THE APPLICATION OF COMPUTER CONTROL TO THE PRODUCTION OF SYNTHETIC RESINS

by

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SUMMARY

The work reported in this thesis was performed as part of a Teaching Company Scheme organised between BIP Chemicals Limited and the University of Aston. The overall aim of the Scheme was to maximise the efficiency and economics of the batch processes used for synthetic resin manufacture. This investigation covers the development of a computer control strategy to be used in the batchwise production of synthetic resins within the overall aim.

The measurement of composition of a recovered solvent feed was investigated to identify a way of monitoring the composition for use in control. This objective could be achieved using a dual analyser system comprising an Ultra-Violet analyser and density meter. Trials were made of a viscometer to provide on-line information about the reaction's progress during full scale production.

A computer control strategy was developed based on the control parameters of reaction temperature, mass of aqueous distillate removed and time. An experimental computer was connected to the instrumentation of a full scale production reactor. The control strategy was implemented using the computer with suitably structured control software. Trials were carried out under production conditions to confirm the strategy and its implementation.

Keywords

Computer Control

Synthetic Resins

Real-Time Control

Butylated-Amino-Formaldehyde Resin

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1. INTRODUCTION

The work presented in this thesis was carried out as part of a Teaching Company Project, established between BIP Chemicals Limited and Aston University in 1983. The task of the project was to investigate the production process used to manufacture butylated amino-formaldehyde resins. The purpose was to propose actions leading to substantial improvements in consistency of both the batch operation and the product quality. Details of the project are given in Appendix 1.

The resins produced by the process are commonly referred to as coating resins; due to their application as a constituent in various paint and surface finishes. The resins produced are variants on a basic resin type. The process plant used is a typical multi-product batch production facility consisting of four reactors each with different configurations. These are largely manually controlled and monitored. If control is not applied correctly, a batch of resin may fall outside its specification limits and, therefore require reworking or possibly scrapping.

There were four research associates working on the Teaching Company Project, each with specific areas of interest. The completed project has resulted in several successful outcomes which are briefly described as follows :-

(i) Improved methods of determining the product quality have been identified and implemented on the production plant.

(ii) A mathematical model for the chemical reaction kinetics was developed in order that a better understanding of the process could be gained. In producing this model an additional benefit was obtained since improved methods of off-line analysis of the final products and intermediates were established.

(iii) In conjunction with the modelling work more appropriate process control measurement parameters were identified allowing the process to be controlled with a greater degree of consistency.

(iv) One of the raw material streams which introduces variability into the system is the recovered solvent stream. This variability has been reduced in two ways. Firstly, improvements have been made to the processing of the recovered solvents on a distillation unit. Secondly, methods of determining the composition of the recovered solvent streams have been identified leading to an improvement in charge consistency.

(v) The final achievement in this project was the interfacing of a control computer to one of the commercial reactors and its subsequent use to control the production of resins on this unit.

The specific area of the work reported in this thesis is the development and refinement of a computer control strategy that could be applied to a full scale production facility in order to prove the feasibility of the control philosophy. In order to do this, work was carried out to identify an on-line chemical analytical technique which could be used to determine the composition of the recovered solvents charged to the reaction vessel, so that the information could be used for control purposes.

Work was carried out on the installation of a micro-computer system interfaced to one of the reaction vessels at BIP. This was done in several stages by the development of both the installed hardware and the writing of software. Initially the process was monitored by the computer and process actions displayed on a terminal for operator response. Once this system had been proved, additional hardware was installed in the computer and software written so that the batch process could be controlled directly by the computer system. This system was tested on the plant during the production of several full scale batches. The results showed the feasibility of this control system within the limits imposed by the hardware available on the plant. It was shown that this control strategy could be easily developed further with the installation of an aqueous distillate weigh system having a higher accuracy and being less prone to fluctuations than the system in existance on the BIP plant, as such fluctuations proved a problem during the trials. Also the installation of a more powerful computer system with more Input and Output capacity to plant instruments and devices would allow the development of a more automated process. The plant instruments and operating devices would need replacing to satisfy this requirement on the reactor system at BIP.

1.1 Literature

The literature covered in this investigation can be considered under three headings: process chemistry; control parameters; and, computer control. The latter can be sub-divided into the hardware and software requirements.

1.1.1 Process Chemistry

In order to consider the application of computer control to the processes involved in the manufacture of synthetic resins an understanding of the basic chemistry must be obtained. The work carried out previously within BIP(1) was reviewed and this identified the main chemical reactions involved. Other workers have examined the rates of chemical condensation(2) but under different reaction conditions to those investigated in this thesis. J.R. Ebdon(3) has carried out work using C^{13}NMR spectroscopy techniques to characterise the reactions of urea and formaldehyde, relevant to the process under study. This has shown that during the condensation reactions to form the resins, methylene-ether bridge formation, figure 1.1, is predominant over the formation of methylene bridges, figure 1.2.

$R\text{-}NH.\ CH_2.O.CH_2.HN - R$ .... Figure 1.1

Methylene ether bridge

$R\text{-}NH.CH_2.HN\ R$ .... Figure 1.2

Methylene bridge

This literature has been reviewed in more detail by V.H. Patel(21) whose work confirms that methylene bridges need not be considered in the industrial process.

1.1.2 Control Parameters

Process control is based on measurements and observations which are made throughout the process in order that the end points for the various reaction stages can be determined and the appropriate actions taken. From a consideration of process chemistry and laboratory and plant observations P.A.Freeman(4) has proposed several measurements which can be used at various stages of the process to ensure effective control. The key measurements which have been determined are reaction time, reactor temperature and the mass of aqueous distillate removed from the system. They provide the basis for the development of the computer control strategy described in this thesis.

1.1.3 Computer System

This area of investigation can be separated into the topics of hardware and software. D.J. Fraude(5) has described several factors relevant to the selection of hardware to apply computer control to batch reactors. These include the choice of sensors, automation concepts and microprocessor based and distributed systems. In a similar fashion, R.J. Uhlig(6) describes the application of computer control to the manufacture of phenolic resins. Other factors in implementing effective control are discussed in a paper by W.R. Hughes(7) on process interfacing. All the work reviewed has highlighted the need for reliable control measurements to be available in order to monitor the reaction. These should be capable of accurate determination using suitable instrumentation under normal process conditions. The review has also highlighted some factors to be considered when selecting a computer system to fulfill the required role for the successful implementation of the control strategy on a production facility.

The basic software requirements for a computer control system and at least one programming language. It is necessary to select an operating system which can respond to events which are occurring on the process plant in sufficient time to collect or send the necessary information. This is termed real time operation. At the same time it should support the concurrent running of several programs which may be performing functions such as data logging or control calculations. This is termed multi-tasking. A paper on real time operating systems and multi-task programming by J.D. Wright and J.W. Wright(8) describes the structure of an operating system needed to meet these requirements and the communications within the system to the input and output devices.

In addition one of the most important areas to be considered is that of the Man/Machine interface which is influenced by both the hardware and software considerations. The present trend is towards Visual Display Units, VDU , with a number of researchers working on topics such as pattern recognition and operator response times. Essentially these investigations have been aimed at discovering the best way of presenting all the required information in a form which can be readily understood by the operator so that quick and effective action can be taken. A brief description of the areas of work which are currently being investigated follows:

Shneiderman(9) has investigated the relationship between the response times of operators and the display refresher rates. N. Mehta(10) has developed a method of producing an operator interface based on the use of a T.V. camera focussed on a panel and generating signals in response to optical events.

The design of VDU displays has been investigated by S.S. Reilly and J.W. Roach(11) who have employed certain techniques commonly used in the advertising industry for the design of posters and displays, in order to highlight the important points. R.F. Sapita(12) has developed several of these methods to produce a control strategy for use in process control, from controller layouts through to strategic pattern recognition.

Such work has demonstrated the need to present information on a display unit in a manner which is easily recognised and understood by the operator before he takes any action. It has also shown that the displays should not be over complicated and should display only the information which is necessary for that stage in the process. There should also be a facility to call up additional information at any time to compliment the main display should any situation require a check to be made by the operator to support any decision made.

1.2 Process Chemistry

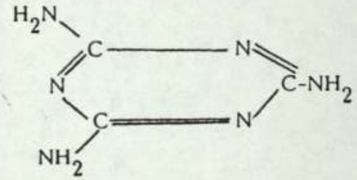
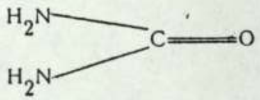
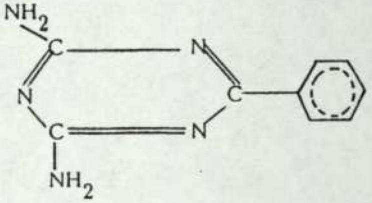
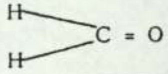
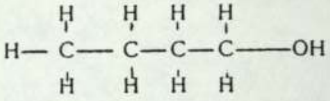
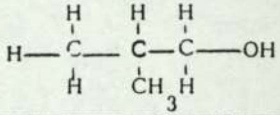
The general term amino-formaldehyde resin is used to refer to several standard resin categories commonly called coating resins. The main reactants in the chemical formulations are :-

1) either, melamine, urea or benzoguanamine

2) formaldehyde, in the form of formalin and

3) either, normal butanol or iso-butanol

TABLE 1.1 Reactants used in the production of Butylated Coating Resins

Compound	IUPAC$^+$ Name	Chemical Structure	Molecular Weight
Melamine	2,4,6 - Triamino-s-Triazione		126 ·10
Urea	Carbonyl-diamide		60·6
Benzoguamine	2,4 Diamino-6-Pheynl- s-Triazine		187.2
Formaldehyde	Methanol		30.03
Normal-butanol	1-butanol	 $H-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-OH$	74·12
iso-butanol	2-methyl-1- propanol	 $H-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{CH_3}{C}}-\overset{H}{\underset{H}{C}}-OH$	74·12

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The chemical structures of these constituents are given in Table 1.1. Melamine, Urea and Benzoguanamine are the base constituents providing the amino groups required to carry out the various chemical reactions. One is chosen for any resin and the names are used to identify the three main resin categories. These compounds are solid under normal conditions and are used in powder form in the commercial process.

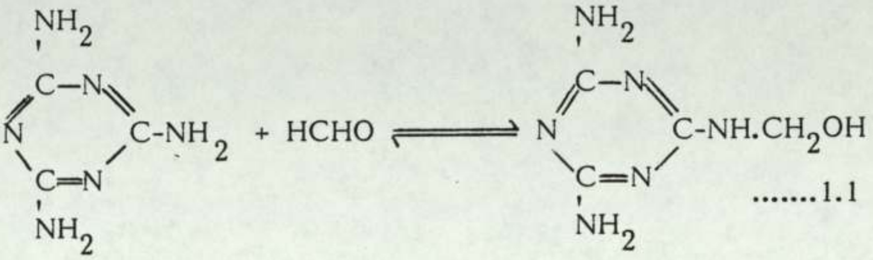
Formalin is the familiar name given to aqueous solutions of formaldehyde. The strength used in the process is 44% formaldehyde in water, with 3% w/w methanol also being present because of the manufacturing route. The formalin is used to combine with the reactive hydrogens in the base materials, with the extent of reaction being dependant on the ratios of formaldehyde to base material in the original charge. Furthermore, the number of formaldehyde groups, methylols, reacted onto the base material affects the subsequent polymerisation reactions and the products which are obtained.

Normal or Iso-butanol is used to etherify the polymerised resin and hence alter the properties of the final product.

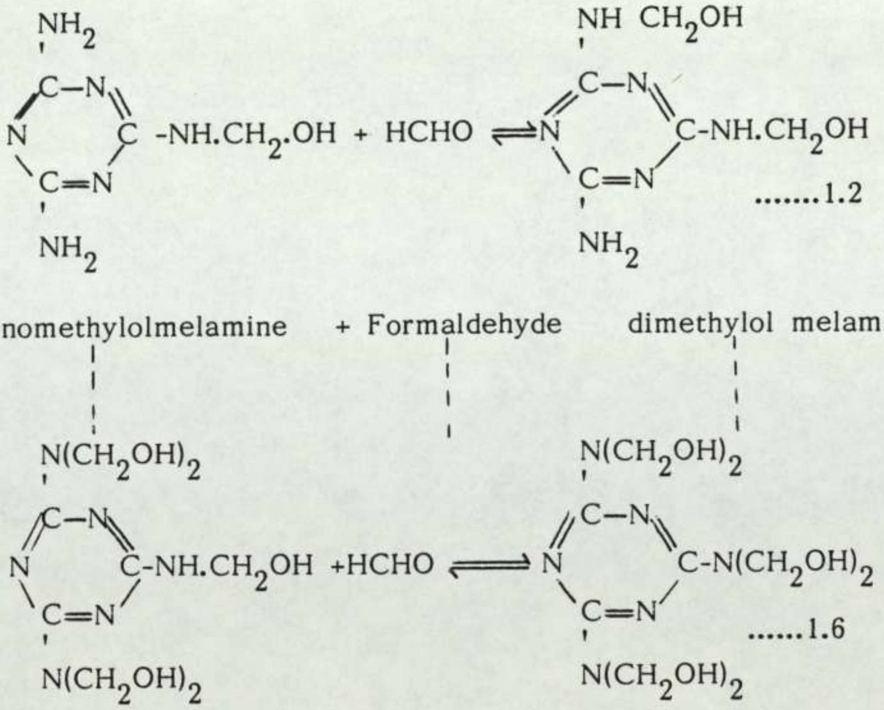
The majority of the experimental work reported in this thesis was conducted on a group of three commercial resins which will be referred to as Types, A, B and C resins. Each is an n-butylated melamine formaldehyde resin, the difference between each being in the degree to which each resin is butylated. There are three main reactions which occur when producing such a resin, methylolation, condensation and butylation(1).

1.2.1 Methylolation

This is an addition reaction and is the first to occur in the reactant mixture. It proceeds with the combination of formaldehyde and melamine to form methylol melamines. The melamine molecule has six reactive hydrogens so depending on the initial molar ratio and reaction conditions up to the hexamethylol melamine molecule can be formed (13,14,15). There are nine possible different methylol melamine molecules which can be formed by several reaction routes. A series of typical reactions are shown in equations 1.1 to 1.6.



Melamine + Formaldehyde Monomethylolmelamine



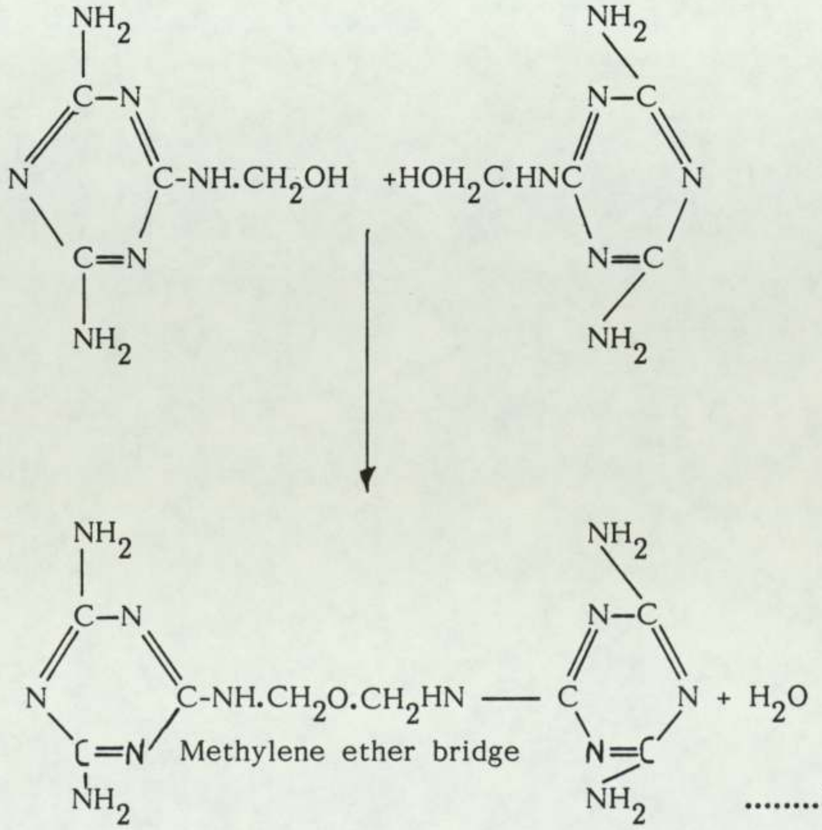
Pentamethylol melamine +Formaldeyde hexamethylol melamine

These reactions can continue until all the reactive hydrogens are used or there is no more formaldehyde available. In the case of A, B and C resin it has been shown through chemical analysis, by V.H. Patel(16) that an average four moles of formaldehyde are reacted per mole of melamine.

