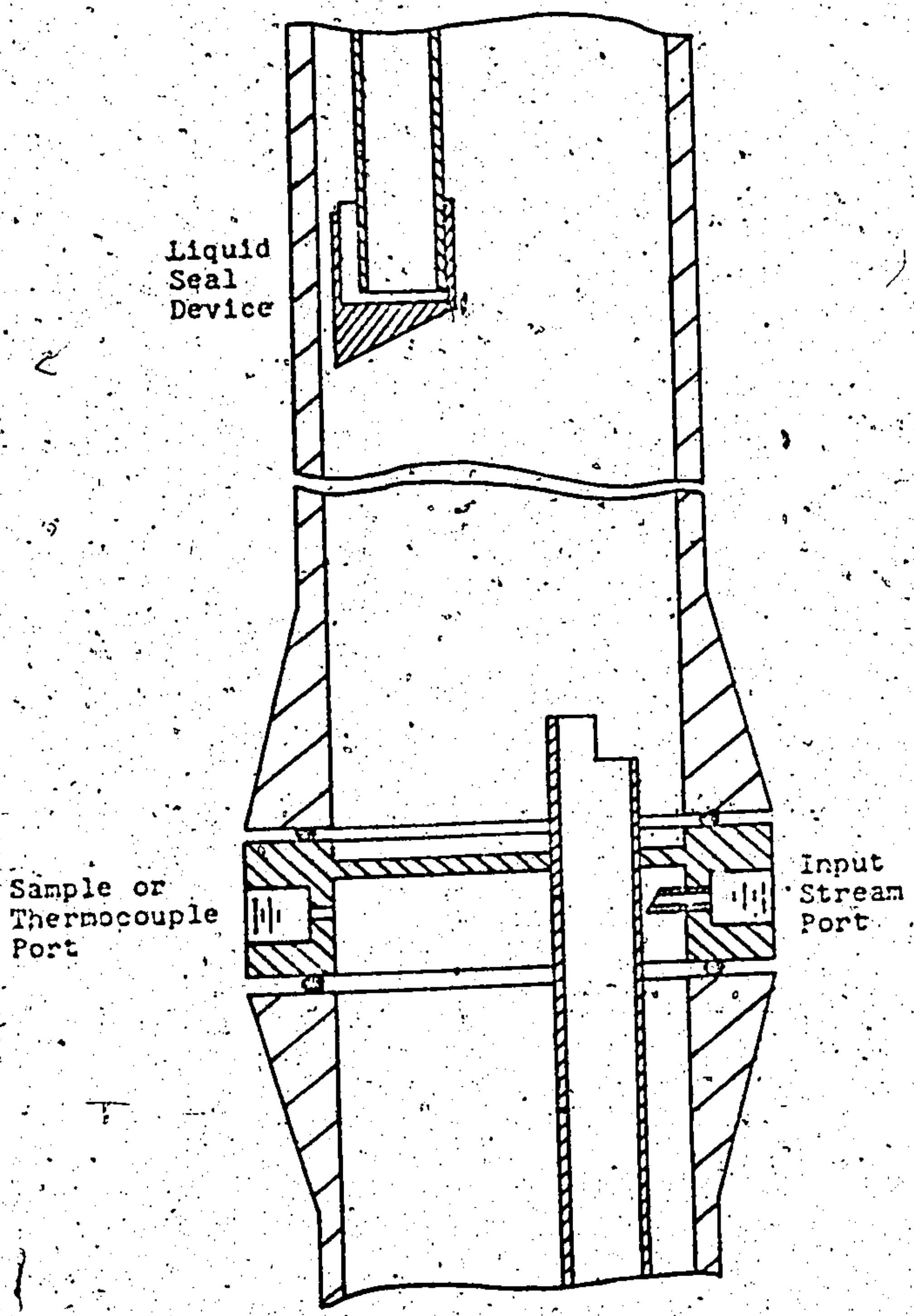
5.3 Current Design Problems and Recommendations

The extractive distillation column is operative with the current design, although considerable maintenance is required and several problems still exist. By correcting these problems, column operation and efficiency would be improved.

The column capacity is currently limited to the capacity of the solvent recovery section. The low vapour rate required for this section is not high enough to prevent excessive weeping and leakage and poor frothing in the larger absorbing/stripping section. A test on this 2" diameter section showed that it could handle twice the flowrates of the solvent recovery section with much better vapour/liquid contact and less weeping. Therefore, it is recommended that the solvent recovery section be replaced with a 2" diameter section.

Because of the semi-fluid characteristics of teflon, the plate collars deform slightly with time and plate leakage increases. Instead of having to dismantle the column regularly to replace the collars, it is recommended that the entire column be replaced with a new column constructed of sieve plates sandwiched between 6" sections of 2" I.D. [illegible].V.F. glass pipe, as shown in Figure 5.3.A. Besides eliminating plate leakage, this design allows more detailed column analysis by locating thermocouple ports and stage sampling ports at each stage. By incorporating charge ports at several stages, the locations of feed and solvent streams can be changed, allowing

Figure 5.3.A Suggested Column Stage Design



greater flexability in column operation.These charge ports were designed so that the side-stream liquid would run down the side of the downcomer and drip onto the stage below with the overflow stream.

In the current stage design,the downcomer is supported by a teflon collar in the plate,and the liquid seal is provided by the liquid hold-up on the plate below.To eliminate the leakage around the downcomer,the new downcomer design has the downcomer soldered to the plate.To remove the inter-dependency of stages for proper vapour flow,the new downcomer is designed with a U-bend to provide the liquid seal.The overflow weir was re-designed to lengthen the liquid flow path across the plate,and the downcomer was designed with a drip-point close to the column wall to increase the plate free-area.Figure 5.3.B illustrates the new plate design and direction of liquid flow.The modifications to increase the liquid path length will increase the stage efficiency by increasing the vapour/liquid contacting area.The overflow weir height was also increased slightly to improve this contacting.

True steady-state operation of the extractive distillation column cannot be achieved with the current practice of on/off control of the bottom stream pump to control the reboiler liquid level.The fluctuations in liquid level cause fluctuations in boil-up rate and,to a lesser degree,reboiler pressure,because of the fluctuations in effective heating

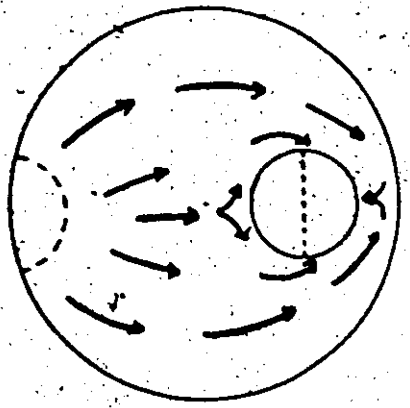


Figure 5.3.B Suggested Plate Design
and
Liquid Flow Pattern

area within the side-arm heater. Other problems will also arise when the solvent recovery column is operated because of the periodic nature of the feed to this column. The bottom stream pump should be operating continuously and the flow adjusted to maintain the proper reboiler level. The control computer could easily adjust the flowrate to the correct amount, based on a reboiler-level measurement obtained with a differential pressure cell.

A problem with the Vibrostaltic pumps exists because the surgical tubing required by the pumps deteriorates with time and use. A gummy residue accumulates in the orifices and flowrators, which then give false flow measurements or stop the flow completely. It would be best to replace these pumps, but if this is not feasible, another type of tubing and/or stream filter would have to be used to prevent the accumulation of residue in stream hardware.

A minor problem exists in the operation of the condenser/reflux divider assembly in that sub-cooled condensate is returned to the top stage of the column. This causes a slight change in the actual vapour rate and liquid rate through the column, but it is doubtful that this has a significant effect compared to the simulation of a proper reflux ratio by a reflux divider. It is estimated that the change in molar vapour flowrate passing through this stage due to sub-cooled condensate is approximately 2%.

6. Summary and Conclusions

A fourteen stage extractive distillation column was re-designed,re-built and evaluated.Under the operating conditions studied,the overall column efficiency was found to be approximately 60%.Recommendations for design modifications were presented and may be summarized as replacing the absorbing,stripping and solvent recovery sections of the column and improving the pumping arrangements of the system.

A stream analysis technique was developed for the ternary system studied.The technique worked very well for manual operation,mainly because sample size was not critical. For complete computer control of the process,a system for automatic stream sampling and analysis must be developed.

A control computer was interfaced to the process and a computer monitor/control software package was developed.The computer was found to be very useful as an aid to the manual operation of the extractive distillation column,but further improvements are needed.These improvements include purchase of proper control instruments to enable continuous computer control of solvent and feed stream flowrates and temperatures, and reboiler liquid level.The executive program and parameter change routine developed are both very flexible in format,so that as more programs are written for the next stages of this project,they may be easily added to the existing software package.

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Appendix A

Stream Sample Analysis

Samples from the process were analyzed using a Varian 90-P3 gas chromatograph and a Hewlett-Packard 3370B integrator. Details of the Chromatograph and integrator are given on the following page. The chromatograph was calibrated by injecting 1 ul samples of known composition and plotting the number of moles of each component in the sample versus the peak area for that component. Figure A1 is the calibration curve developed with this method.

The thermocouples and differential pressure,(D-P), cells were calibrated using the computer data logging routing. The conversion constants were,A=0,B=1,C=0,so that the values logged were analog-to-digital converter units. The thermocouple measurements were recorded at three temperatures,and the D-P cell measurements were recorded at a variety of flows,to evaluate the correct conversion constants,based on a least-squares fit. Table A1 lists the conversion constants,while Figure A2 is a plot of recorded values and conversion equation calculations versus A/D units.