1.2.2 Condensation

This is the stage of the process in which the polymerisation reaction predominates over the butylation reaction. The methylol melamines formed by the addition reactions, react together producing polymers of increasing molecular weight. and structural complexity. One main reaction has been shown to occur(16). This is the reaction of two methylol groups from methylol melamine molecules to produce a methylene ether bridge as illustrated by equation 1.7 and shown in Section 1.1.

Methylene Ether Bridge Formation



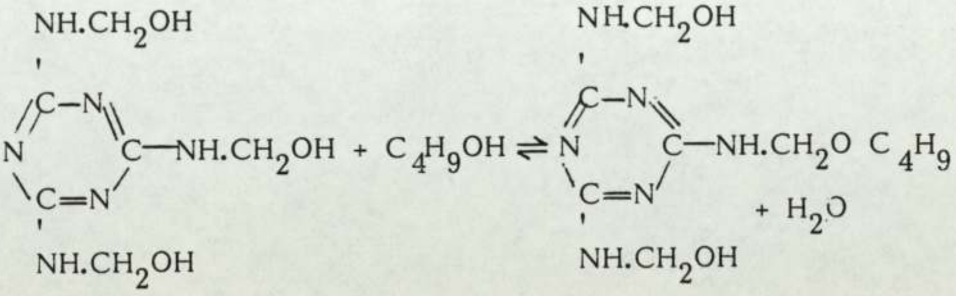
........1.7

As the condensation reaction illustrated above proceeds and the chain length increases then a viscous resin is produced. The greater the degree of condensation then the more viscous the resin becomes.

The reaction illustrated above shows in general terms what happens during the condensation stage of the process, specific data on the reaction rates and kinetics are not available. Investigations carried out by Okana and Ogata(2) into the condensation rates were made at different reaction conditions to those employed in the manufacturing process and therefore cannot be used. V.H. Patel has made progress in developing the understanding of these reactions.

1.2.3 Butylation

During this stage of the reaction the amino resin is modified by the combination of butanol with the methylol groups of the resin as illustrated by equation 1.8.



This reaction produces water and since this reaction is an equilibrium reaction the water must be removed if the butylation reaction is to proceed in the forward direction. The butanol present in the reaction system fulfills two functions. Firstly, it is a reactant. Secondly, it acts as the solvent which controls the overall concentration of the reaction species and hence controls both the overall rate and extent of reaction. It is therefore important that the butanol is not removed from the system during reaction.

The reaction with butanol of the amino-formaldehyde resin is required to increase the compatability of the resin with other resins and solvents. Therefore, for any one particular product to be obtained the condensation and butylation reactions must take place in the correct balance and so the required butanol concentration must be maintained throughout the reaction if the product quality and consistency are to be achieved.

1.2. 4 Water Removal

For the butylation reaction to proceed it is essential that the water is removed from the reactant system efficiently otherwise the possibility exists for the condensation reaction to continue until the reactant mixture gels. This is because the butylation reaction is an equilibrium reaction producing water. If too much water is present in the system then the butylation reaction is inhibited and the condensation reaction occurs preferentially. This can lead to the gelation of the reactant mixture.

The water present in the raw materials and the water produced by the reactions are removed from the mixture by evaporation. When boiling occurs the vapour contains a different concentration of the solvents and water from that which is present in the reaction mixture. Both n- and iso-butanols and water form a minimum boiling point azeotrope having a composition well outside the mutual solubility range of the two components in the liquid phase. Therefore when this azeotrope is condensed, two separate liquid layers are formed, the top layer being mainly solvent. If only these two components are present then the equilibrium separation is as in Figure 1.3.

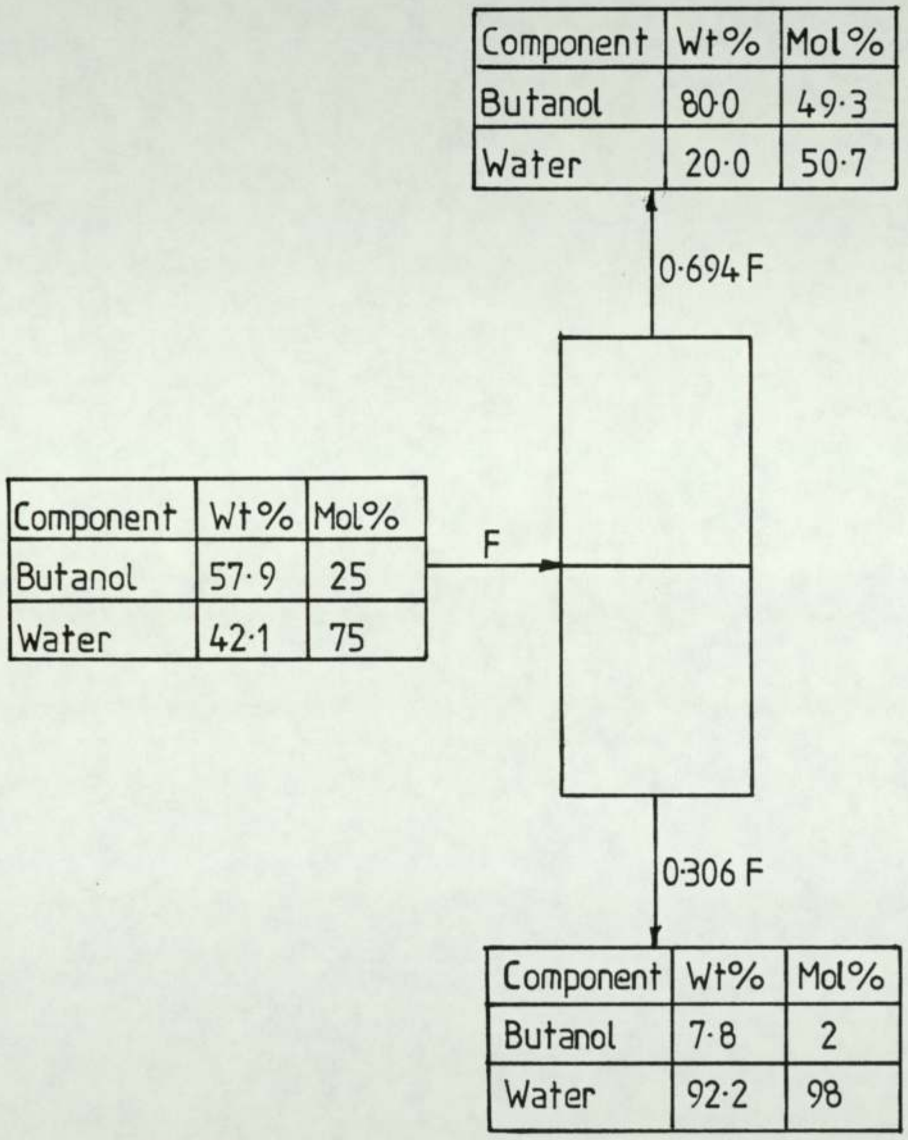
The bottom layer which contains mainly water and usually termed aqueous distillate is removed from the system, whilst the solvent layer commonly known as wet solvent, is returned to the reactor. From Figure 1.3 it can be seen that the wet solvent contains 20% water, which is an undesirable level since it is being returned to the reactor. Also, the rate of separation of the liquids is rather slow so an entraining agent, in this case Xylol, has normally been used in the manufacturing process both to reduce the water content of the wet solvent and to speed up the physical separation process of the two liquid phases. Xylol is a commercial mixture of the three isomers of xylene, whose structures are shown in Table 1.2.

With this third component present in the system, a different equilibrium between the solvent and aqueous phases is achieved, as shown in Figure 1.4. However, xylol is not detected in the aqueous distillate at separation temperatures and so it is continually re-circulating. Thus only one initial charge is required, the xylol content in the reactor remaining essentially constant.

1.3 Manufacturing Procedure

For type A, B and C resins the current manufacturing process

Figure 1·3 Equilibrium Separation of the Butanol/Water System



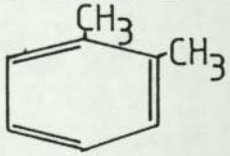
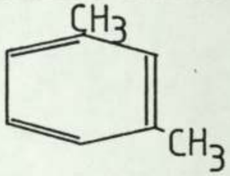
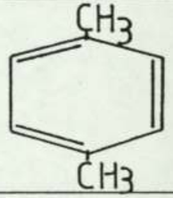
Component	Wt%	Mol%
Butanol	80·0	49·3
Water	20·0	50·7

Component	Wt%	Mol%
Butanol	57·9	25
Water	42·1	75

Component	Wt%	Mol%
Butanol	7·8	2
Water	92·2	98

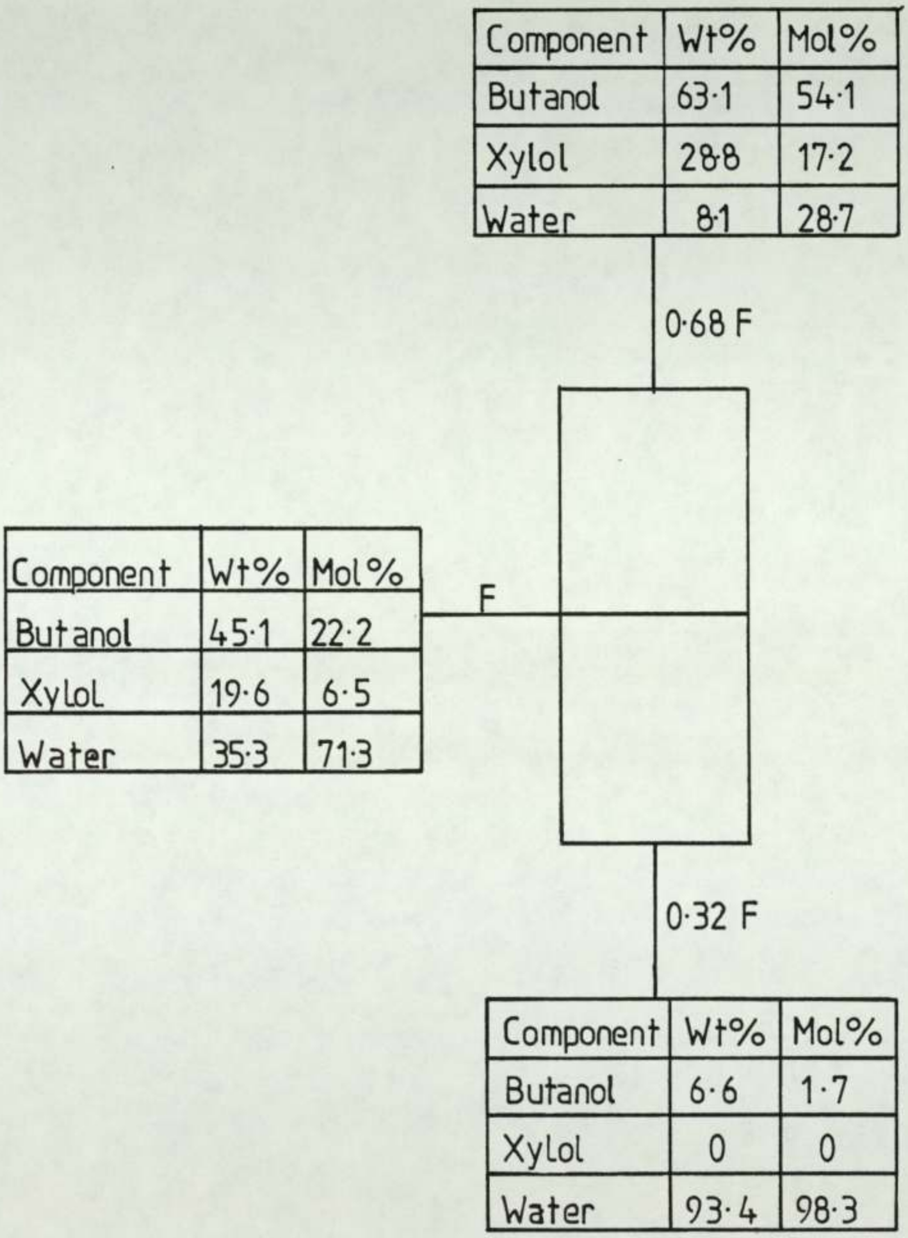
F = mass flow rate at 20°C

TABLE 1.2 XYLOL CONSTITUENTS

Compound	IUPAC$^+$ Name	Chemical Structure	Molecular Weight
O-xylene	1,2-dimethyl benzene		106·16
m-xylene	1,3-dimethyl benzene		106·16
p-xylene	1,4-dimethyl benzene		106·16

+ International Union of Physical and Applied Chemistry

Figure 1·4 Equilibrium Separation of the Butanol/Xylol/Water System



Component	Wt%	Mol%
Butanol	63·1	54·1
Xylol	28·8	17·2
Water	8·1	28·7

Component	Wt%	Mol%
Butanol	45·1	22·2
Xylol	19·6	6·5
Water	35·3	71·3

Component	Wt%	Mol%
Butanol	6·6	1·7
Xylol	0	0
Water	93·4	98·3

F = mass flow rate at 20°C

for raw materials through to saleable product, can be divided into seven distinct phases of operation, each having its own method of control. The cycle time for the resins under investigation can be up to 24 hours. The following describes each stage and their appropriate control methods :-

1.3.1 Initial Charging

Initially dry n-butanol and 44% formalin are volumetrically charged to the reaction vessel, via an automated solvent distribution system and the mixture agitated. At this point recovered butanol containing water, formaldehyde and butanol may also be charged.

1.3.2 pH Adjustment

A sample of the reactor contents is withdrawn and tested to determine its pH. If it is outside the specified pH range then 32% w/w sodium hydroxide is added to the charge to bring the contents within the range.

1.3.3 Alkaline Boil

The melamine is now charged by weight, manually, to the reactor and when this is completed, full steam is applied to the vessel's coils. The mixture is brought to the boil gently and refluxed for a fixed period, with all the distillate being returned to the reactor. It is during this stage that the addition reaction, methylolation, occurs between the melamine and formaldehyde, as described in Section 1.2.1. The control in this stage is solely the fixed time period.

1.3.4 Dehydration

At the start of this stage Xylol is volumetrically charged to the reactor and the distillate directed to the separator. This begins the water removal stage. The water which has entered the reactor with the formalin and recovered butanol if used, is distilled out of the system. As this process proceeds the synthetic resin is formed by the condensation and butylation reactions. The water of reaction produced is also removed during this stage.

During the dehydration stage the reactant temperature is used as the key control parameter and the water removed is assumed to be complete when the reaction mixture reaches 102°C. On achieving this temperature the heating to the reactor is reduced, before commencing the next stage. Also three hours from the start of the dehydration stage, a further charge of butanol, normally termed butanol(2) is made. This charge is made then because water removal has reduced the total volume in the reactor sufficiently to allow it to be made.

1.3.5 Acid Reaction

At this stage the appropriate type and quantity of acid is charged to the reactor in order to further catalyse the butylation reaction. The heating is then increased and the water removal process continued, in order to progress the butylation reaction. This water removal can be further enhanced by the operation of a distillation column on the reactor to reduce the water content of the returning wet solvent. During this stage both the condensation and butylation reactions are occurring. The reactions are controlled by testing resin samples hourly for both viscosity and solvent tolerance, which measures the degree of butylation. These measurements are adjusted to be within certain limits by the addition or removal of butanol from the reactor.

In the resins which use recovered solvents the initial quantity of butanol charged to the reactor is often not known precisely, due to the variability in the water content of the recovered solvents. By controlling the resin viscosity during the acid reaction stage a limited safeguard against the production of an irreversibly over condensed resin is introduced. This is needed especially when the butanol could be undercharged due to the uncertainty in the recovered solvents composition.

On attaining a value for the solvent tolerance which is within specified limits for the specific resin, cooling is applied to the reactor. This marks the end of the chemical reactions and the beginning of the final adjustment and processing.

1.3.6 Vacuum Stripping

This stage is used to adjust the viscosity of the resin so that it falls within the specified limits. The operation is carried out by applying vacuum to the reactor and evaporating the solvent from the resin until the viscosity and density are within the desired limits.