Gas Chromatograph

- Varian Model 90-P3
- column - 6' X 1/4" O.D. stainless steel tube
  - Porapak Q, 80 mesh, packing
- carrier gas - Helium @ 65 psig
  - carrier flowrate - 60 ml/min
  - reference flowrate - 20 ml/min
- dial settings - column power - 55
  - inject power - 45
  - detector power - 40
  - attenuator - 1
- operating temperatures - column - 175°C
  - inject port - 200°C
  - detector - 211°C

Hewlett-Packard 3370B Integrator

- settings - noise suppression - maximum
  - recorder presentation - 100 mV=full scale
  - slope sensitivity - up=0.03
    - down=0.03
  - BL reset delay = 0.5 minutes
  - area threshold = 100.
  - shoulder control - front - on
    - rear = 100 mV

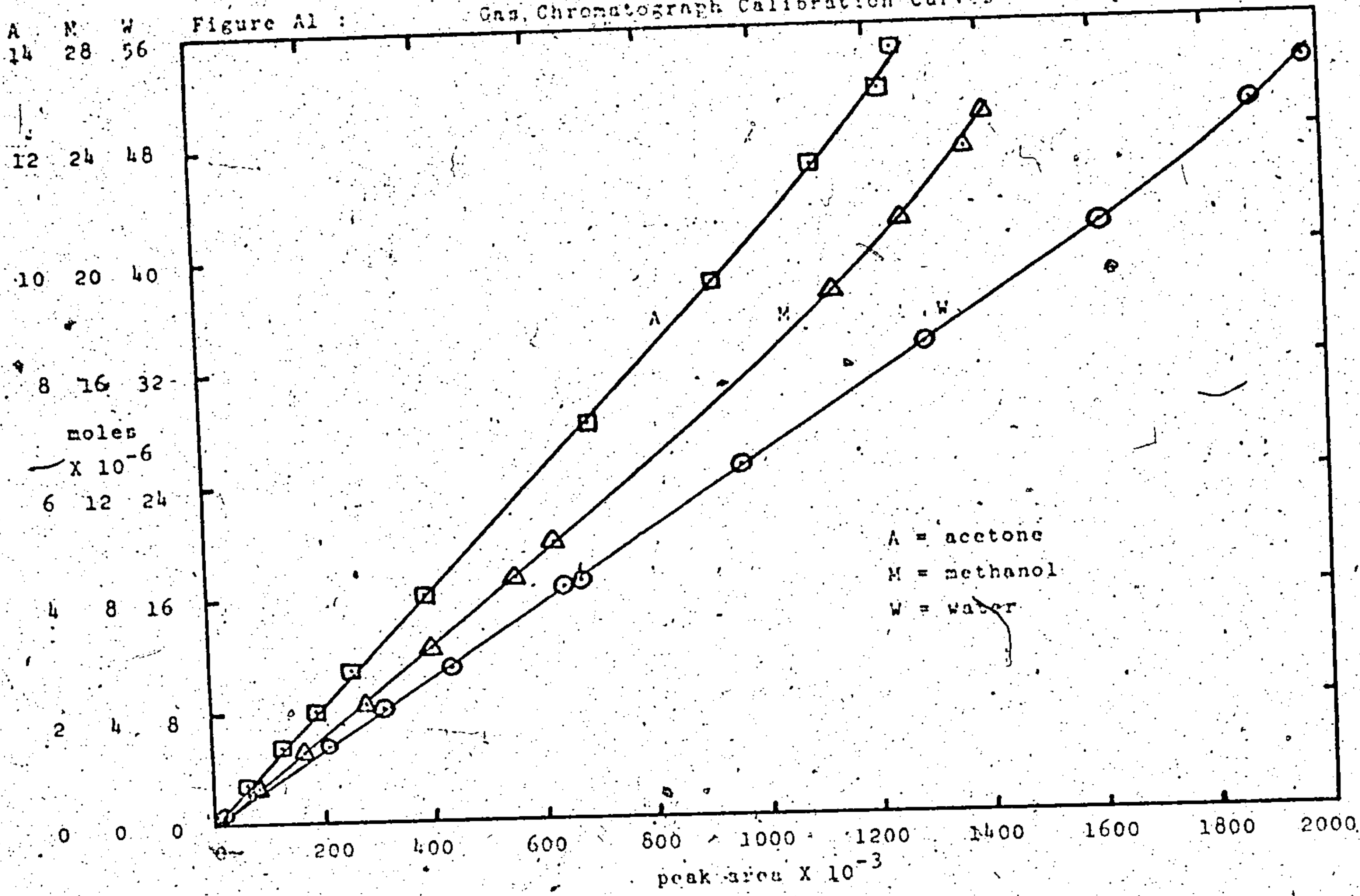


Figure A1 : Gas Chromatograph Calibration Curves

Table AI

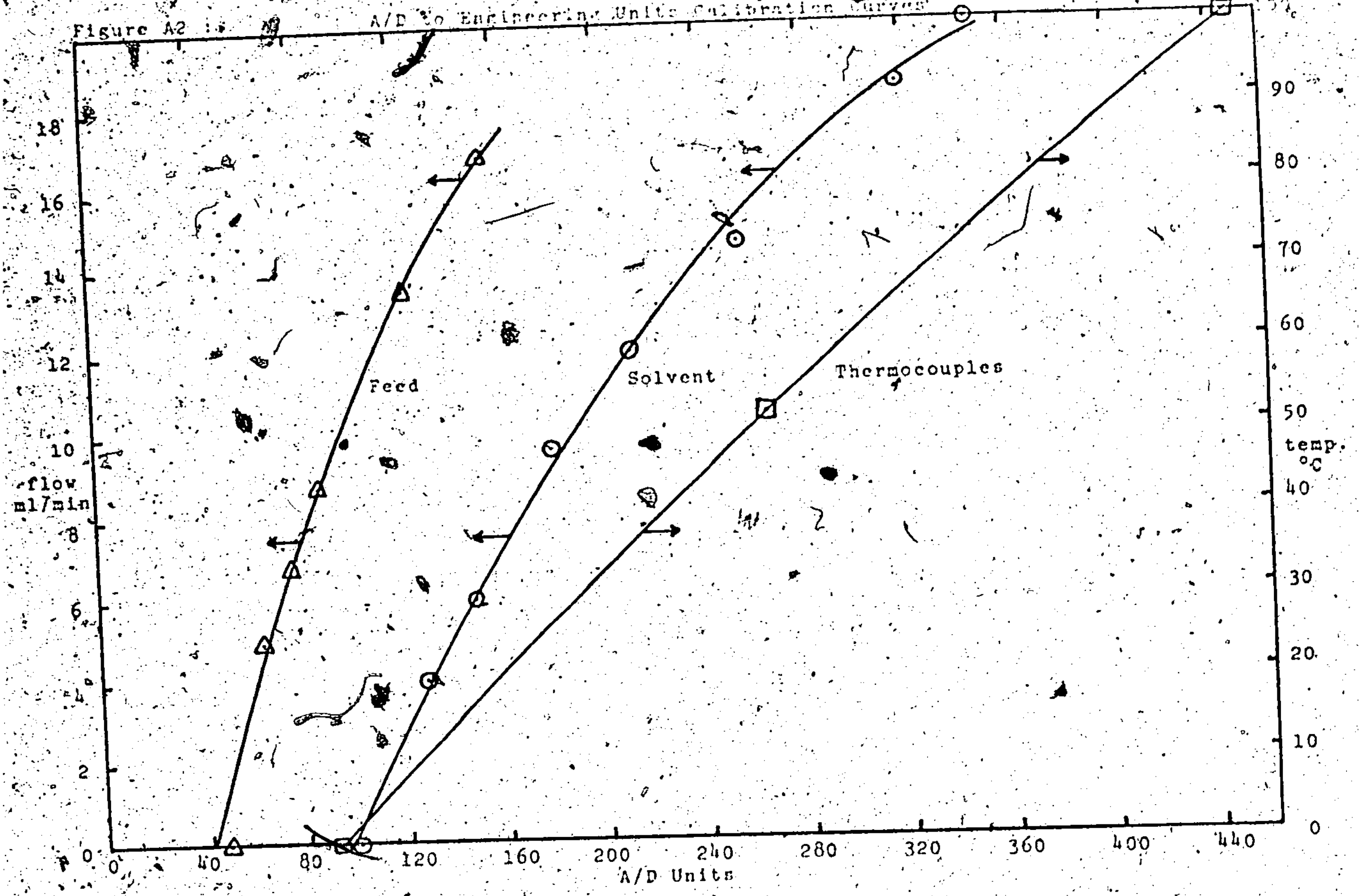
Conversion Constants for the Equation

$$Y = A + BX + CX^2$$

Y	X	A	B	C
Temperature °C X 100	$A/D - A/D_{ref}$	0.	31.93	-0.0108
Solvent ml/min X 10	A/D	-140.	1.63	-0.0019
Feed ml/min X 10	A/D	-99.	2.65	-0.0060

A/D_{ref} = current reading from reference thermocouple

Figure A2 A/D to Engineering Units Calibration Curves



Appendix B Computer Patch Panel Wiring

The patch panel at the computer site consists of a panel in the form of a 20 X 22 matrix, with rows lettered A to T and columns numbered 1 to 22.

The interface panel is connected to the computer hardware via four cables with multi-pin connectors; one each for the relay outputs, contact sense inputs, D/A converter channels and A/D converter channels. The computer terminals on the face of the patch panel form a 12 X 16 matrix, (rows I to T, columns 1 to 16), divided into six sections. Rows I, J, K are the relay output terminals, with row I the normally closed contact, row J the common terminal and row K the normally open terminal. Rows L and M are the contact sense input terminals, row M being the common terminal. Row N is the higher voltage side of the D/A terminals while row O is the lower voltage side. Row P is a row of terminals connected to computer ground. Row Q is the high voltage side of the A/D terminals and row R is the low voltage side. Rows S and T are wired for a future 16 A/D channels in the same manner as rows Q and R.

On the back side of the computer site interface panel, (Figure B1), are the connectors for the computer hardware cables and cables from various locations in the Chemical Engineering Laboratories. Connectors X1 to X5 are currently used as inputs from process sites in the laboratories, X9 is a connector for an external relay box,and X6 to X8 and X10,are not used yet. X1,X3,X5 and X9 are 24-pin Amphenol connectors while X2 and X4 are 14-pin Amphenol connectors.Pins 1 to 12 of X1 are connected to terminals A1 to A12 and pins 13 to 23 are conconnected to terminals B1 to B12.Similarly,X3 is connected to rows C and D and X5 to rows E and F.Pins 1 to 6 of X2 are connected to terminals A13 to A18 and pins 7 to 13 are connected to terminals B12 to B18.Connector X4 is similarly connected to rows C and D.These connectors are normally used for 11-pair and 6-pair Belden cables,with the shields connected to pin 12 or 7 respectively.Thus,the terminals in column 12, rows A to E,are cable shield connections,to enable grounding at the computer site or at the process site.

Only 16 of the pins in X9 are being used currently, and are connected to the terminals of the square matrix formed by rows Q to T and columns 19 to 22.Pins 5 to 8,17 to 20,9 to 12 and 21 to 24 are connected to terminals Q to T in columns 19,20,21 and 22 respectively.

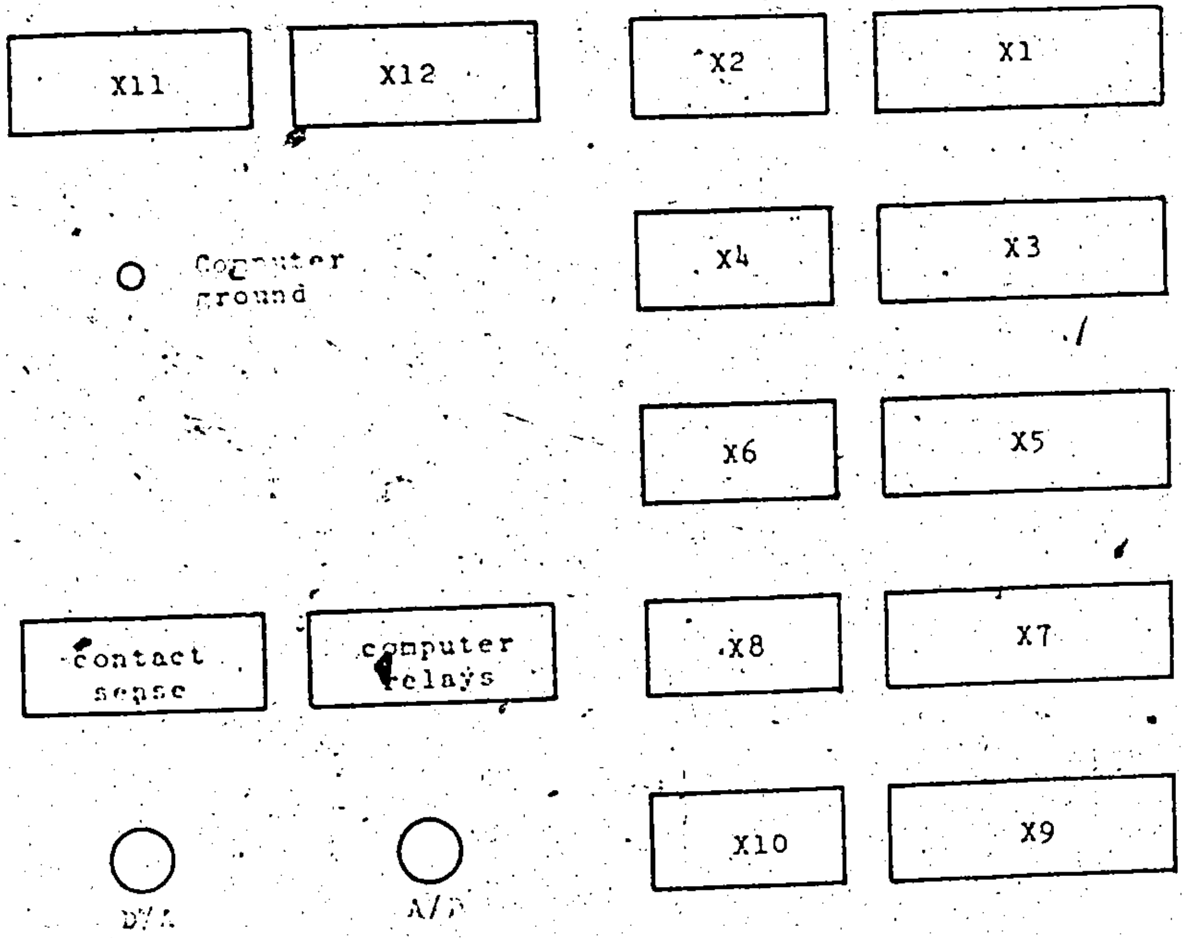
There are four terminal boxes located in the computer room for interfacing analog computers to the digital computer. Each box consists of two columns of four pairs of terminals, forming a 4 X 4 matrix,plus a ground connector.Boxes 1 and 2 are connected to the interface panel at X11,and boxes 3 and 4 are are connected to X12 by a similar cable.Both X11 and X12 are 36-pin Amphenol connectors.Pins 1 to 8 and 19 to 26 are used by the even-numbered boxes,and pins 11 to 18 and 29 to 36 are used by the odd-numbered boxes. These pins are connected to four 4 x 4 sections on the interface panel, such that the rows and columns are identical to the rows and columns of the boxes.

The four sections use columns A to P, rows 19 to 22; i.e.- four 4 x 4 sections in a column, with sections, from top to bottom, representing boxes 1 to 4. As an example, the pins for box A are connected in the following manner, -

11 to 14	column 19, rows A to D
15 to 18	column 21, rows A to D
29 to 32	column 20, rows A to D
33 to 36	column 22, rows A to D

The other three boxes are connected in the identical manner.

Figure B.1 Computer Site Interface Panel Cable Connections



Appendix C

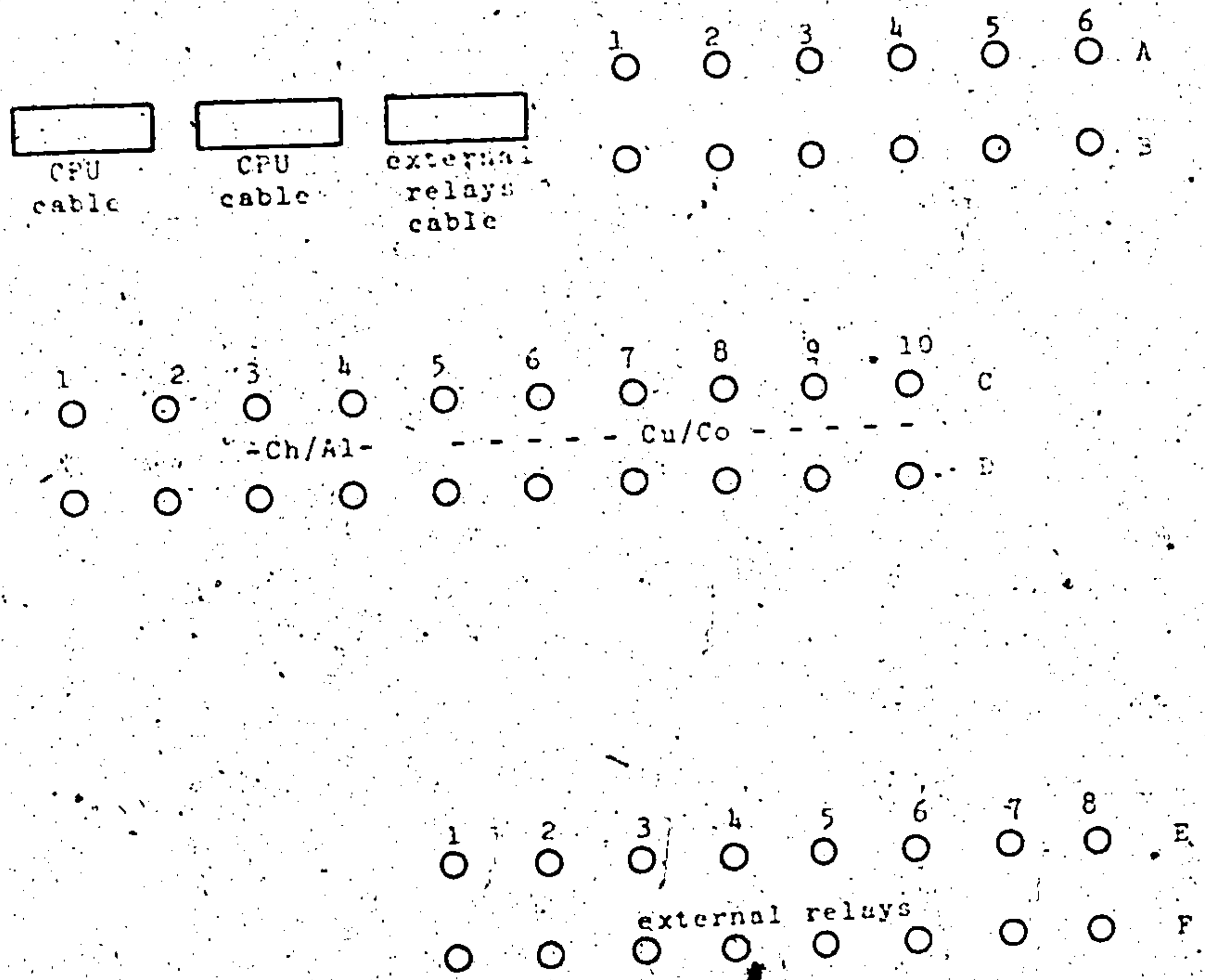
Interface Panels Connections

Computer Site Connections		Process Site Connections *		Use
from	to	from	to	
J1 to J8	B14	B2	F8	common-external relays
K1	A15	A3	E1	PRUN light
K2	A16	A4	E2	SOLFM light
K3	A17	A5	E3	FEEFM light
K4	A18	A6	E4	Overhead sample light
K5	B15	B3	E5	Bottom sample light
K6	B16	B4	E6	solvent heater
K7	B17	B5	E7	feed heater
K8	B18	B6	E8	reflux divider
L1	A13	A1		overhead switch
L2	B13	B1		bottom switch
M1	A14	A2		sample switch commons
Q1	A3	C5	+	reboiler thermocouple
R1	B3	D5	-	
Q2	A4	C6	+	column thermocouple
R2	B4	D6	-	
Q3	A5	C7	+	overhead thermocouple
R3	B5	D7	-	
Q4	A6	C8	+	solvent thermocouple
R4	B6	D8	-	
Q5	A7	C9	+	feed thermocouple
R5	B7	D9	-	
Q6	A8	C10	+	reference thermocouple
R6	B8	D10	-	
Q7	A9	C2	+	solvent DP cell
R7	B9	D2	-	
Q8	A10	C1	+	feed DP cell
R8	B10	D1	-	

NOTE: the computer site connections for the thermocouple inputs are made with pairs of leads which are connected via a 200 ohm resister

* see Figure C.1

Figure C.1 Process Site Interface Panel



Appendix D

Computer Software Program

Figures D.1 to D.12 are the logic flow diagrams for the main programs in the computer monitor/control software package. The symbols used are,

calculation

teletype output

decision

Table D.1 lists the programs in the software package and the code names for operator-initiated/terminated tasks.

To use the package, the operator loads and initiates XTRAC, the executive program. To start a task, the operator types "control E" and responds to the question with an acceptable code name. The four-character code names belong to programs which may be stopped by entering the code name terminated by an "S". Complete listings of all programs are available.

The flag bit column indicates which bit, if any, in JOBW, the program control word, each program will refer to to determine if that program is to continue running or if it is to halt.

Table D.2 lists the variables which may be changed using EXPAR. This routine obtains and prints the current value of the variable desired and allows the operator to change the values on-line. To implement a change, the operator enters the variable name, waits for the printout, and then types "control I" before entering the new value. Any other input after a value has been output will cause the last value printed to be stored in the memory.