1.3.7 Filtering, Blending and Packaging

The resin is required to be filtered to remove any gelled particles and insoluble material. This operation is continued until a clear sample of resin is obtained. The product resin is then pumped to a blender where, if necessary, further solvent additions can be made or another batch blended in. From here, the resin is either drummed off or transferred to road tankers for shipping to the customer.

1.4 Production Equipment

The production facility under investigation by the Teaching Company consists of four, identical ten thousand litre stainless steel reactors, shown in Figure 1.5, fitted with jackets and coils for heating or cooling. Variations do exist in the overhead equipment with some reactors only having a vapour riser, condenser and separator, whilst others have distillation columns to fractionate the returning wet solvent from the separator to reduce the amount of water returned to the reactor.

One reactor, 12, is fully fitted with electronic instrumentation thus making it suitable for interfacing to a computer for control purposes and also as a test site for new sensing instruments.

This reactor was used for the practical work reported in this thesis and is illustrated in Figure 1.6. The reactor has a distillation column associated with it which is used to contact directly the rising vapour from the reactor with the wet solvent returning from the separator. This operation is effectively a batch distillation, the reactor acting as the reboiler. The distillate is condensed and flows into the separator, where two liquid layers form. The wet solvent layer overflows from the separator to the buffer tank and is either removed from the system or returned to the reactor directly or via the column. When returned to the column the wet solvent flowrate is controlled by the column top temperature which is set to maintain the azeotropic composition. During the

Figure 1·5 Coating Resin Plant-Existing Plant and Instrumentation

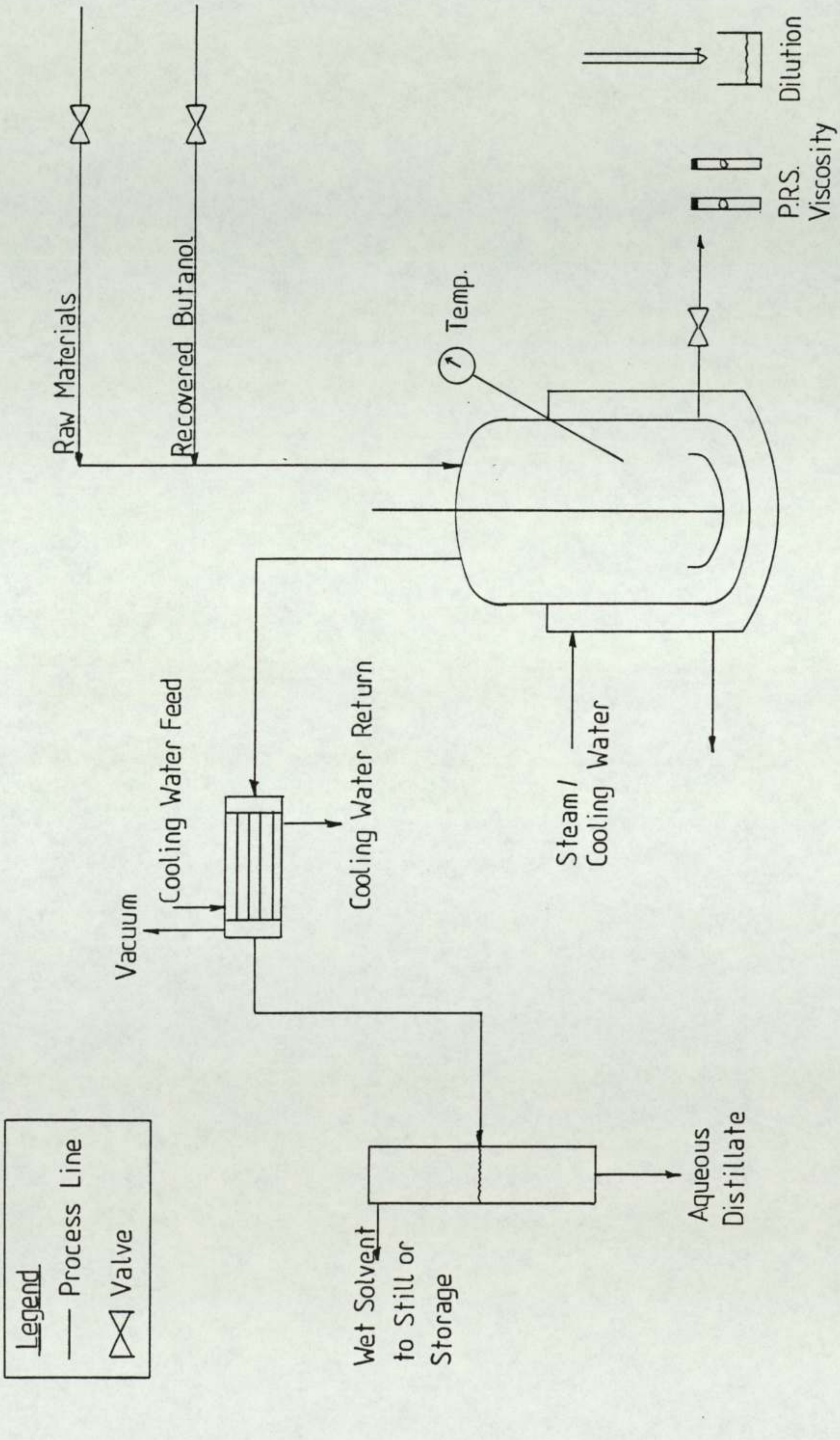
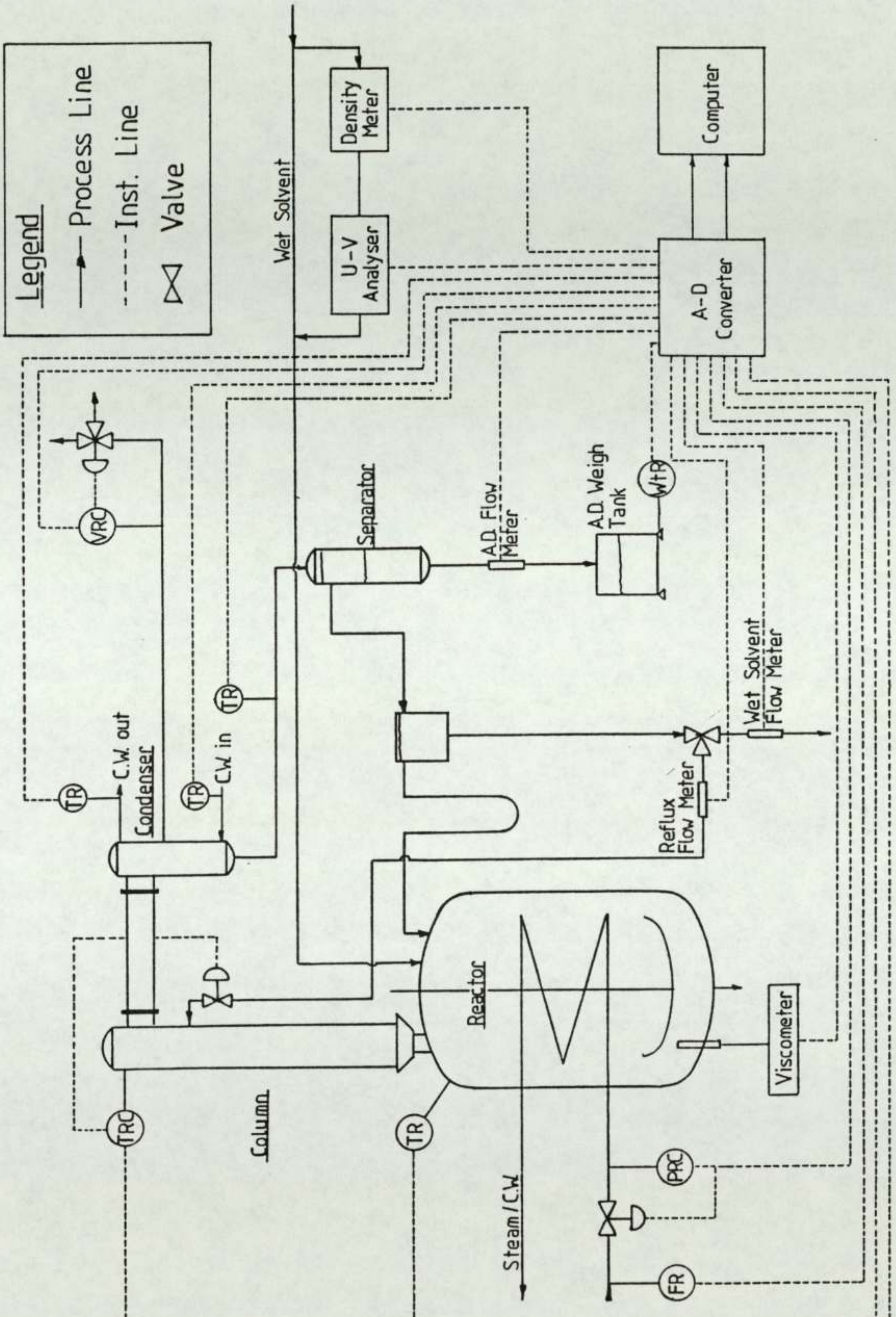


Figure 1·6 Computer Installation on Test Reactor



acid reaction stage there is sufficient wet solvent available for reflux to maintain this condition, so at this point the control is switched to buffer tank level control and all the wet solvent is returned as reflux. The temperature is allowed to increase.

The aqueous distillate layer in the separator is pumped away at a flowrate controlled by a level controller determining the interface level in the separator. It is pumped and metered either directly to a storage tank or via an intermediate weigh tank.

1.5 Discussion

The process described above can be carried out successfully only if appropriate corrective action is taken when the measurements made show a divergence from the intended values. Some of these measurements require at present manual intervention and delays. The task of this thesis is to describe the way in which computer control methods have been developed and applied to Reactor 12. This has also involved the assessment of automatic means of analysis of the previously manually controlled variables. In some cases, it has been possible to implement online measurements. Work has also been done to measure other variables not previously monitored. This section outlines these main activities.

The literature reviewed covering the process chemistry has shown that water is produced during the condensation and butylation reactions. By stoichiometry the quantity produced at each stage is an indication of the extent of the forward reaction. This has been discussed in detail by V.H. Patel(21). In the commercial process, water is also introduced into the system from two raw material sources, the formaldehyde solution and the recovered solvents. It has already been shown by Freeman(4) that monitoring the mass of aqueous distillate removed from the reactant mixture can be used to indicate the process end points. In order to determine accurately these end points a method for determining the variable water content of the recovered solvent needs to be identified. The formaldehyde solution is tightly controlled and monitored and does not require to be analysed in the same manner. This work is fully reported in Chapter 3.

It is an important requirement to have a suitable computer system. This can be considered in two parts, the software which is needed and appropriate computer hardware and interfaces. The software was developed in a modular form to allow the development and implementation of individual procedures. The computer hardware required must run the programs and have inputs and outputs to several peripheral devices such as printers and VDU's, as well as to the plant controller and instruments. It must also be expandable to allow for the development of the control strategy and provide the operator with an easily useable and understandable interface to the computer and the process. The development work is reported in Chapter 2 with the final development and results from control trials reported in Chapter 4.

2. COMPUTER SYSTEM: DESCRIPTION AND INSTALLATION

2.1 Introduction

The most important factors which must be taken into account for the introduction of computer control to a process, are the constraints imposed by the current plant equipment and instrumentation and by the involvement of the operators in the resin processing. In order to work within these constraints the work presented in this chapter sets out the system requirements, along with the philosophy adopted to bring about computer control, both in relation to the production equipment and the operator's role in the process.

2.2 Computer System

The production equipment and instrumentation in this study was Reactor 12 of the resin plant. The major equipment requirement for this project was therefore the purchase of a computer system which would fulfill the developing needs throughout the project together with the necessary interfaces. The general needs of such a system are outlined as follows:-

(i) The ability to take in signals representing the measured values from the process instrumentation; carry out calculations on this information in real time and return appropriate control signals to the plant within the control band of time.

(ii) The need to support multi-tasking, to allow several control and monitoring procedures to be operated simultaneously.

(iii) The capacity to support more than one terminal operating simultaneously. This facility was achieved using the operating system installed on the computer.

(iv) The need to be flexible in the type of input and output devices installed on the computer, especially for receiving measurements and sending out control signals.

(v) The need for sufficient computer memory so that enough is available for the initial requirements, with the option to add to this as the software is developed.

The system which was selected to meet these requirements was a SEED System 19. This computer was based around a Motorola 6809 microprocessor and was supplied with the OS-9 operating system. This is a UNIX type operating system supporting multi-user, multi-tasking operations. The basic computer unit comprised of dual 5¼" disc drives, 256K of memory as standard, four input/output cards to allow connection to devices such as terminals and printers and a 16 - channel multi-plexed Analogue - Digital Converter, ADC, to receive signals from plant instrumentation. In order to provide an operator interface a terminal was required. For this a BBC microcomputer Model B was purchased, based on a 6502 microprocessor with a Microvitec Cub monitor. This computer was modified to become a terminal by the installation of a PLC Emulator Read Only Memory, ROM, chip in one of the spare ROM sockets on the computers circuit board. Finally, an Epsom FX - 80 F/T printer was purchased to allow not only for the production of software listings during the development stages, but also for the generation of log-sheets during the plant trials. Other hardware acquisitions were made during the course of the work which included a second BBC Model 'B' computer and a 6502 second processor for one of the BBC's to provide increased memory for graphics applications. Also needed were extra memory for the SEED and digital output and input devices. These are described in more detail in the appropriate sections of this chapter and Chapter 4.

2.2.1 Microware Systems Corporation OS-9 Operating System(17)

The operating system, OS-9, used in the SEED 19 computer is extremely flexible. The operating system and each program running is considered as a task, allocated a section of memory and a priority for access to the CPU, Central Processor Unit. Thus control tasks requiring action in real time can be given priority to ensure their completion. Each task can have access to the input and output devices when required, with the operating system resolving any conflicting requirements.

The basic philosophy for input and output used in this operating system is that all data transfers to Input/Output, I/O, devices are performed in virtually the same manner despite the differences in the hardware devices involved. This is achieved by the definition of a set of standardised logical functions for all types of peripheral device. All I/O devices conform to those conventions, through the use of software routines to eliminate hardware dependencies whenever possible, thus producing a simple and versatile input/output system. This system is termed a Unified Input/Output System and is based upon logical entities called I/O paths which are used to route data from a program to mass storage files or other I/O devices. The data which is transferred through these paths will be processed automatically by OS-9 to conform to the hardware requirements of the I/O device selected. These data transfers can take one of two forms, firstly, bidirectional, read or write, or secondly undirectional, read only or write only. This depends on the device type and on the way in which the path was constructed. The data is transferred as a stream of 8-bit bytes that have no type or value associated with them and is only meaningful to the programs using it.

Each of the input/output devices supported by the system requires to have a unique name. These names actually refer to the device descriptor modules stored by OS-9 in an internal data structure called the module directory, which is set-up automatically during the start-up sequence. The device names which can be used are arbitrary but for common peripherals the following standard names are commonly used and were used in the programming work for this thesis :-

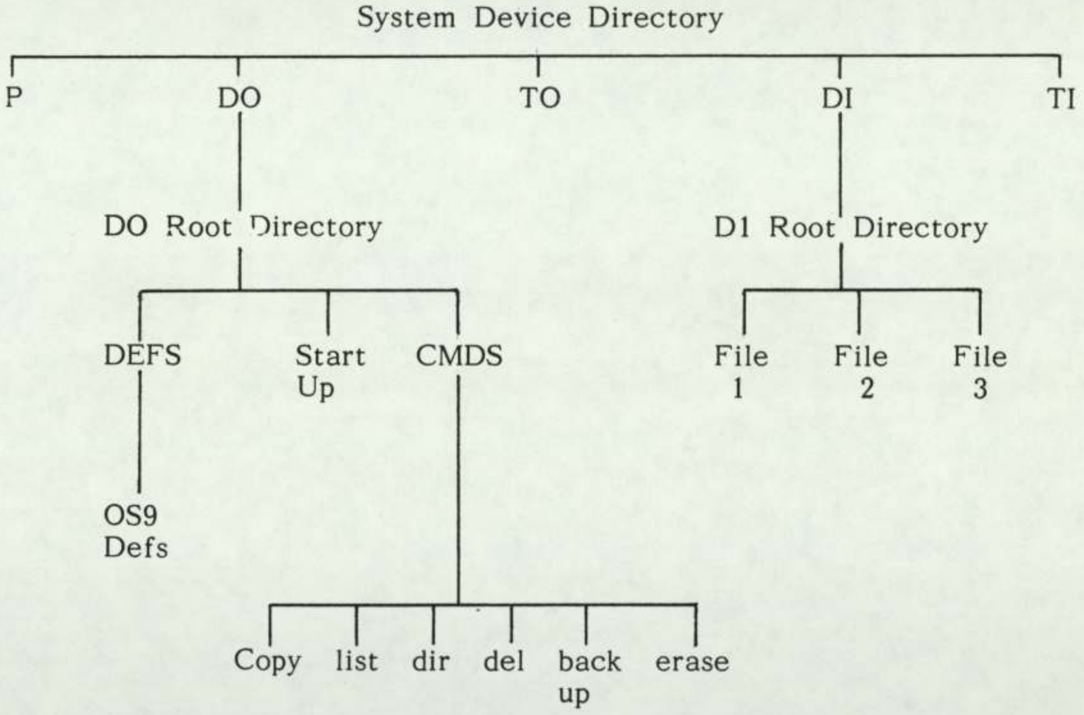
T.0 - Primary system terminal
T1,T2 - Other serial terminals
P - Parallel printer
Ps - Serial printer
D0 - Disc drive unit zero
DI - Disc drive unit one

Other device descriptors were used in the programming work and they will be mentioned in the appropriate section.