Figure D.1

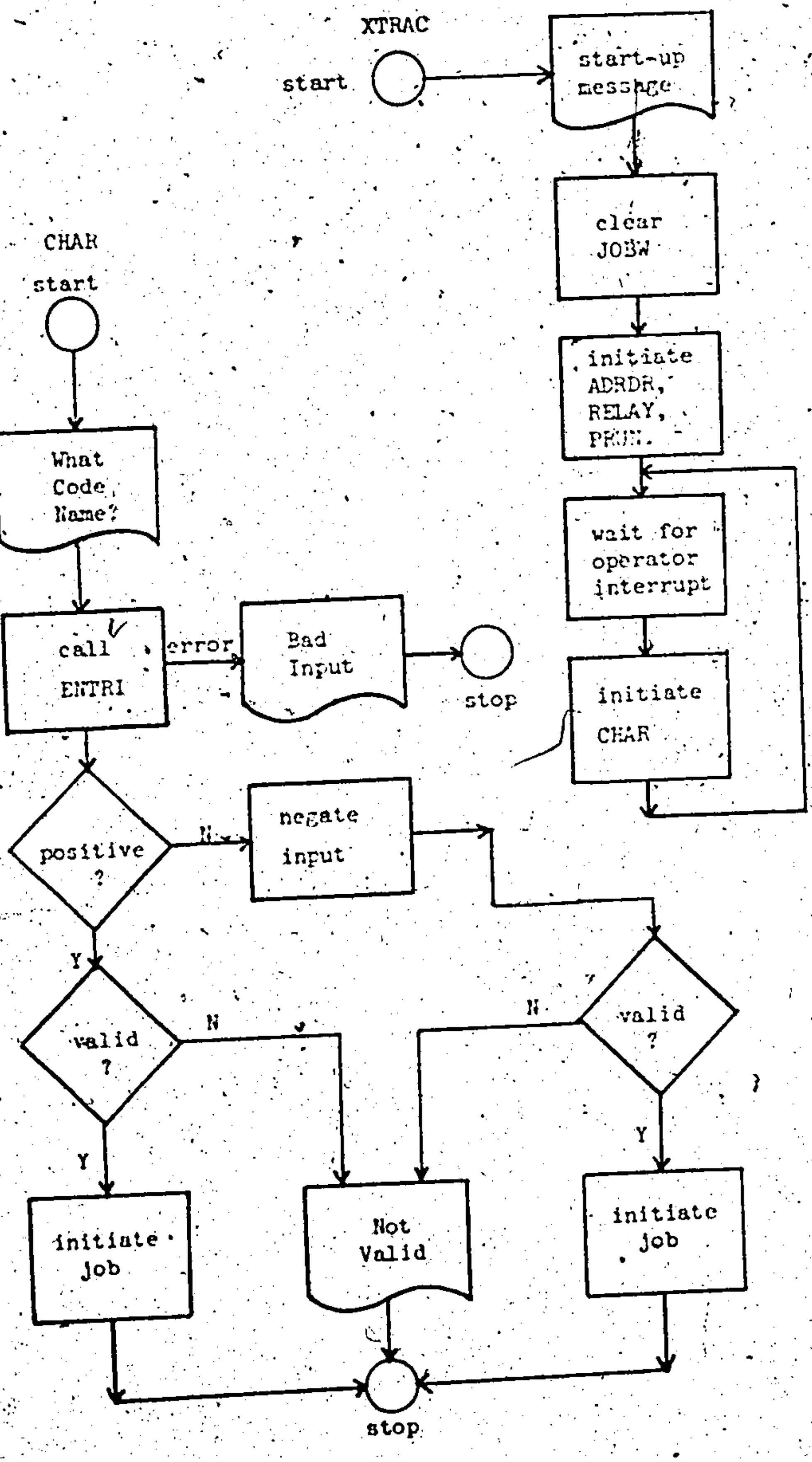

XTRAC
start
start-up message
clear JOBW
initiate ADRDR, RELAY, PRUN.
wait for operator interrupt
initiate CHAR
CHAR
start
What Code Name?
call ENTRI
error
Bad Input
stop
positive ?
N
negate input
Y
valid ?
N
Y
initiate job
Not Valid
valid ?
N
Y
initiate job
stop

Figure D.2

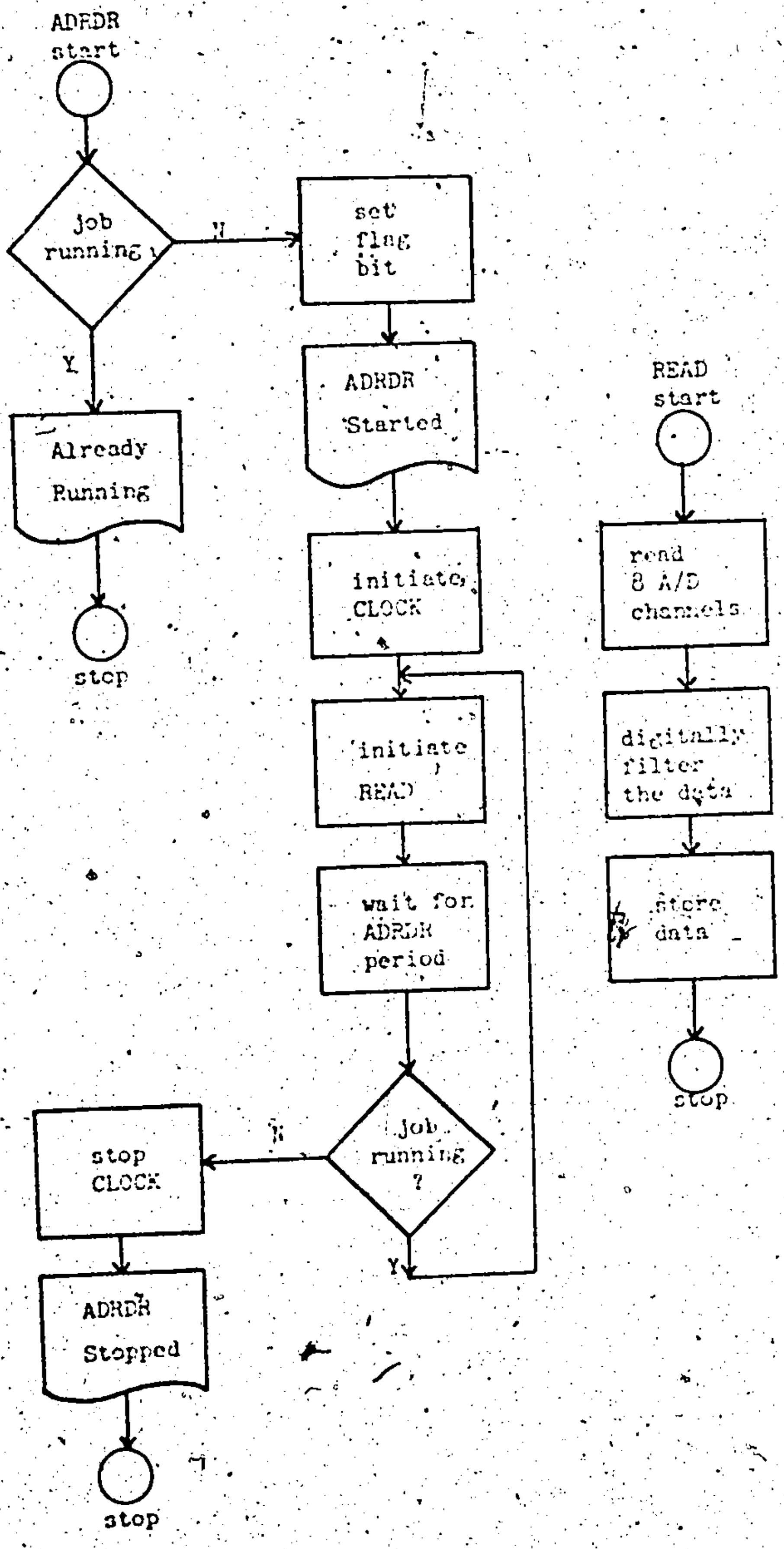

ADRDR start
job running ?
N
Y
set flag bit
Already Running
stop
ADRDR Started
initiate CLOCK
initiate READ
wait for ADRDR period
job running ?
N
Y
stop CLOCK
ADRDR Stopped
stop
READ start
read 8 A/D channels
digitally filter the data
store data
stop

Figure D.3

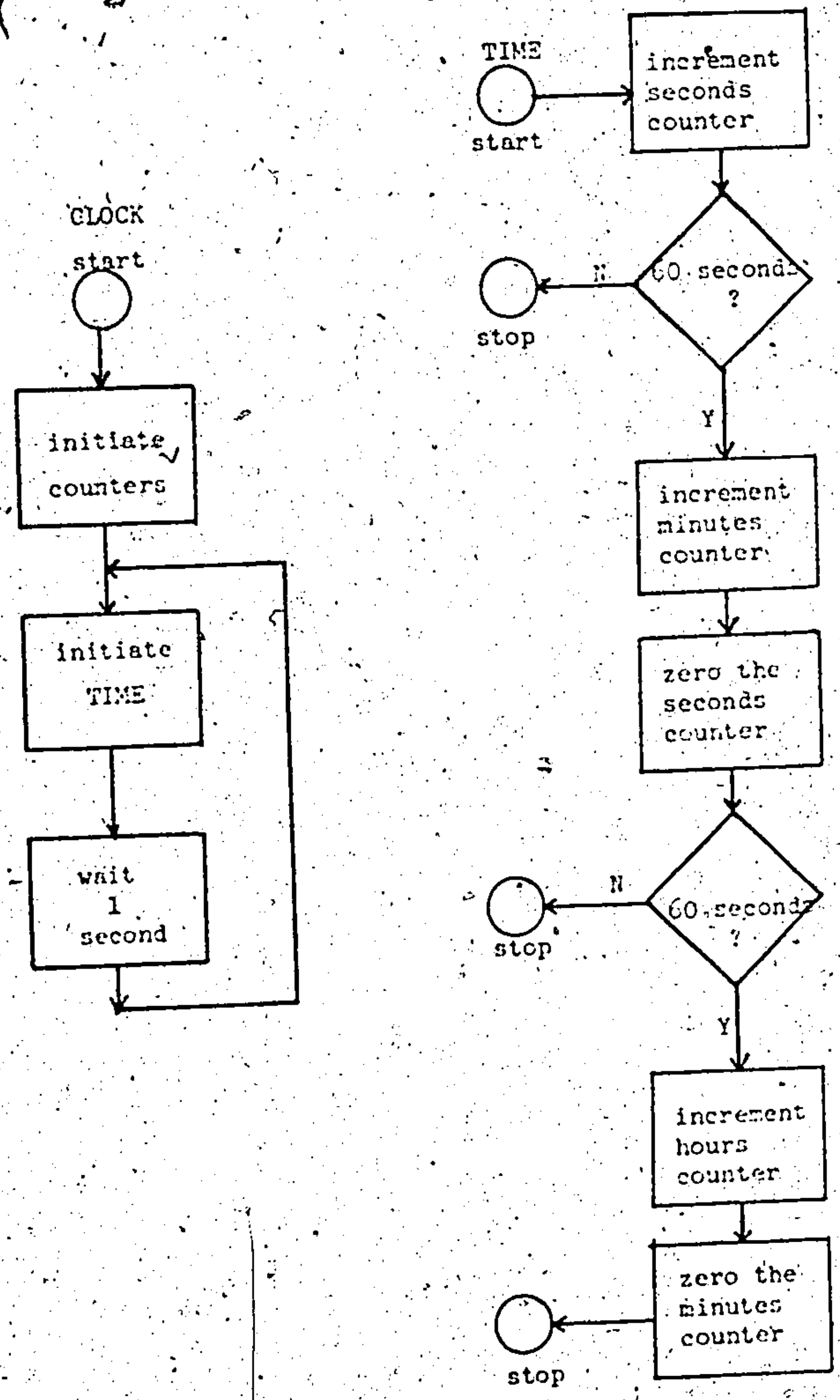

CLOCK
start
initiate counters
initiate TIME
wait 1 second
TIME
start
increment seconds counter
60 seconds ?
N
stop
Y
increment minutes counter
zero the seconds counter
60 seconds ?
N
stop
Y
increment hours counter
zero the minutes counter
stop