2.2.2 Multifile Devices and Directory Files

The multifile devices employed by this system are two disc drives using 5¼" floppy discs. The data which is being stored on the device is organised into logical entities termed "files". In order to understand the relationship between the directory files and data files it is easier to refer initially to Figure 2.1.

Fig. 2.1. Multifile Device Storage Structure



From Fig 2.1 it can be seen that the directories are organised in the form of a tree-structured hierarchy. The highest level directory is the "system device directory", which contains the names and linkages to all the Input and Output (I/O) devices for a given system configuration. In the case of multifile devices, here represented by DO and D1 the next level in this hierarchy structure is the master directory, termed root directory which contains the names of the files and sub-directories stored on the device. The root directory is created automatically when the disc is initialised during its formatting routine.

When OS-9 is requested to establish an I/O path to a file stored on one of the disc drives, it uses the names given in a pathlist sequentially from left to right to search various directories and obtain the necessary routing information. As an example of the formation of a pathlist referring to the file list, the following is used :-

```
DO/CMDS/list
```

It should be noted that it is customary practice that the directory names are given in capitals whilst ordinary file names are not. This allows the easy identification of directory files on file lists.

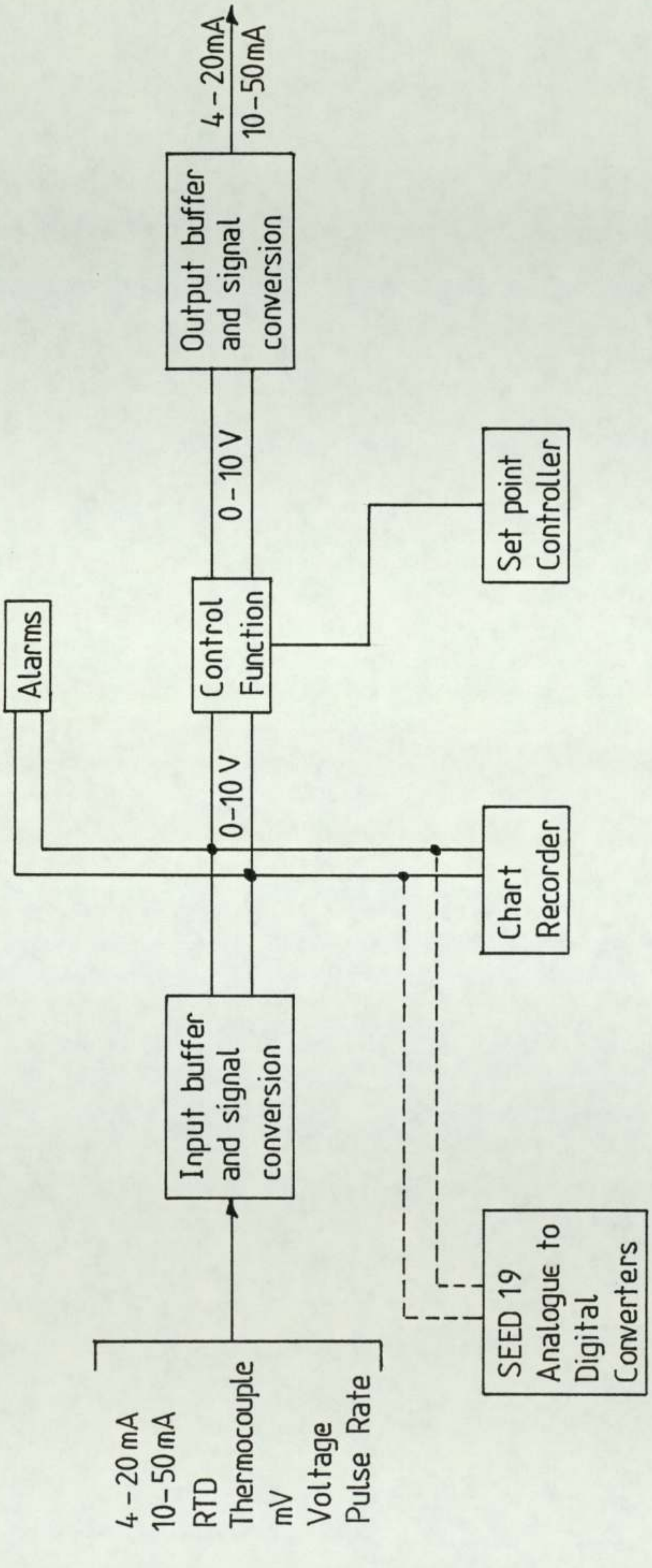
2.3 Installation of the System

The first step in installing the above system on the Butylated Resins Plant was to determine the physical locations for the computer hardware, with special precautions being made because of the hazardous nature of the plant environment. It was decided to locate the SEED-19 computer in the plant control room. This minimised the cabling requirements between the reactors' instruments, which are also housed in this room, and the sixteen channels of the A-D converter on the computer. Due to the lack of space in this room the terminal and printer had to be located in a separate room in a safe area, which was approximately 20m away. The two sites were connected by 3 serial channels conforming to standard RS423. To allow for verbal communication between operators in the terminal room and the control room, an intercom system was installed.

2.3.1 Current Plant and Instrumentation

The current plant and instrumentation has been previously illustrated by Fig 1.6. This diagram depicts one of the reaction vessels within the butylated resins department at BIP Chemicals Limited, these vessels are commonly referred to as Stills. The particular vessel illustrated in the diagram has electronic control and instrumentation which makes it the most suitable for interfacing to a computer. The instrumentation belongs to the Foxboro Spec 200(22) family and a basic arrangement for these instruments is given by Figure 2.2.

Figure 2.2 Basic Arrangments of SPEC 200 Instrumentation



The instruments installed on the production plant were of the following types :-

1) Temperature Probes - Degusa 100 Platinum Resistance Thermometers

2) Pressure Transmitters - Foxboro, Differential Pressure Cells

3) Flowmeters - Rhodes, Semi Positive Displacement Meters

4) Steam Flowmeter - Orifice Plate with Foxboro Differential Pressure Cells

5) Vacuum - Foxboro Differential Pressure Cells

6) Aqueous Distillate Tank Weight - Darenth, 4 point stood lever system with a pneumatic weigh head transmitter.

From the diagram, Figure 2.2, it can be seen that the outputs from these instruments pass into signal converters before being displayed on chart recorders, in this case Chessell recorders, or digitally displayed in the case of the Aqueous Distillate Weight. The instruments and the ranges in which they display are shown in Table 2.1. For outputs see Table 4.2., Section 4.3.

Table 2.1 Reactor Instrumentation and Ranges

Instrument	Range	Units
Coil/Jacket Pressure	0-4	bar
Steam Flow	0-1400	kg/hr
Aqueous Dist. Flow	0-1500	l/hr
Wet Solvent Flow	0-3000	l/hr
Reflux Flow	0-3000	l/hr
Still Temp	30-130	^oC
Column Top Temp	55-105	^oC
Cooling Water Feed	0-50	^oC
Cooling Water Return	0-50	^oC
Distillate Temp.	0-50	^oC
Vacuum	0-1000	mmHg
Aqueous Distillate Weight	0-4000	kg

2.3 Interfacing of the SEED 19 to the Plant Instrumentation

In order to physically connect the computers analogue to digital converter to the plant instrumentation, twenty, twin core, shielded cables were laid between the instrument cabinet and the computer a distance of approximately 3m. The cables were passed into a junction box where they were connected to two sixteen core cables leading to the computer, where the appropriate cable cores were attached to selected pin numbers on both of the 25 pin D type female connectors mounted on the ADC, as in Appendix 2.

The calibration of the analogue, O-10V signals being fed to the computers converter was conducted in the following manner. A high accuracy generating unit was placed across the cable cores corresponding to a particular instrument. A voltage was generated in the circuit corresponding to a particular temperature, pressure, flow or mass for that particular instrument. The digital value registered by the computer for the voltage generated was sampled by the system ten times and then averaged. A print out of the engineering value against the digital value was obtained and then another voltage was generated and the process repeated until the range of the instrument had been completed. From this data calibration curves were drawn for each instrument and mathematical relationships obtained by the use of a linear regression program. The calibration curves are given in Appendix 2, whilst the mathematical relationships are given in Table 2.2.

Table 2.2 Relationship between Digital and Engineering Values (Straight Line of the form E = MD + C)

Instrument	m	C
Still Temp	$2{\cdot}452X10^{-2}$	+30
Cooling Water Feed	$1{\cdot}226X10^{-2}$	-
Cooling Water Return	$1{\cdot}226X10^{-2}$	-
Column Top Temp	$1{\cdot}226X10^{-2}$	+ 55
Distillate Temp	$1{\cdot}226X10^{-2}$	-
Steam Flow	0·367	-
Aqueous Dist. Flow	0·37121	-
Wet Solvent Flow	0·7424	-
Reflux Flow	0·7424	-
Vacuum	0·2466	-
Coil/Jacket Pressure	$9{\cdot}997X10^{-4}$	-
Aqueous Distillate Weight	2·004	-

The relationship given in Table 2.2 take the form, E=mD+C where D represents the digital value and E is the corresponding engineering value for any of the instruments, given the constants m and C, See Appendix 2.

It should be noted that the input voltage, to the computer for the Aqueous Distillate Weight was in the range 0-5V and this value has been converted from a pneumatic to an electrical signal prior to being converted to a digital value. This caused some errors in the values obtained from this instrument. Because of the narrower range, any small variations in the pneumatic signal, which have an effect on the converted voltage being fed to the computer, will be attenuated by a factor of two in the converted engineering value compared to similar 0-10 Volts signal on another instrument. In effect the value for the aqueous distillate weight is more sensitive to minor fluctuations in the voltage signals. The effect of this on the system and data integrity is discussed in Chapters 4 and 5.

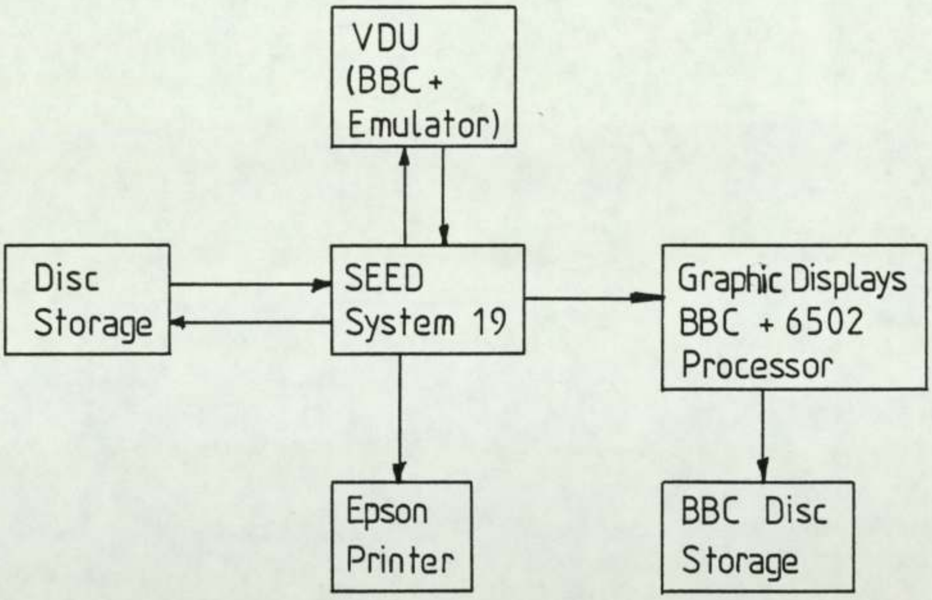
During these calibration procedures the absolute accuracy of the instrument was not as important as its repeatability over the desired range. Any deviations in the absolute value, provided they are consistent can be allowed for during the calibration procedure, or the computer software can be structured to allow for this.

With the limited number of input and output channels available on the SEED 19 computer and the lack of valve actuators with proximity switches on the test reactor, control actions required to be carried out and checked manually.

2.4 Software and Hardware Structure

The software for this system was written in two languages, Basic 09 for the SEED 19 system and BBC Basic used by the BBC Acorn computer. The reason for the use of these two languages is that the SEED 19 does not support its own graphics routines and the graphical display was therefore taken over by the BBC computer. The BBC computer installed for this purpose comprised of a BBC Model B microcomputer, a Microvitec Cub High Resolution Monitor, a Cumana dual disc drive and an Acorn 6502 second processor, which would allow much larger graphics procedures to be written for the BBC and also increase their speed of operation. The system configuration is best illustrated by Figure 2.3. The communication between the SEED 19 and the graphics BBC was arranged by a serial line at RS423 standard using ASCII, American Standard Code for Information Interchange Code.

Figure 2.3 System Configuration



2.4.1 Man-Computer-Process Interaction

To begin writing the software for the first stage of the computer control experiments an intitial general philosophy for the operators role in the process was defined. Certain aspects of the process cannot easily be automated, such as the process sampling and certain material additions. These were to be prompted by the computer system for the operator to take the necessary action. With the computer system at the centre, figure 2.4 illustrates the role of the operator in the control of the manufacturing process, for the initial stages of this work, where the computer acts as an aid to control, while the control strategy was being developed and refined.

2.4.2 Software Structure

In order to be able to develop the software from computer aided control through to full computer control the procedures were written in a modular form. Essentially two packages were written, firstly the data logging routines to generate graphics displays and logsheets, and secondly a package containing the monitoring and control procedures which were used to prompt the operator for actions to be taken. The interaction between the procedures for each package are shown by Figure 2.5 and Figure 2.6. The names which are given in these figures refer to the various procedures or files which are used.

The advantages of this approach in writing the software are that each individual procedure was written, tested and

Figure 2·4 Man-Computer - Process Interaction

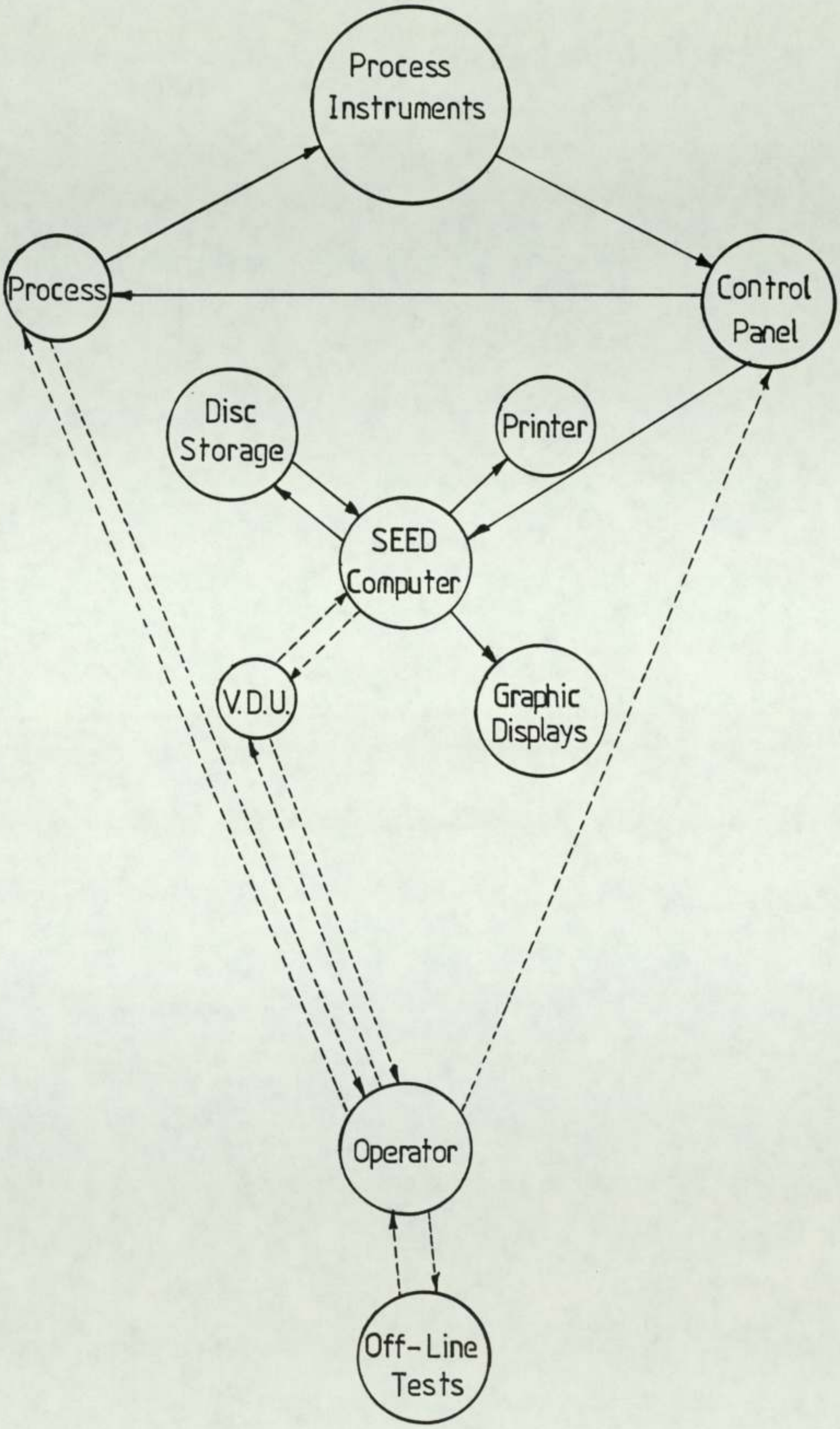


Figure 2·5 Procedure/Flag File Interaction for Logging and Display Package

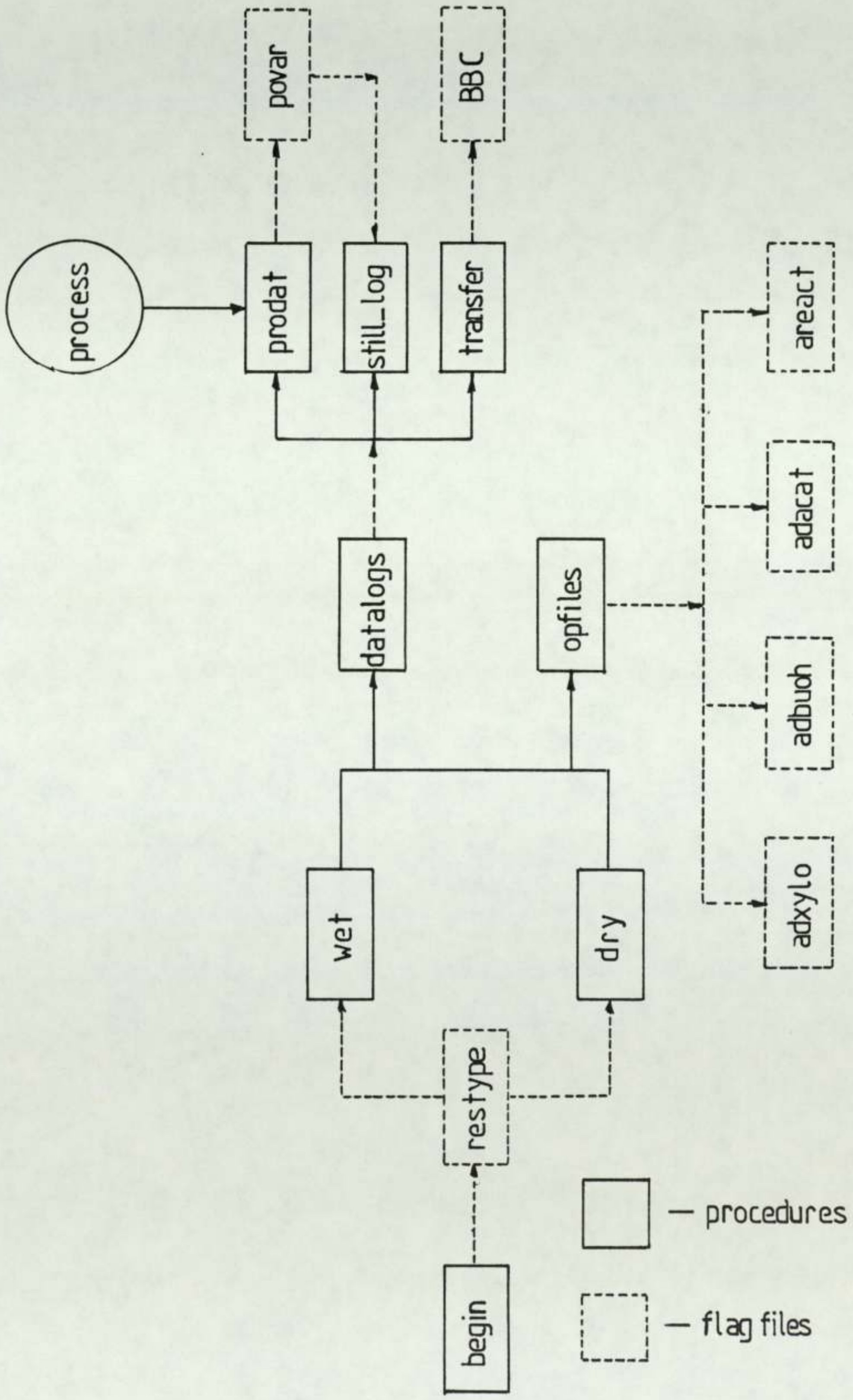
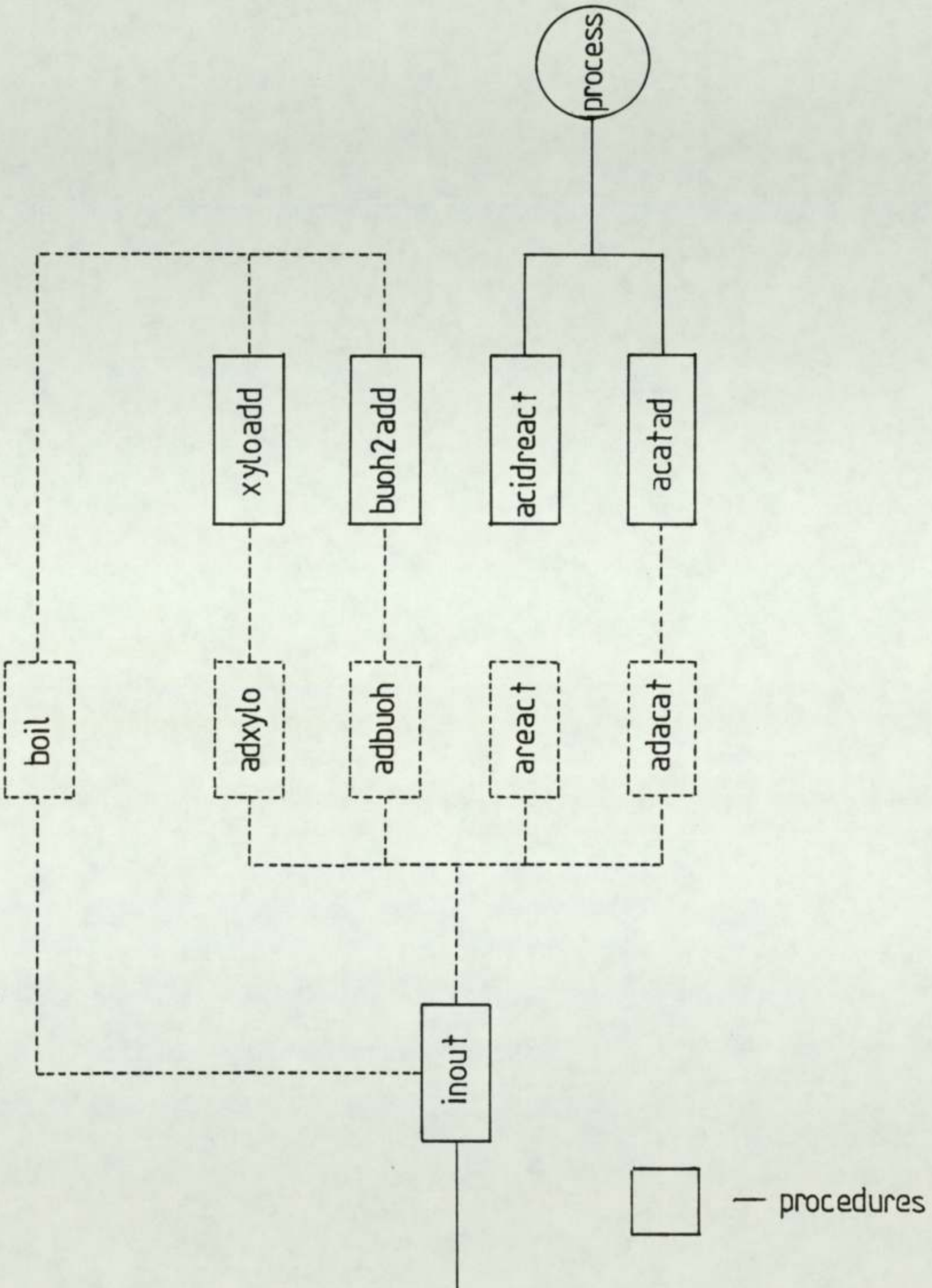


Figure 2·6 Procedure/Flag File Interaction for Monitoring and Control Package



de-bugged before being linked into the system. This made it much easier to detect faults in each procedure, compared with finding an error in a large complex program. Another advantage is that as well as being used individually the procedures could be re-organised to allow for different trials and tests to be carried out, especially where data gathering was required to confirm or refine a particular piece of the control strategy. The procedures were flag driven. This means that a data file was periodically read by a particular procedure to check the current status. When a value was detected that required action by that procedure it carried this out before proceeding onto its next operation. For example the procedure xyloadd monitors the flag file boil. When the value in this file has changed from O to 1 the procedure starts a timing routine which when complete indicates that Xylol should be added. This is done by the procedure adjusting the value in the file adxylo.

2.5 Software

As mentioned previously the software has been written in a modular form and split into two distinct packages as illustrated by Figures 2. 5 and 2.6. The following briefly describes the purpose and operation of the various procedures illustrated on the above mentioned figures. Appendix 3 gives the Flowsheets and Listings for these procedures.

2.5.1 Procedure **'begin'** creates and initialises the flag files which are used throughout the production run by the other procedures. The second function of this program is to request the selection of the resin to be manufactured, after receiving the resin type the standard formulations are displayed for wet or dry batches and a further choice is required, as to which type of butanol charge is to be used. Based on the selections made, several operational data files are created containing the relevant charge information for use by subsequent programs.

2.5.2 Procedure **'wet'** uses the charge composition data files created by **'begin'**, displaying the material quantities in the order in which they should be charged and requesting a positive acknowledgement of charging by requiring that the actual material quantity be entered via the keyboard. This procedure also needs the composition of the wet solvent and the water content of the formalin to be entered, to allow for the calculation of the adjusted butanol (2) and xylol charges, as well as calculating the amount of water which will be removed during both the dehydration and acid reaction stages. After assembling the relevant data, further operational files are created containing the actual charges made, the adjusted secondary charges and the water to be removed.

2.5.3 Procedure **'dry'** is similar in function to 'wet' but does not require any data input relating to the wet solvent composition. It does still however require the formalin's water content to be entered for a similar purpose as **'wet'**. Similar files are also created for use by the monitoring and control procedures. Both **'wet'** and **'dry'** before termination start the execution of the procedures **'datalogs'** and **'opfiles'** in the background mode.

2.5.4 Procedure **'datalogs'** initiates the execution of the data logging procedures **'prodat'**, **'still-log'** and **'transfer'** which generate data for the disc files, logsheet and graphics display respectively.

2.5.5 Procedure **'opfiles'** initialises the flag files which are to be used by the monitoring procedures to communicate with the operator prompting procedure.

2.5.6 Procedure **'prodat'** accesses the analogue to digital converter to obtain the digital values of the 0-10V signal outputs from the Spec 200. These values are then converted to engineering values using the appropriate calibration equations. This data is then down-loaded to disc files for use by the logsheet generation routine. The procedure operates by using ten blocks of stored data, on individual files, updated every minute and overwriting the files in the set every ten minutes.

2.5.7 Procedure **'still-log'** produces a logsheet of the process values at ten minute intervals throughout the production cycle, using the data stored by **'prodat'**. It also accesses the flag files used by the controllers, detecting and logging any changes made to the controller set points. At the end of the production cycle a table is produced giving all the raw materials used both recovered and pure.

2.5.8 Procedure **'transfer'** is used to acquire data from the process instrumentation for use by the graphics package on the **BBC Computer.** The digital data is acquired from the A to D converter and then converted to engineering units before being broken into **ASCII** characters for transmission to the **BBC,** this data is updated and transferred every ten seconds.

2.5.9 Procedure **'inout'** provides the interface between the operator/terminal and the monitoring and control routines operating in the background mode. The operation of this procedure relies on the changing of flag values by the monitoring procedures running in the background. Whenever a change in value is detected the appropriate message is generated and a suitable response to this prompt is requested, either the entering of a raw material quantity charged or an appropriate answer to the question. All the values entered are stored and used by the logsheet procedure to generate a hardcopy at the end of the cycle.

2.5.10 Procedure **'xyloadd'** is a timing procedure whose counter is triggered at the onset of boiling by accessing the appropriate flag file. The timer counts for a period of 15 minutes, during which time the methylolation reaction occurs, at the end of this period the value in the flag file **'adxylo'** is altered and the procedure terminates.

2.5.11 Procedure **'buoh2add'** is another timing routine whose function is to time the reaction for a period of three hours after the addition of the xylol. The value in the flag file **'adbuoh'** is then altered to indicate that the dehydration stage has progressed sufficiently for the addition of the second butanol charge. On completion of this, the procedure then terminates.

2.5.12 Procedure **'acatad'** monitors the still temperature and mass of aqueous distillate removed to determine the end point of the dehydration stage. At this point the value in the file **'adacat'** is altered to indicate that the acid catalyst should now be added. To obtain the end point more accurately a ten point rolling average is used to determine the aqueous distillate mass at any time, this is due to the fluctuations introduced into the system by the pneumatic converter and the sampling period of ten seconds.

2.5.13 Procedure **'acidreact'** monitors the aqueous distillate removed to detect the end point of the acid reaction stage and then bring about the completion of the reaction cycle by altering the value in the file **'areact'**. This procedure also uses rolling average for the aqueous distillate mass.

2.6 Graphics Displays

As previously explained the SEED 19 does not support its own graphics displays so this duty has been given to a BBC Model B microcomputer with a second processor and dual disc drive. Since the BBC computer is a single tasking system a single program has been written to provide the facility to receive data from the SEED 19 and display it in a graphical form. The program consists of several sub-routines which are either concerned with data reception or represent a particular form of display.

The key sub-routine is that which accepts the coded data in ASCII form from the SEED system through the RS423 serial port on the BBC.

The operation of this whole program is dependant on the information being received by this subroutine. The program will not proceed to plot the new data or change displays until the package of process values has been received from the SEED. Once this information has been received the program executes another routine where a decision is made to either produce a graphic display routine or to store the data on the local disc. It does this if ten minutes have elapsed since this last occurred, prior to executing the graphics routines. Once into the graphics routine there are two options, firstly the current display can be updated with the latest data or secondly, if a change of display has been requested then the display is redrawn in the new format with the latest information displayed and with negligible delay. The program then returns control to the data reception routine to await the next package of data. The time between data transmission is ten seconds which gives a mean waiting time for displays to be updated of five seconds. This is more than adequate for a system of this nature.