Figure D.4

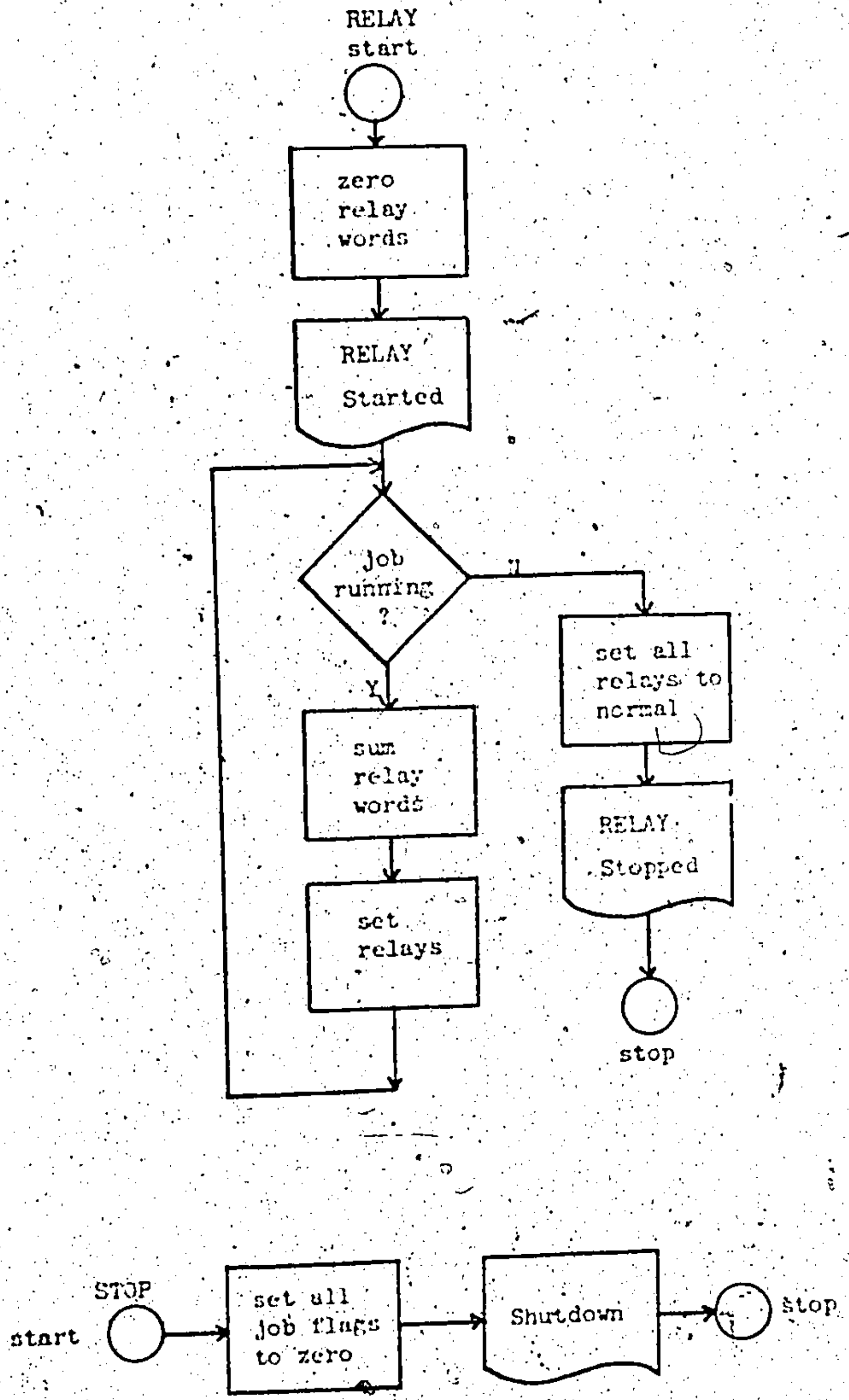
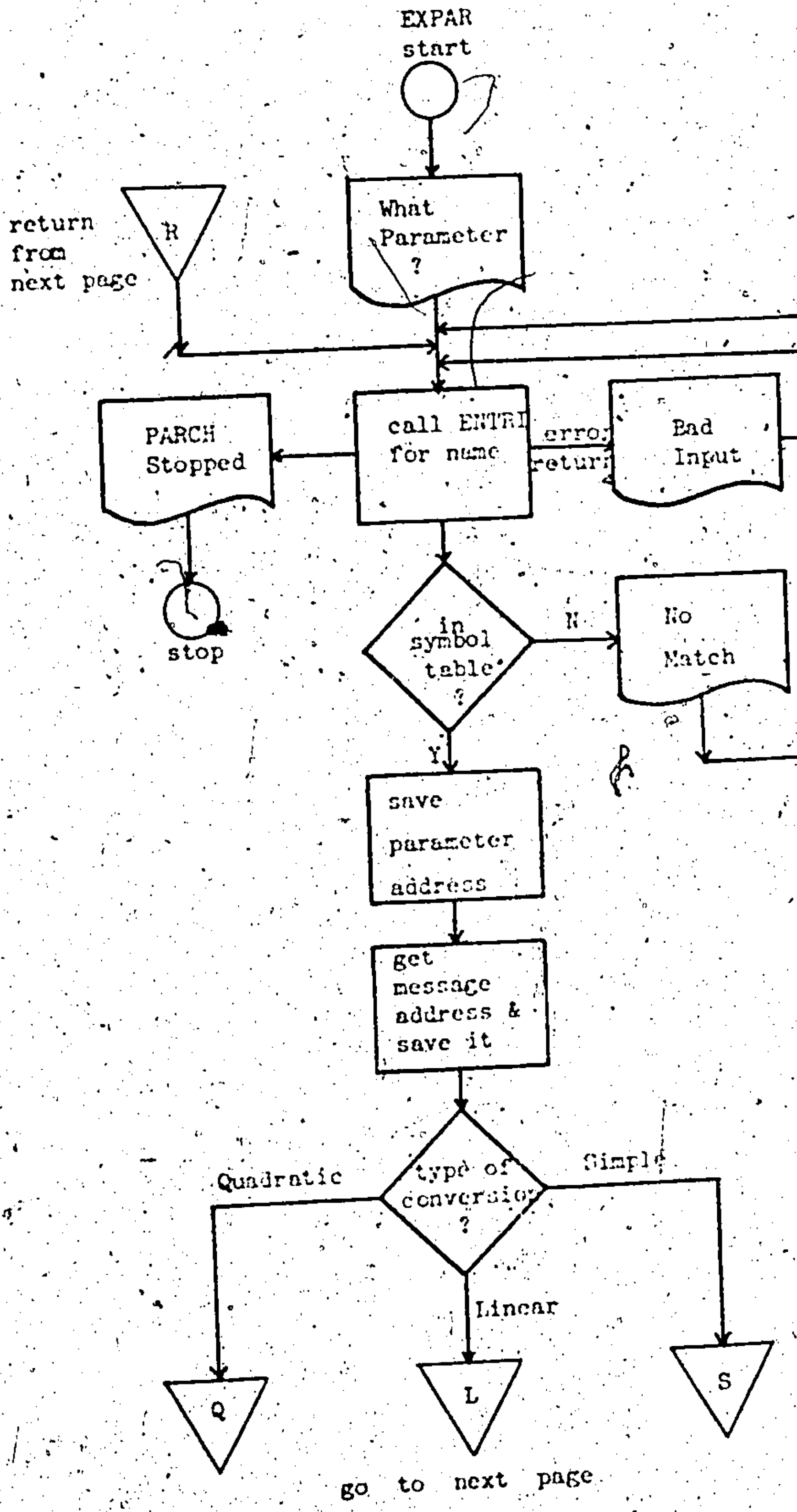
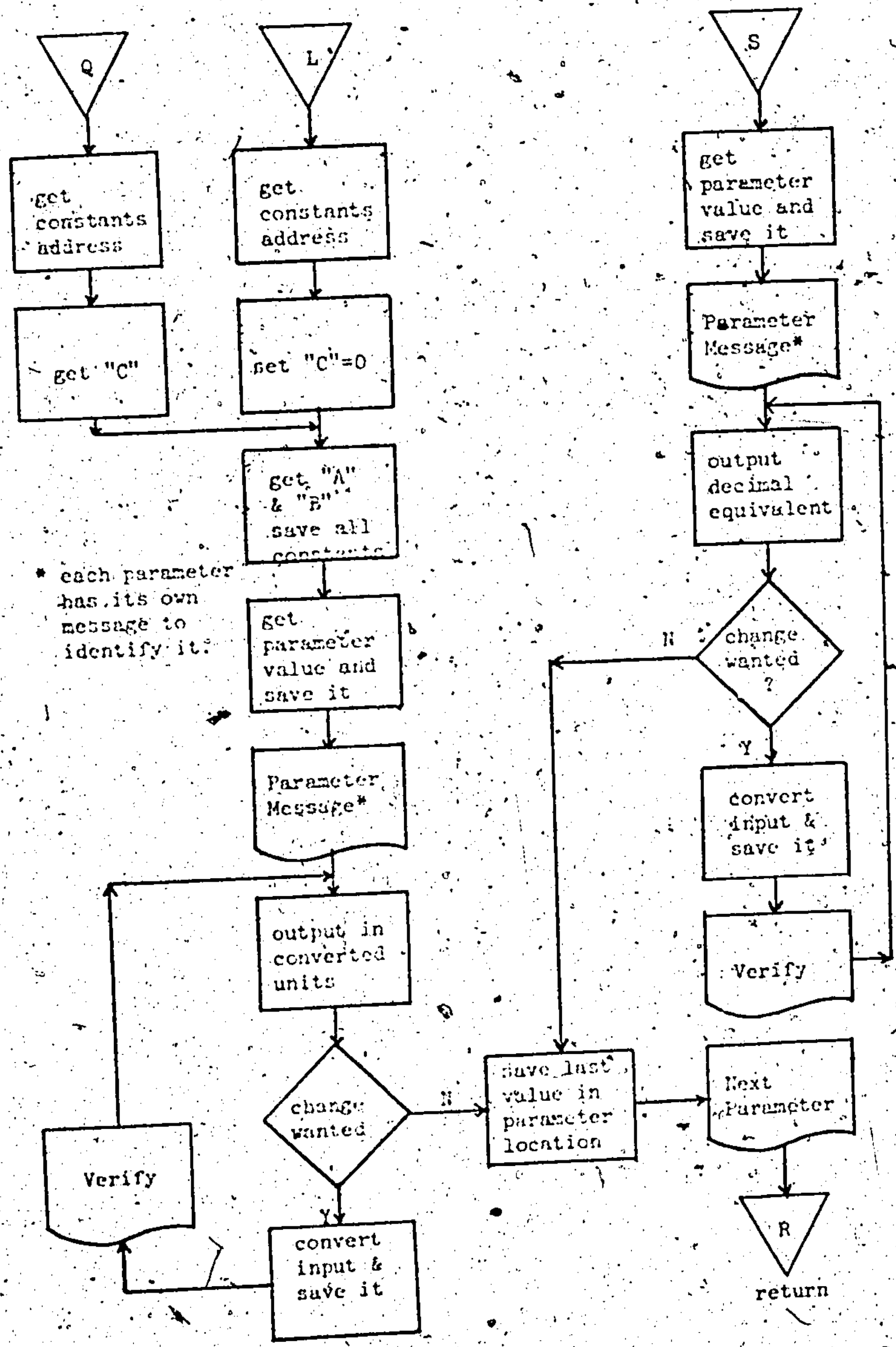

RELAY
start
zero relay words
RELAY Started
Job running ?
N
Y
sum relay words
set relays
set all relays to normal
RELAY Stopped
stop
STOP
start
set all Job flags to zero
Shutdown
stop

Figure D.5


EXPAR
start
return
from
next page
R
What
Parameter
?
PARCH
Stopped
stop
call ENTRI
for name
error
return
Bad
Input
in
symbol
table
?
N
No
Match
Y
save
parameter
address
get
message
address &
save it
Quadratic
type of
conversion
?
Simple
Linear
Q
L
S
go to next page



* each parameter has its own message to identify it.