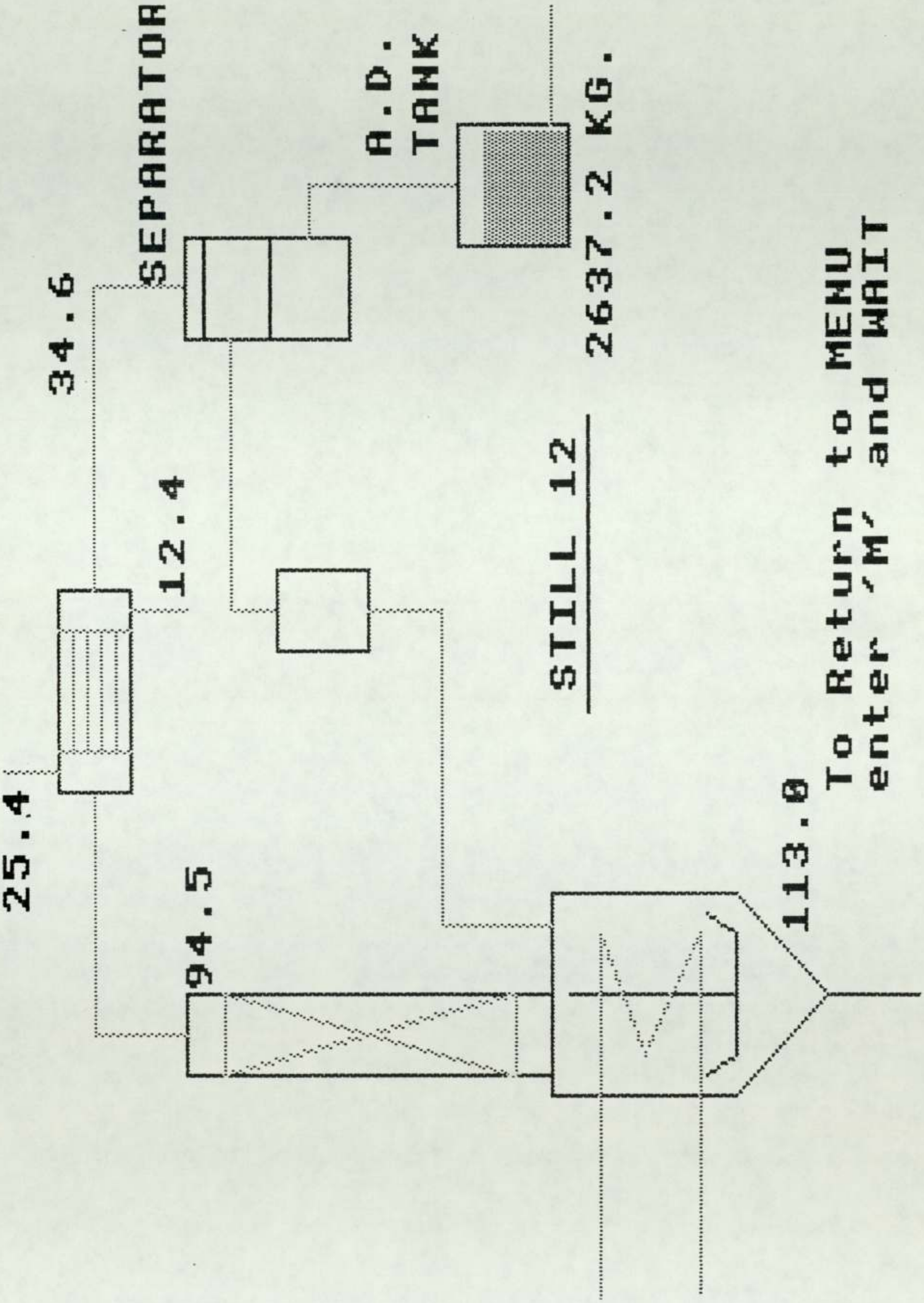
The actual graphics displays consist of the following. Firstly a menu selection display itemises the five basic displays available. Three of the displays give the trends of the key control parameters of Reaction Temperature, Aqueous Distillate Weight Off and Resin Viscosity. All are plotted against the reaction time. The two remaining displays contained various process values. One consisted of a series of bar graphics, whilst the main display was a mimic diagram of the production plant, with the process values displayed to one decimal place. These displays are illustrated by figures 2.7, 2.8, 2.9.

Limitations are placed on the colours which could be used in the high resolution mode graphic displays by the operating system of the BBC Model B and the available memory of 64K. The display is limited to modes where two colours are available, foreground and background, and a large proportion of the memory was required for the screen mapping. The colours chosen for those displays were black for the background and green for the foreground. These were selected because they produced a display which was clear and easily readable compared with the other colour combinations available on the BBC Model B microcomputer.

2.7 Conclusion

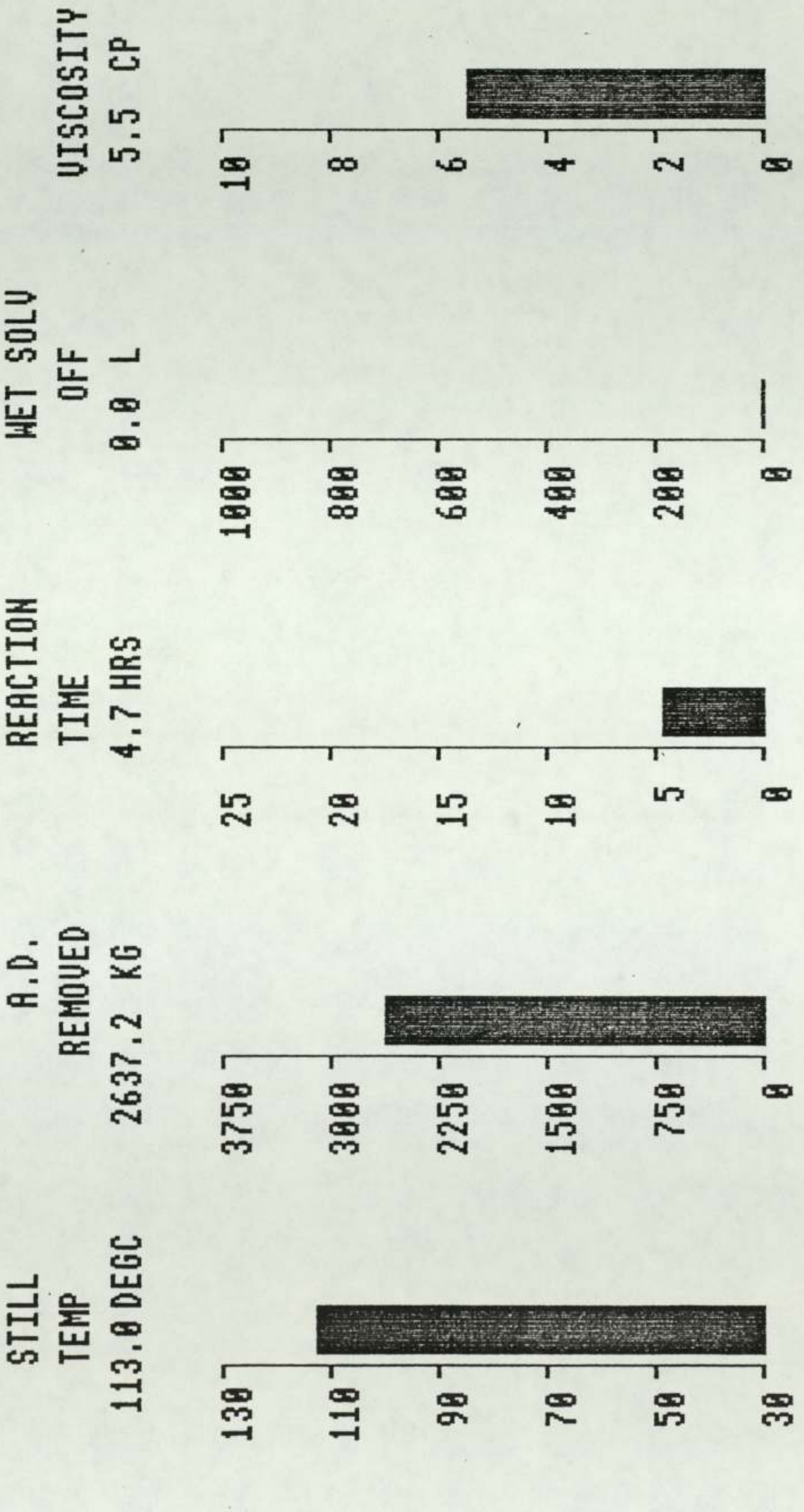
The system described here was installed on the production facility at this stage of development. It was configurated in such a way that it could be expanded. This allowed for the development and implementation of the control strategy over several stages, as well as the full testing of the software at each stage. The power of the SEED 19 system lies not only in the ability to expand its memory as required for more procedures, but also in its ability to interface with peripheral devices through the installation of additional cards such as Digital-Analogue Converters.

FIGURE 2.7 Mimic Diagram Display



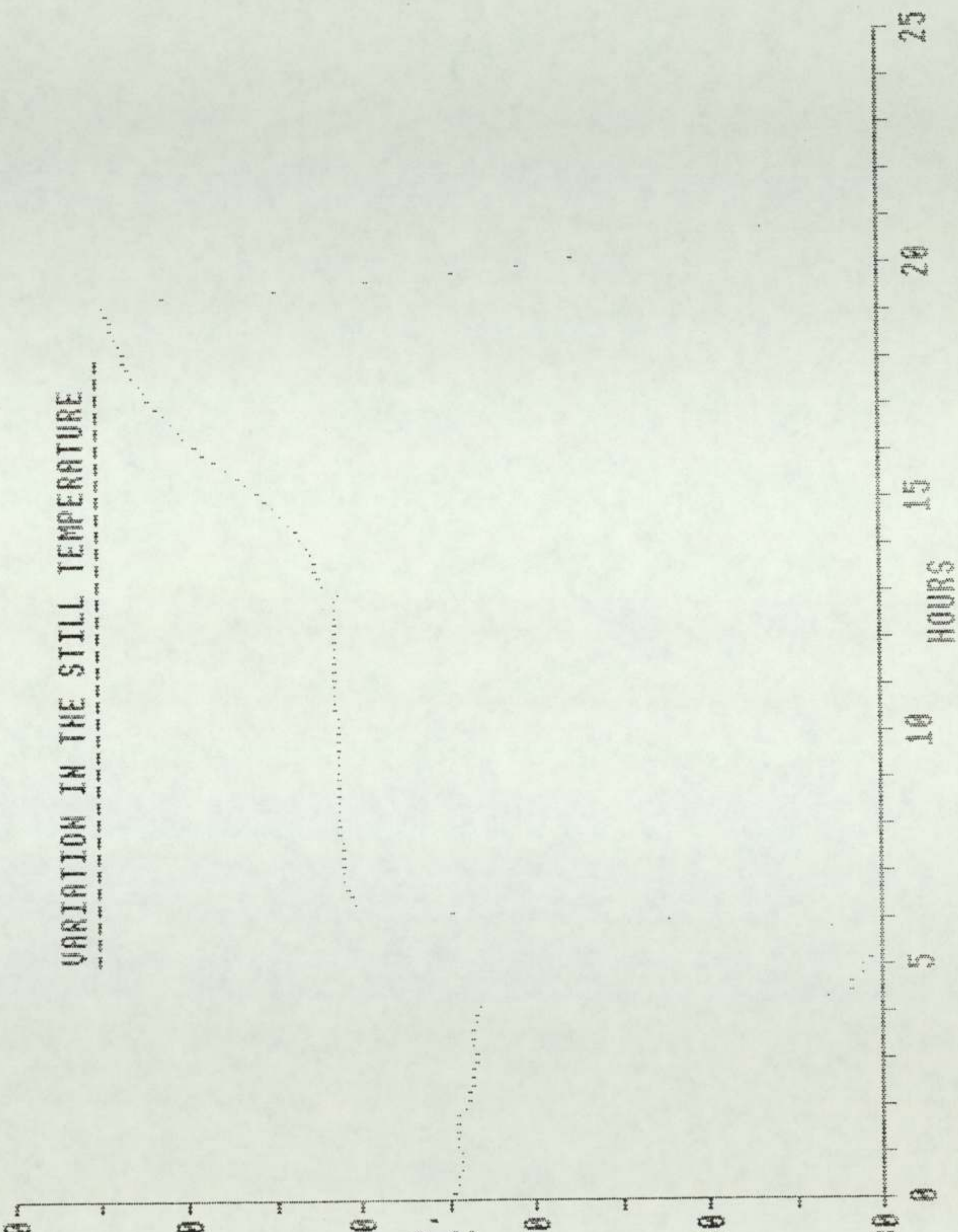
To Return to MENU
enter 'M' and WAIT

FIGURE 2.8 Bar Chart Display



To Return to MENU,enter 'M' and then WAIT

FIGURE 2.9 Typical Trend Display



The software, having been written in a modular form, takes full advantage of the multi-user, multi-tasking operating system, OS-9 and allows several procedures to be resident in memory and sharing the Central Processor Unit, CPU, time concurrently. It also allows for the writing and installation of additional procedures, where required for peripherals which have been added to the system or where further software operations are required. The graphics package on the BBC microcomputer provides a usable interface between the plant and the operator, using displays which are easily readable and understandable and supplying the process values which are known to be the key control parameters in a historical trend format.

Therefore the computer and its peripheral devices provided a basis for the development and implementation of a computer strategy on the full scale production facility within the limits imposed by the current plant hardware.

3. WET SOLVENT ANALYSIS AND PROCESS VISCOMETRY

3.1 Introduction

It was shown in Chapter 1 that the mass of aqueous distillate removed from the reactor is a suitable measurement for indicating the desired end points at various stages of the reaction. The use of this measurement is suitable for laboratory or pilot scale production where the total amount of water to be removed from the reactor system, comprising the formalin and that due to the reaction, is known. On the full scale production facility this is seldom the case due to the use of recovered solvents which contain variable amounts of water. These solvents come from two sources, the vacuum stripped solvents which are removed from the resin mixture at the end of the reaction to bring the resin into the appropriate specification range and the solvents recovered from the aqueous distillate which is removed continuously throughout the reaction. The aqueous distillate undergoes a series of distillation operations to increase the solvent concentration before it is mixed in with the vacuum stripped solvent for re-use.

The recovered solvents are multi-component mixtures of butanol, xylol and water with small amounts of formaldehyde also present. In order to bring about effective control of the process one or more measurements are required to determine this composition so that adjustments can be made by calculating the actual charges of butanol and xylol and obtaining an accurate value for the water which had been added. All this extra water has to be removed from the reactor in addition to the water of reaction. This chapter deals with the methods investigated to meet these requirements along with the outcome of the plant trials which were carried out to evaluate the methods selected.

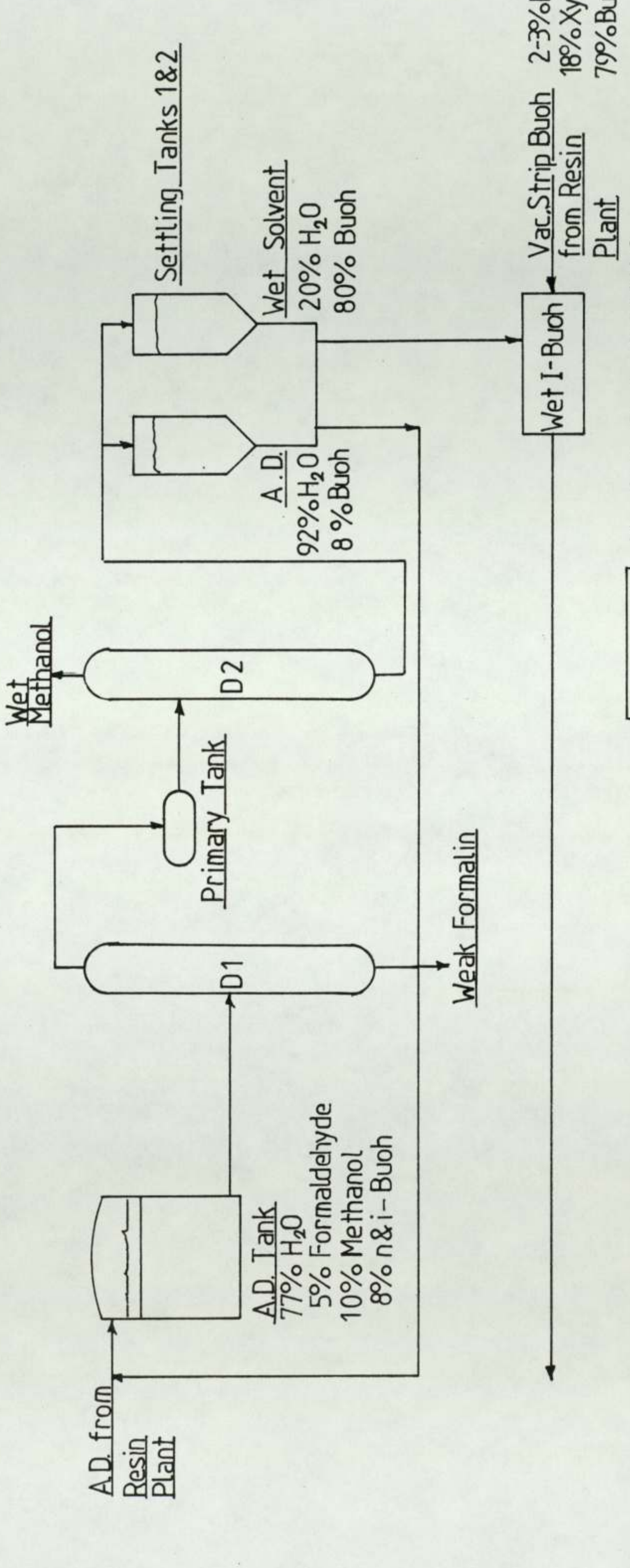
3.2 Solvent Recovery Operations

As previously described the recovered solvents are obtained from two sources. The vacuum stripped solvents are of low water composition and can be re-used without further processing. The aqueous distillate contains only about 8% butanols and therefore requires to be processed further in a solvent recovery plant before it can be re-used.

This processing is carried out on Solvent Recovery Plant No. 1 at BIP Chemicals Limited. The basic feedstock is aqueous distillate which is a multi-component liquid. In order to separate it into streams of the required quality it has to be processed through two distinct distillation operations. The plant is a 20-plate, bubble cap column which is used for each of the distillations termed D1 and D2. Figure 3.1 illustrates the solvent recovery operations as well as the recovered distribution system for both the iso-butanol and n-butanol.

It should be noted from this diagram that only the recovered iso-butanol, termed wet iso-butanol, receives solvent from the solvent recovery plant. This is because the aqueous distillate from both the iso-butylated and n-butylated resins

Figure 3·1 Solvent Recovery Plant Operation 1



are mixed into one tank prior to being processed. The recovered solvents are then fed to the wet iso-butanol tank. The recovered wet iso-butanol is therefore contaminated with n-butanol. This choice is made to ensure resins made with n-butanol do not contain iso-butanol.

3.2.1 D1 Distillation

The aqueous distillate, containing water, butanols, formaldehyde and methanol is fed to the fourth plate of the twenty plate column, numbering from the bottom up, and distilled. The bottom product is a water and formaldehyde solution termed weak formalin since it contains between 3 - 6% formaldehyde. This is removed to be used as a diluent in other plants. The top product is a mixture of water, methanol and butanols and is stored in a tank as feed for the D2 distillation.

3.2.2 D2 Distillation

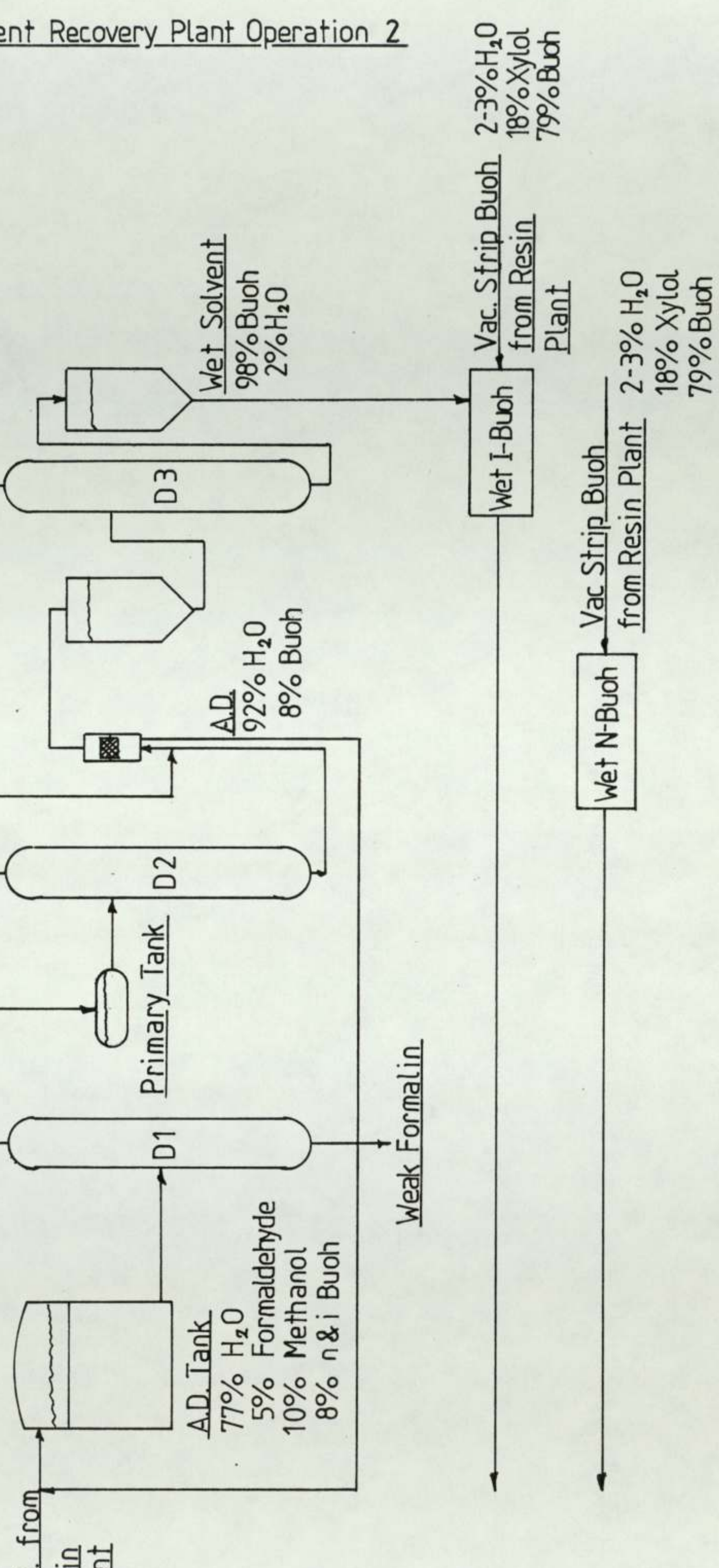
The feed for the D2 operation is pumped to the fifteenth plate of the same column. The feed is separated into a top product of methanol and water, which is pumped to the recovered methanol tank. The bottom product is an azeotropic mixture of butanols and water. On cooling, the bottom product settles into two layers, an aqueous phase and a solvent phase. The aqueous phase is re-cycled back to the main aqueous distillate storage and the remaining solvent phase is transferred into the wet iso-butanol tank.

This operation not only contaminates the iso-butanol with a quantity of normal butanol but due to the physical separation of the azeotrope a solvent containing about 20% water is fed to the wet iso-butanol tank. This introduces a variable water content into the reaction system through the subsequent use of this wet solvent. P.A. Freeman(4) has identified and installed a process which will reduce this water content to a minimum. This was achieved by installing a continuous knitmesh separator, which operates on the principle that one of the misible liquids will preferentially wet the material used to construct the knitmesh. This will cause droplets to coalesce and due to the difference in liquid densities then two layers will form. The two storage tanks freed by this method of separation can be used as holding tanks for the solvent layer so that it may be further processed by a D3 distillation operation. This is illustrated by Figure 3.2.

3.2.3 D3 Distillation

The feed to the D3 operation contains about 20% water in butanols and is pumped to the fifteenth plate of the same column as D1 and D2. This operation distills the feed into a bottom product of almost dry butanols (~2% water) and a top product which consists of the butanol-water azeotrope. The top product is separated into an aqueous layer which is pumped back to the aqueous distillate tank and a solvent layer which is re-cycled back to the D3 feed tank. The dry, bottom product which has a water content similar to that of the vacuum stripped solvents is sent to the wet iso-butanol tank. A diagram of this operation along with D1 and D2 is given in Figure 3.2.

Figure 3·2 Solvent Recovery Plant Operation 2



3.3 Methods for Determining the Wet Solvent Composition

So that batch to batch consistency can be obtained in the manufacture of butylated resin some means of determining the wet solvent composition on-line is required. This can then be used to make adjustments to ensure the correct amounts of reactants in the charges. The following are the methods investigated for this application :-

1) Conductivity
2) Gas Chromatography
3) Infra-Red Analysis
4) Capacitance
5) Ultra-Violet Analysis
6) Density Measurement

The extent of investigation varied depending on the initial laboratory results or the response of instrument suppliers when approached regarding the possible applications. The methods which gave promising results were installed for trial periods on the production plant. These methods and the outcome of the plant trials are discussed in this chapter.

3.3.1 Conductivity

Because of the presence of water in the ternary mixture it was thought that a conductivity cell might be of use in determining the water content of the mixture. Since no equipment was readily available to test this application at BIP, several instrument manufacturers were approached to determine the feasibility of this application under the conditions present in the wet solvent transfer lines. The results of these consultations were that if the water content was within the range 0 - 5% w/w, then the application would be feasible. Since the water content of the wet solvent can be up to 10% w/w it was decided not to proceed any further with this method.

3.3.2 Gas Chromatography

This method was originally used off-line by P.A. Freeman(4) to determine the compositions of the wet solvents and aqueous distillates in order to produce accurate mass balances for the reaction process and solvent recovery plant. This technique has the advantage that it will produce a complete analysis of the solvent stream. For this application to be investigated further Beckman Industrial were approached to determine if this method could be applied on-line. They indicated that this would be possible using one of their 6750 series Process Gas Chromatographs which was certified for operation in areas handling flammable materials, Zone 1. The solvent would be sampled approximately half way through the charging cycle and its analysis would take about twenty minutes. The output signals for the analyser unit are of a form which would allow them to be fed to a computer for further manipulation, such as charge adjustment calculations.

There were two disadvantages to applying this system to the production plant. Firstly, the cost of an analyser unit was in the order of £30,000 and could not be justified since it would only be used during wet solvent charging operations which only occur two or three times a day. Secondly, during the charging operation only one sample of solvent would be analysed since the analysis takes about twenty minutes which is about the maximum time taken to charge solvent to the reactor. This sample may not represent the bulk of the solvent charged since additions to the wet solvent holding tanks could be made during the charging operation, therefore changing the solvent balance. Therefore, this analytical technique was not pursued any further for on-line application.

3.3.3 Infra-Red Analysis

Work carried out by B.B. Gandhi(18) reported various laboratory techniques which could be utilised to carry out quick analysis of the wet solvents. This work indicated the possibility of using a dual Infra-Red Analyser system to determine the water and xylol composition of the wet solvent stream and hence the butanol concentration by difference. This route was pursued by contacting manufacturers of on-line infra-red analysers to investigate this application further. The results indicated that the water measurement would be affected by the butanol in the system and therefore the value obtained for the water content would be unreliable. This method was therefore not progressed for evaluation trials on the production plant.

3.4 Capacitance

Little previous work had been carried out using this method for determining solvent compositions, so a series of laboratory experiments were carried out to determine whether this method was worth pursuing.

The basic theory behind capacitance measurement is given by equation 3.1

$$C = Er\ Eo\ \frac{A}{d} \quad \ldots\ldots\ldots\ldots \quad 3.1$$

Where:

C - Capacitance

A - Area of plates

d - Separation of plates

Er - Relative permittivity of the Dielectric (ie. Dielectric Constant)

Eo - Permittivity of free space

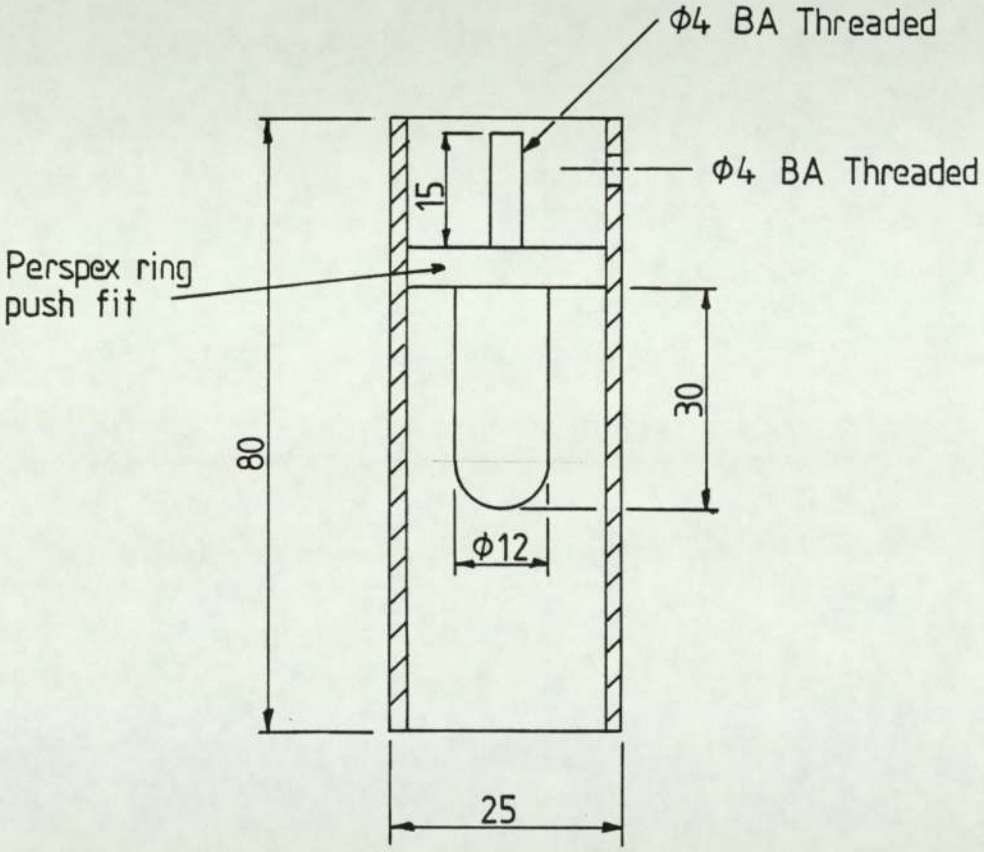
Therefore since EoA/d is a constant for the cell the capacitance is proportional to the dielectric constant which is a property of the material between the plates.

3.4.1 Experimental Investigations

For this work a direct reading digital capacitance bridge was used and a capacitance cell was fabricated, Figure 3.3. Initially samples of n-butanol/xylol were used in the cell and their capacitances obtained. These results are given in Table 3.1, the relationship is linear and is illustrated by Figure 3.4.