Figure D.6

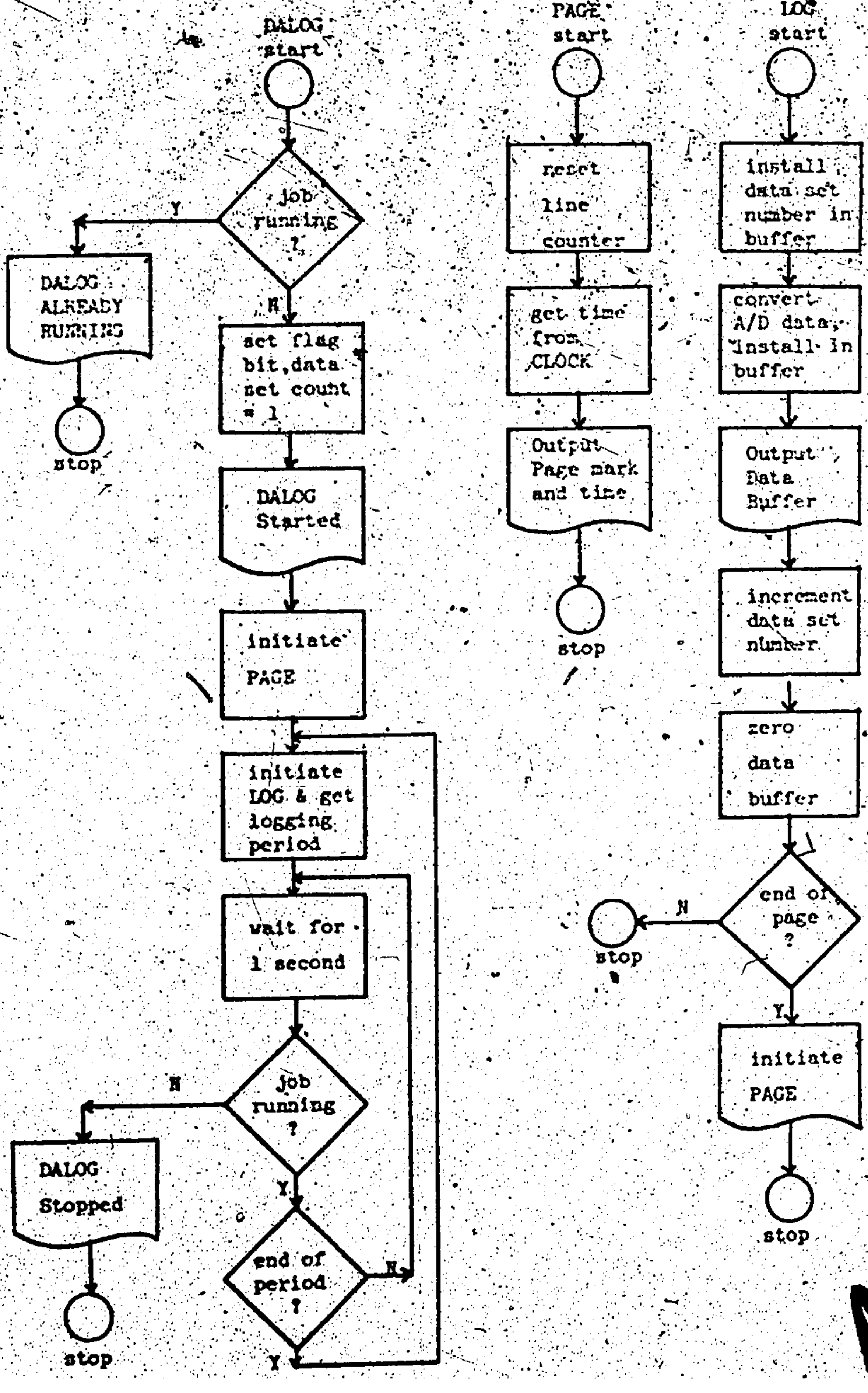
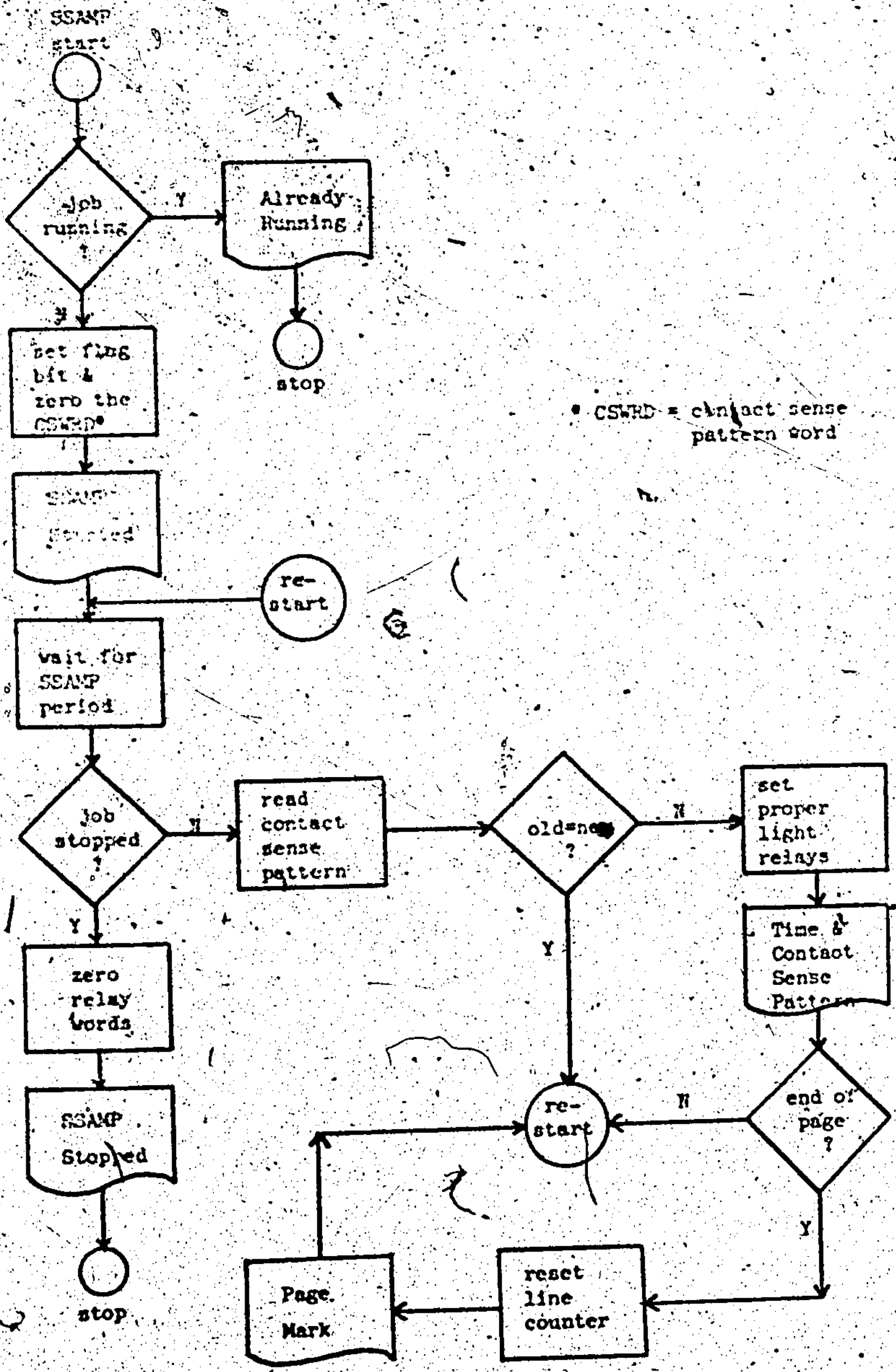

DALOG start
job running ?
Y
N
DALOG ALREADY RUNNING
stop
set flag bit,data set count = 1
DALOG Started
initiate PAGE
initiate LOG & get logging period
wait for 1 second
job running ?
N
Y
DALOG Stopped
stop
end of period ?
N
Y
PAGE start
reset line counter
get time from CLOCK
Output Page mark and time
stop
LOG start
install data set number in buffer
convert A/D data, install in buffer
Output Data Buffer
increment data set number
zero data buffer
end of page ?
N
stop
Y
initiate PAGE
stop

Figure D.7



* CSWRD = contact sense pattern word

Figure D:8

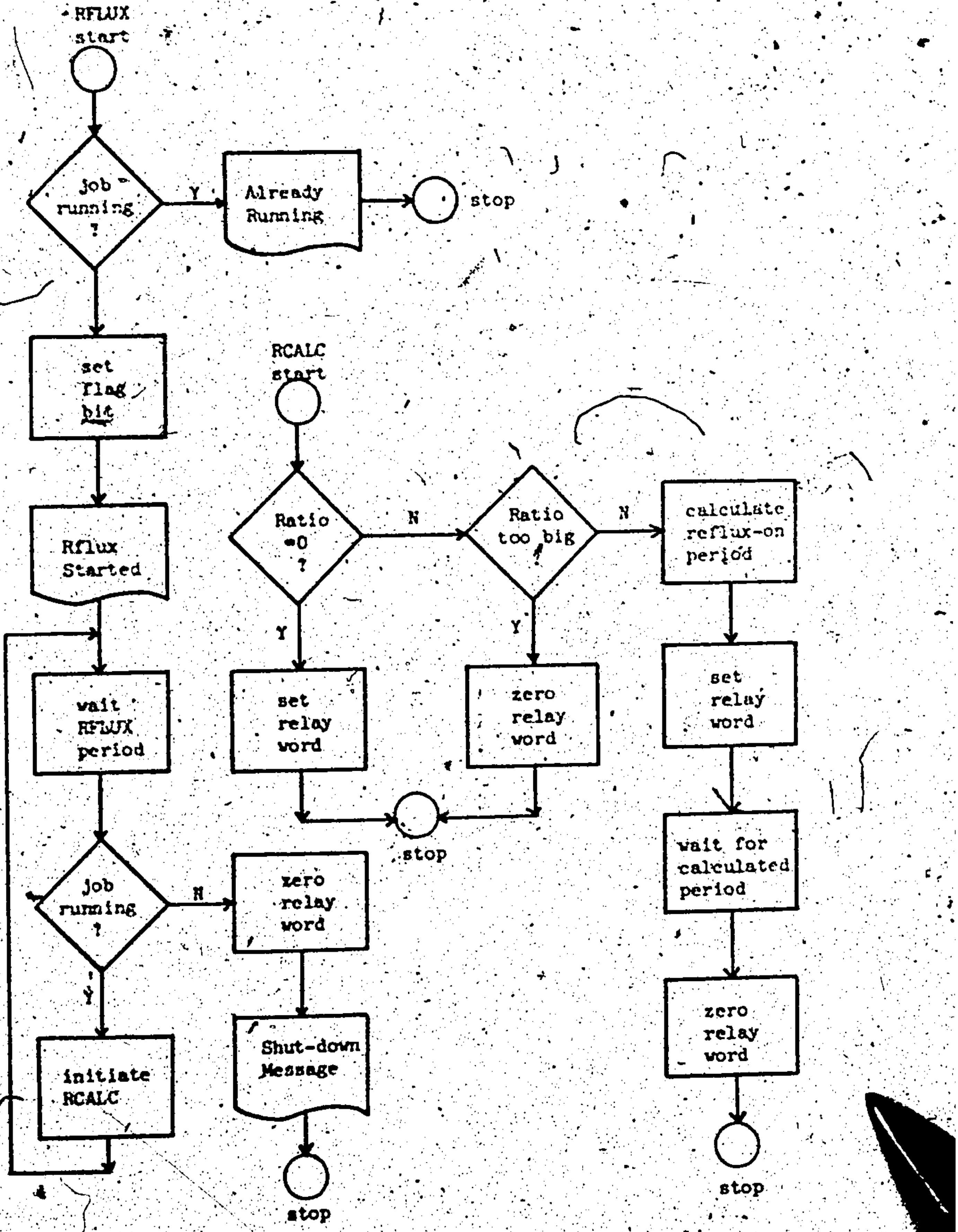

RFLUX start
Job running ?
Y
Already Running
stop
set flag bit
Rflux Started
wait RFLUX period
Job running ?
N
Y
zero relay word
Shut-down Message
stop
initiate RCALC
RCALC start
Ratio =0 ?
N
Y
set relay word
Ratio too big ?
N
Y
zero relay word
stop
calculate reflux-on period
set relay word
wait for calculated period
zero relay word
stop

Figure D.9

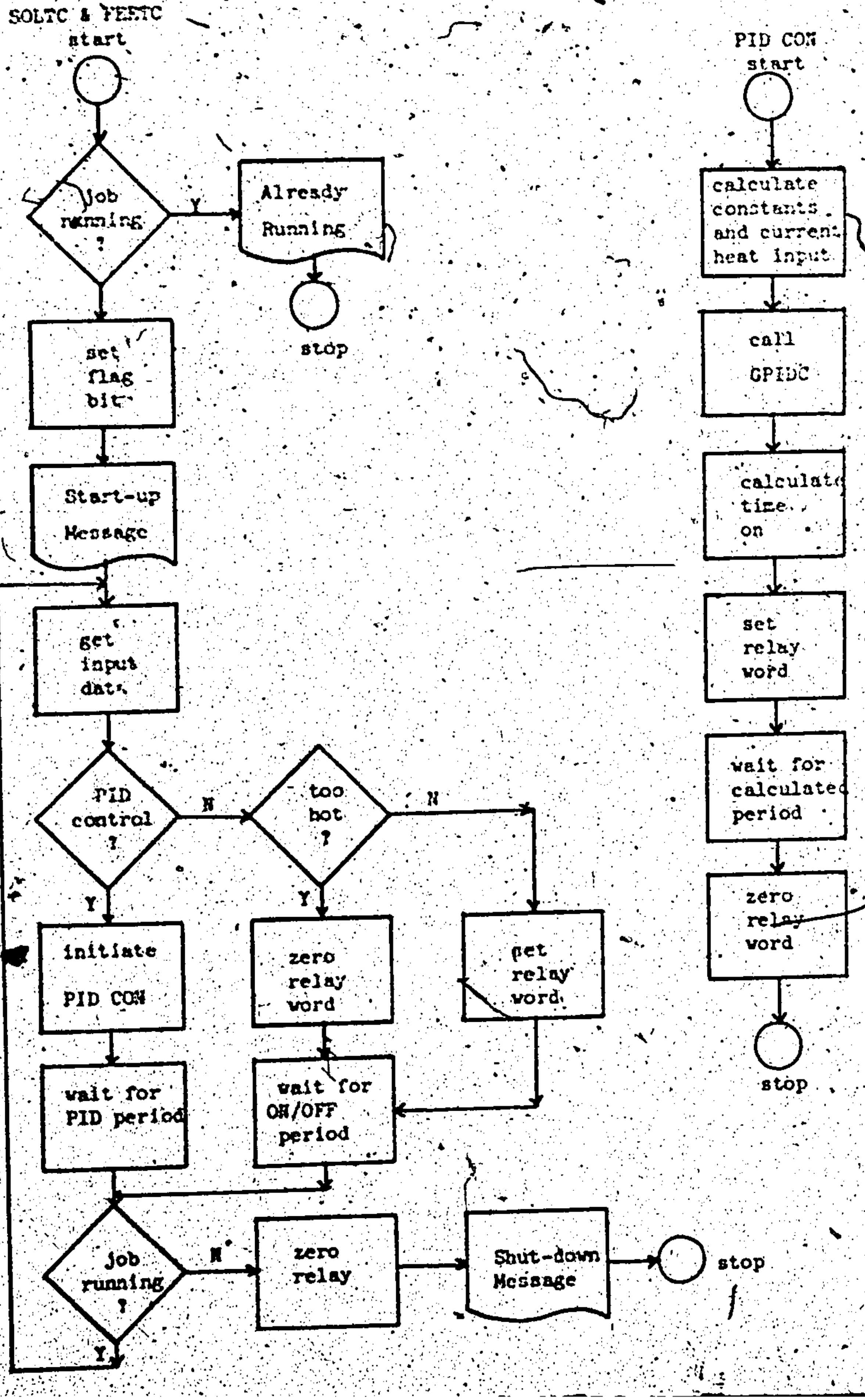

SOLTC & FERTC
start
job
running
?
Y
Already
Running
stop
set
flag
bit
Start-up
Message
get
input
data
PID
control
?
N
too
hot
?
N
Y
Y
initiate
PID CON
zero
relay
word
set
relay
word
wait for
PID period
wait for
ON/OFF
period
job
running
?
N
Y
zero
relay
Shut-down
Message
stop
PID CON
start
calculate
constants
and current
heat input
call
GPIDC
calculate
time
on
set
relay
word
wait for
calculated
period
zero
relay
word
stop

Figure D.10

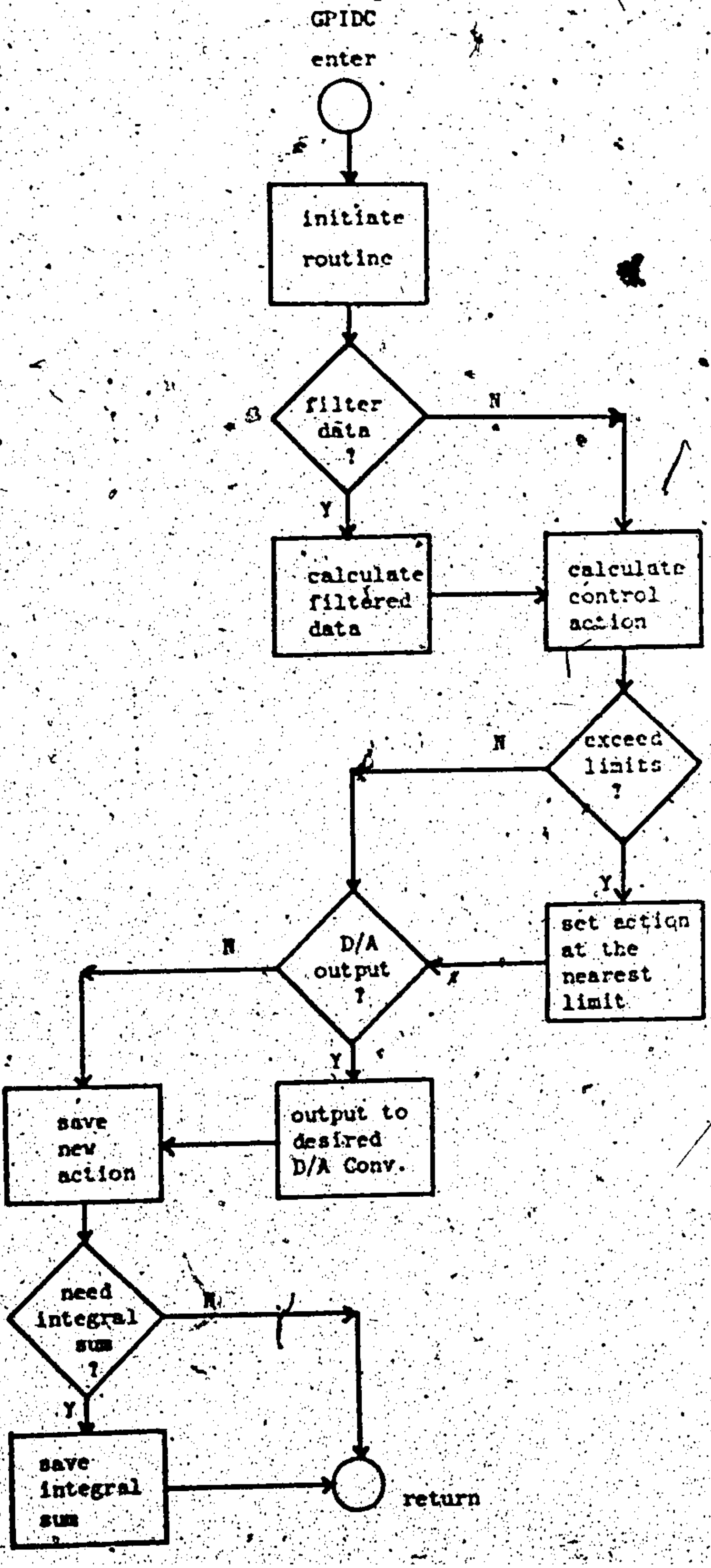

GPIDC
enter
initiate routine
filter data ?
N
Y
calculate filtered data
calculate control action
exceed limits ?
N
Y
set action at the nearest limit
D/A output ?
N
Y
output to desired D/A Conv.
save new action
need integral sum ?
N
Y
save integral sum
return

Figure D.11

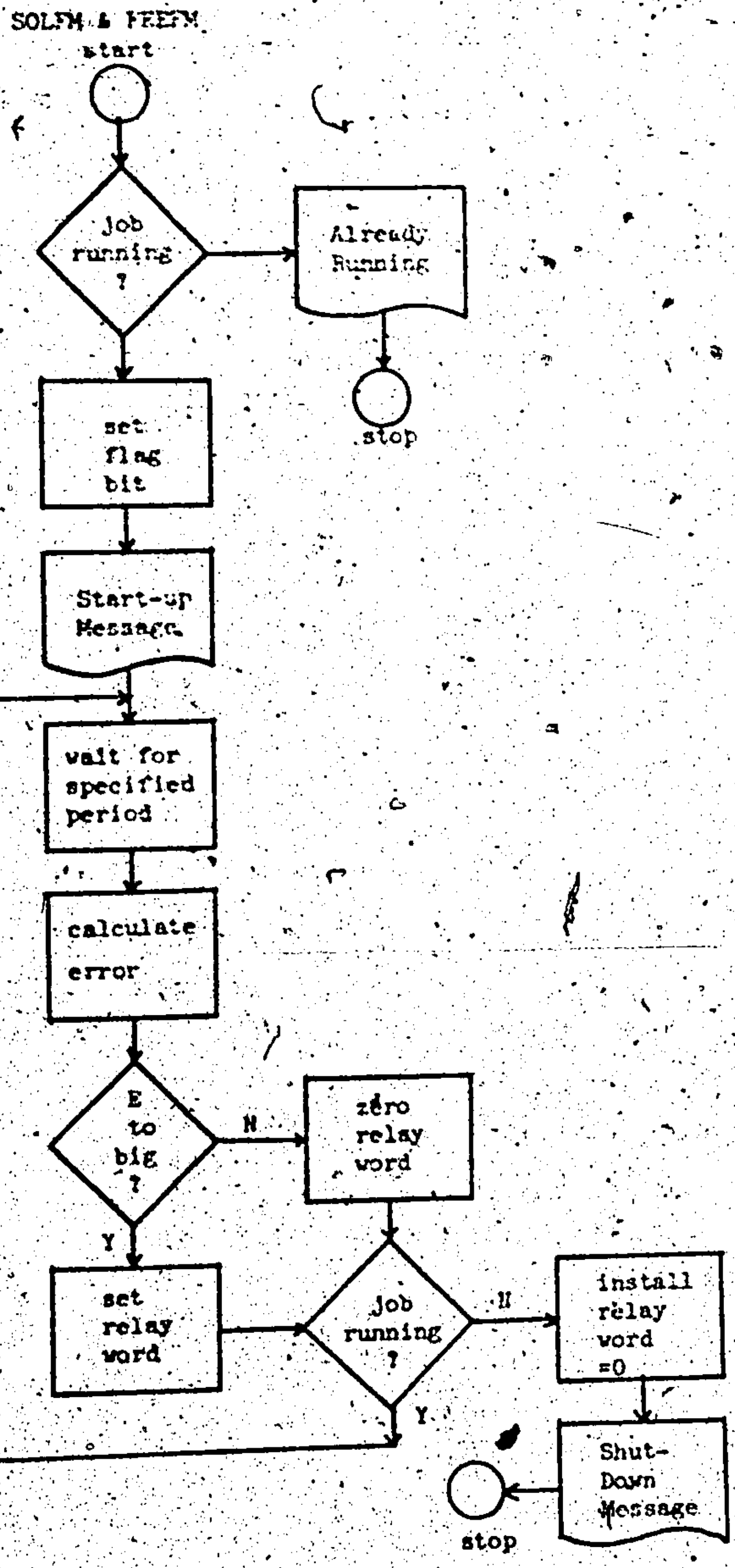

SOLFM & FREFM
start
Job running ?
Already Running
stop
set flag bit
Start-up Message
wait for specified period
calculate error
install new relay word
E to big ?
N
zero relay word
Y
set relay word
Job running ?
N
install relay word =0
Y
Shut-Down Message
stop

Figure D.12

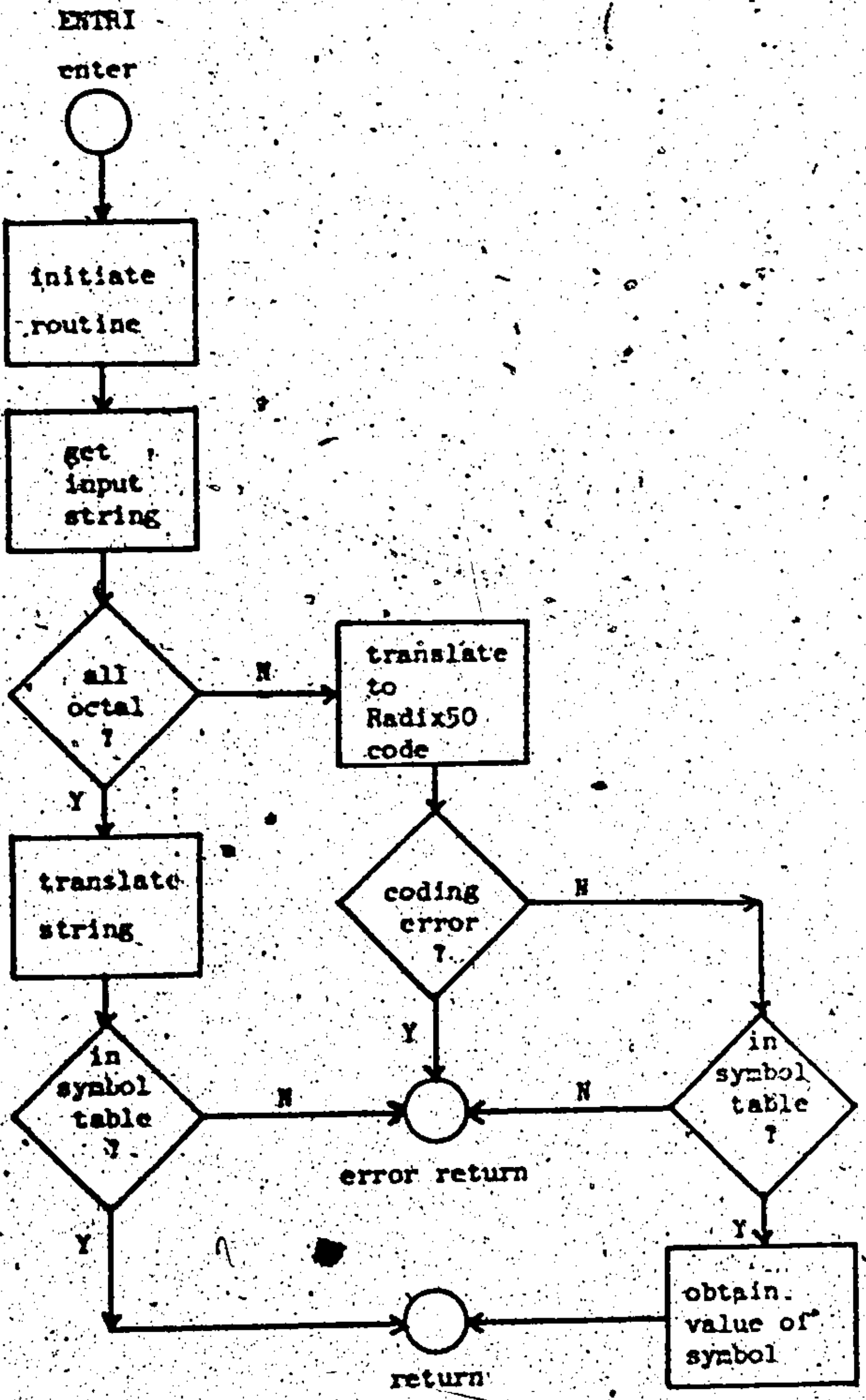

ENTRI
enter
initiate routine
get input string
all octal ?
N
translate to Radix50 code
Y
translate string
coding error ?
N
Y
in symbol table ?
N
error return
N
in symbol table ?
Y
Y
obtain value of symbol
return

Table D.1 Individual Program Information

Flag bit	Code Name	Program	Description
0	-	ADRDR	A/D reader
0	-	RELAY	Relay activator
0	-	PRUN	Program running
1	DLOG	DALOG	Data logging
2	SOLT	SOLTC	Solvent temperature controller
3	FEET	FEETC	Feed temperature controller
4	SOLF	SOLFM	Solvent flowrate monitor
5	FEEF	FEEFM	Feed flowrate monitor
6	SMPL	SSAMP	Stream sampling monitor
7	RFLX	RFLUX	Reflux divider
-	ENDAL	STOP	Shutdown
-	PARCH	EXPAR	Parameter change
-	-	XTRAC	Executive program
-	-	GPIDC	General proportional - integral - derivative controller
-	-	EXTAB	Constants table for GPIDC
-	-	MATHJ	Various math subroutines
-	-	ENTRI	Accept octol of ASCII inputs from keyboard and search symbol table for match

Table D.2 Parameters to Change With EXPAR

Program	Variable Name	Meaning
ADRDR	ADPER	ADRDR period
	ADREF	reference thermocouple reading
DALOG	LOGP	DALOG period
	TA,TB,TC	temperature conversion constants
	SA,SB,SC	solvent flow conversion constants
	FA,FB,FC	feed flow conversion constants
SOLTC	SPIDP	SOLTC - PID period
	SNOFP	SOLTC - ON/OFF period
	SCB	SOLTC control bit, 0=PID, 1=ON/OFF
	STSET	solvent temperature set point
	STIN	solvent temperature in
	STMAX	solvent temperature maximum
	SFSET	solvent flow set point
	SFMAX	solvent flow maximum
FEETC	FPIDP	FEETC - PID period
	FNOFP	FEETC - ON/OFF period
	FCB	FEETC control bit, 0=PID, 1=ON/OFF
	FTSET	feed temperature set point
	FTIN	feed temperature in
	FTMAX	feed temperature maximum
	FFSET	feed flow set point
	FFMAX	feed flow maximum
EXTAB	KP	proportional gain
	KI	integral gain
	TD	derivative time
	SF	scale factor
	VA	valve action
	FFAC	filter factor
	UL	upper limit
	LL	lower limit
		Note: loop 0 for SOLTC, loop 1 for FEETC
FLOWM	SFMP	solvent flow monitor period
	SFLIM	solvent flow error limits
	FFMP	feed flow monitor period
	FFLIM	feed flow error limits
SSAMP	SSPER	SSAMP period

RFLUX	RFLXP	RFLUX period
	RATIO	reflux ratio, maximum = 10 if RATIO 10, total reflux

Appendix E Process Start-Up and Operation

Start-up procedure for the extractive distillation column.

1. connect process equipment to recorders or computer interface panel
2. start computer monitor/control package if operating with computer aid
3. load feed and solvent storage tanks
4. prepare thermocouples and bottom sampler ice bathes
5. turn on cooling water and hot air gun (∼52V)
6. turn on level controllers
7. turn on reboiler heater (∼60V)
8. when vapour reaches mid-column, decrease reboiler heater (∼55V)
9. start solvent pump (∼10ml/min) and turn on solvent heater (∼27V for manual operation, ∼35V for computer aided operation)
10. when bottom 3 plates loaded, slowly start feed and turn on feed heater (∼16V manual, ∼25V computer aided operation)
11. when bottom 8 plates loaded, slowly adjust solvent and feed flows and temperatures to desired values
12. when total column loaded, start reflux divider

For manual operation, the stream heaters must be adjusted to maintain the stream temperatures at the desired set points. The temperatures are recorded on a 12-point Honeywell recorder. The stream flowrates must be frequently checked and adjusted.

For computer-aided operation, the computer turns the stream heaters on and off to maintain the desired temperatures and monitors the stream flowrates. With the computer-aided operation, the operator need adjust the stream flowrates only when requested to by the computer.