This experiment was repeated for n-butanol/water and n-butanol/xylol/water mixtures. No usable results were obtained for the mixtures due to the water content

Figure 3·3 Capacitance Cell



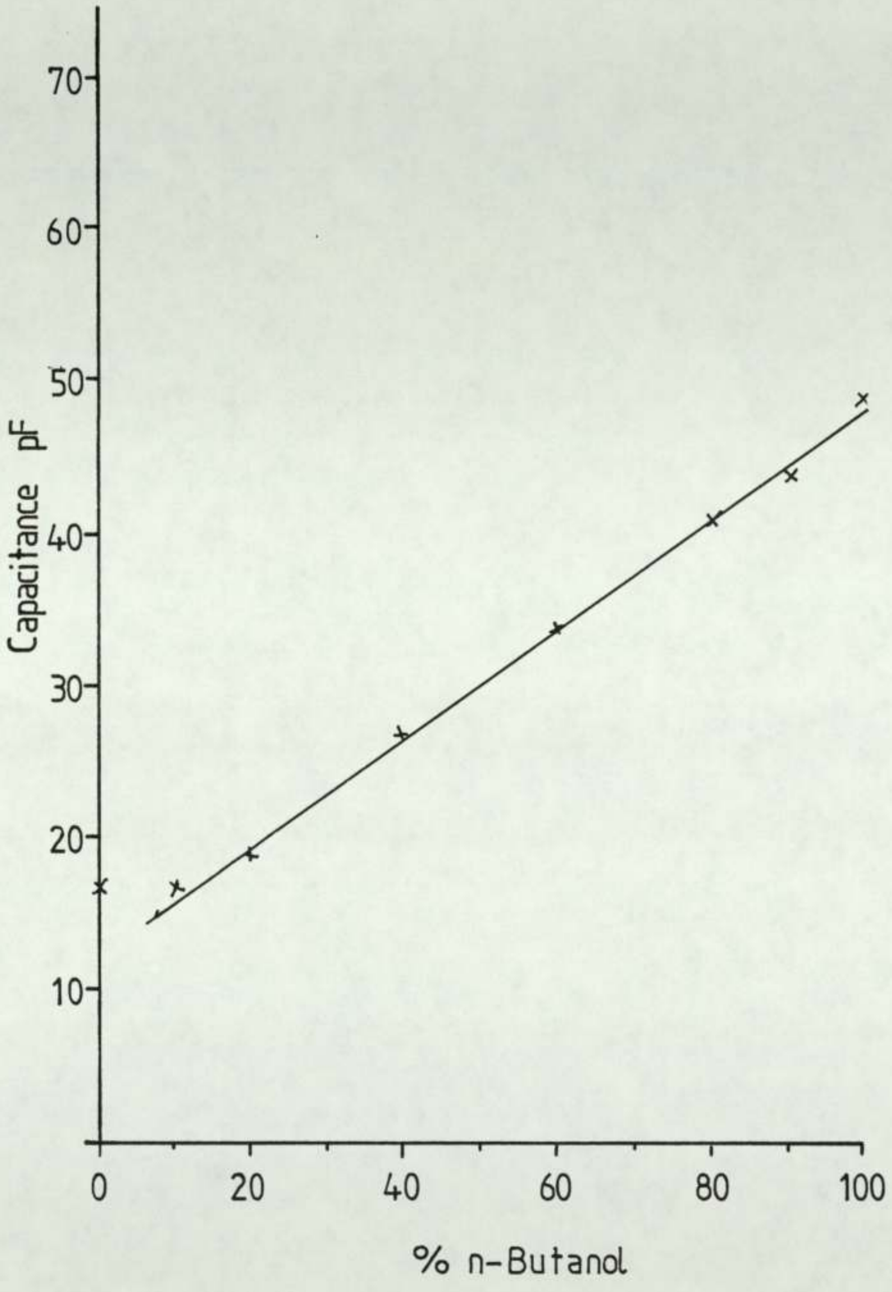
All dimensions in mm

Material – Brass & Perspex

Table 3.1 Composition vs Capacitance for n-Butanol/Xylol mixtures

Sample No.	(w/w)% n-Butanol	Capacitance pF
C1	100	49
C2	90	44
C3	80	41
C4	60	34
C5	40	27
C6	20	19
C7	10	17
C8	0	17

Figure 3·4 Graph of Capacitance vs n-Butanol Concentration



causing a short-circuit. This problem would not be solved by using a non conducting cell, since this would only measure the capacitance of the non-conductive material in the cell. Therefore this method was not progressed any further.

3.5 Ultra-Violet Analysis

In the work reported by B.B. Gandhi(18) the use of an Ultra-Violet Analyser was investigated in order to determine the xylol content of any given recovered solvent. It was not possible to use this method for the other components due to cross interference of the absorbance peaks. This work proved successful on laboratory based equipment and this method was investigated further as an on-line analytical method. An on-line analyser unit was hired and installed in the recovered n-butanol stream feeding the butylated resins plant. The n-butanol line was selected since it would link up with the work conducted on computer control which related mainly to n-butylated resins. The following describes the experimental work carried out on this unit.

3.5.1 Equipment Description

The instrument supplied by Telsec Process Analysers consisted of two separate rack mountable units, these were the Optical Unit and the Electronics Unit. Both these units were sealed and could be purged with air where the requirement was for an installation in an area where a flammable atmosphere could be present, termed Zone 1.

3.5.2 Optical Units

This unit contained the complete optical system for the analysis of the solvent stream. The unit operated in the following way. The spectrum being analysed was in the Ultra-Violet range and in this case the light source was provided by a Pen-Ray lamp producing light of a wave length of 285 nm. The light beam was split and passed through a reference cell and a sample cell. The sample cell for this application had a path length of 1 mm and was supplied continuously from the wet solvent stream via a ¼" sample line. The two beams were chopped by a rotating disc which allowed the detector to relate the absorbtion of the measuring cell and the reference cell before sending a signal via the pre-amplifier to the Electronics Unit.

3.5.3 Electronics Unit

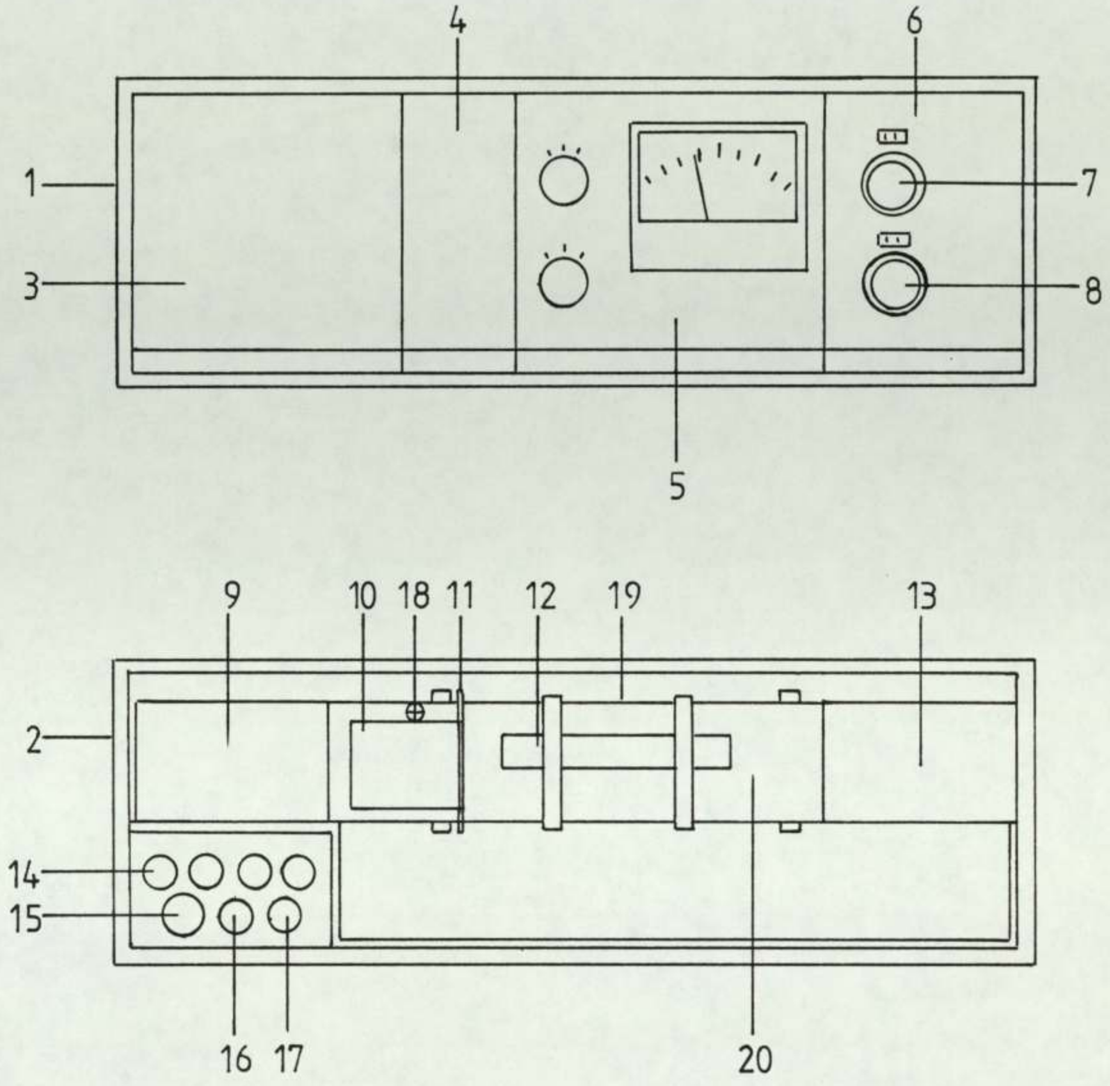
The Electronics Unit could comprise of up to four plug-in modules. In this system three were used, consisting of the Power Supply Module, the Amplifier Module which contained the zero and span control and the Display Module. The output was presented on a 0 - 20 mA meter. This unit could be purged, the need for this depending on its location, which could be up to 100 metres from the Optical Unit.

A diagramatic outline of the units described above is given in Figure 3.5.

3.5.4 Installation and Calibration

The Optical and Electronics Units were located on the

Figure 3·5 U-V Analyser Diagram of Main Components



1. Electronics unit
2. Optical unit
3. Power supply module
4. Spare module position
5. Display module
6. Amplifier module
7. Span control
8. Zero control
9. Light source
10. Modulator
11. Rotating segment plate
12. Measuring cell
13. Detector assembly
14. Process connections
15. Purge coupling
16. Plug PL 1
17. Socket SK2
18. Shutter adjustment (optical zero)
19. Optical bench
20. Position for calibration disc

outer wall of the Tank Farm which contained the recovered solvent tanks. This location was chosen because a sampling point was already available on the discharge side of the wet n-butanol pump. As well as this, the sample return could go back to the wet solvent tank, thus alleviating the need for an alternative method of returning the sample to the process stream. Both the units were housed in the wooden cabinet, and each was purged, because of the units' location next to the Tank Farm. The power for the instruments was supplied via an armoured cable. The 4 - 20 mA output signal from the electronics unit, which was related to the xylol composition of the wet solvent stream, was sent to the control panel in the Butylated Resins Plant.

Here the signal was converted to a 2-10 V output by placing a 500Ω resistance in parallel with the current output. This voltage signal was then transmitted to the SEED 19 microcomputer, previously described in Chapter 2, where it was converted into a digital value. This value was then used to calculate the xylol composition of the wet n-butanol stream using the Lagrange Interpolation Formulae, Appendix 4, since the relationship between the instrument output and xylol composition is non-linear.

The analyser was pre-calibrated by the suppliers for the required xylol composition range. These settings and calibration values are given in Appendix 4 and the instrument was set up as indicated. The calibration work required in-situ was to the analogue signal being fed to the computer. This required that currents in the 4-20mA range be sent along the communication line to the control panel for conversion into a voltage which is then further converted by the SEED 19 to a digital value. This produced a calibration curve of current supplied versus digital value, Appendix 4, and could be related to the xylol composition.

Table 3.2 Results of Analyser Trials

SAMPLE NO *	XYLOL COMPOSITION WT%	
	GLC	ULTRA VIOLET
UV 1	14.8	13.7
UV 2	14.6	13.0
UV 3	15.4	14.8
UV 4	16.5	15.1
UV 5	17.2	17.6
UV 6	19.9	20.0 +
UV 7	22.0	20.0 *

* For complete analysis of each sample refer to Appendix 4

\+ Analyser output reading over full scale deflection

3.5.5 Practical Problems

Initially several problems were encountered during the installation of the analyser unit due to the confusion over its Zone Classification. At first the unit was to be housed in the pump room on the tank farm, hence providing a short sampling lag time. This was not possible due to the classification problem and the unit was eventually located on the exterior wall of the tank farm in a purged cabinet, the sampling lag time was therefore increased but did not appear to have any significant effect on the values obtained during any charging operation. A problem was also encountered with the signal being transmitted to the SEED microcomputer from the analyser unit, which resulted in a maximum output being registered on the computer at only about three quarters of the maximum xylol content. This was caused by a resistance in the link due to the routing of the signal through several control panels. The problem was eventually rectified and the output signal from the analyser was calibrated with the digital values produced by the computer.

3.5.6 Discussion

The trials carried out using the analyser showed that the xylol content could be determined accurately taking into account the system error of the U-V analyser of ±0.4 Wt% in the xylol composition and the error in the independant analysis using Gas-liquid Chromatography of ± 0·5 Wt%. The accuracy and reproducibility would be improved by a much more rigorous initial calibration period, which was not available during the trials. Overall continuous analysis of the Wet n-butanol stream has shown that the xylol content varied between 14 - 18 Wt % with only occasional movement outside of this range. Towards the end of the hire period the analyser was found to be generating a maximum output for the xylol composition. This was initially thought to have been a fault in the system but subsequent analysis has shown that the xylol content of the stream was greater than 20Wt% on these occasions. This was due to a change in the resin being manufactured which in turn produced vacuum stripped solvents with higher than normal xylol contents. These results would suggest that the operating range for the instrument should now be calibrated as 5 - 25 Wt%.

The use of the Lagrange Interpolation formula by the computer software to determine the xylol content from the input signal, operated well and had the advantage that it could interpolate between known values rather than linearising the calibration date, which would then introduce an error into the evaluation of the xylol content.

3.5.7 Conclusion

From the trials carried out it was concluded that the use of an in-line U-V analyser provided a method of determining the xylol content in a wet solvent stream, provided that a rigorous calibration procedure was followed and that the maximum and minimum limits for the xylol content were known. These should be taken as 5% and 25% respectively.

3.6 Density Measurement

The use of an Ultra-Violet Analyser unit can only determine the xylol composition in the wet solvent stream. The mixture comprises of three components and an additional method was required to fully determine the composition of the wet solvent. The measurement of its density was considered to be a suitable parameter which could be used for this determination.

Two methods could be used to determine the streams overall composition. A series of calibration curves could be obtained for solutions with a fixed xylol content but varying butanol and water ratios and so for any known density and xylol content the two other components could be determined. Alternatively, for this work it was assumed that the relationship between the density and composition was linear. This meant that the composition of the stream could be calculated by a computer using the density and xylol content data.

The measurement of the solvents density meant that the mass of the wet solvent charged to the reactor could be determined, since this solvent is normally metered volumetrically to the reactors. This gave the mass of each component charged to the reactor, the most important of these being the water, which was used to determine the mass of aqueous distillate which was required to be removed during the reaction.

To test the accuracy of an on-line density measuring instrument, a DPR 417Y unit was borrowed from Stanton Redcroft for an evaluation period. It was originally planned that trials would be carried out in conjunction with the U-V analyser. Unfortunately due to installation difficulties this was not possible. Hence, separate trials were carried out to evaluate the accuracy and reproducibility of the density instrument. In order to check the instrument accuracy an off-line laboratory density meter, Anton-Paar DMA46, was used. This produced a density value accurate to 4 decimal places.

3.6.1 Equipment Description

The measuring system obtained from Stanton-Redcroft consisted of two pieces of equipment, the Measuring Cell and the Electronic Evaluation Unit.

The liquid sample was passed through a U-shaped oscillator tube, in this case constructed of stainless steel. The tube was electronically excited at its natural frequency. This frequency was therefore not only a function of the tube's mechanical properties but also of the density of the fluid filling the unit, hence a linear relationship was produced over specified density ranges between the frequency of oscillation and the fluid density. The unit borrowed was housed in an explosion proof casing allowing it to be located in Zone 1 areas. It also came equipped with a mechanical temperature compensator, which involved subjecting the U-tube to a tensioning force depending on the coefficient of expansion of the fluid involved.

3.6.2 Electronic Evaluation Unit

The advantage of this unit was that it could be assembled in a modular form allowing for various options to be included in the interpretation and output of the signals received. This particular unit was equipped with the following modules. Power Supply 220/110/24 V, Processor, Twin Wire Receiver, Quartz Time Base, Indicator Module, Signal Interface set for analogue outputs of 0-10V. The unit, due to the electronics contained within the modules was not intrinsically safe, but when provided with transmission wires of a low resistance could drive the measuring cell from up to 2 km away.

3.6.3 Calibration

The instrument was calibrated before being located on the plant, in the following manner. Several samples of wet solvent of a known composition and density were used for calibration purposes. These samples were individually placed in the Measuring Cell and the system constants were adjusted to give a suitably sensitive and accurate frequency range for the compositions under investigation. Having obtained suitable system constants the next step was to set the temperature compensation on the U-tube. This was carried out by heating a typical solvent sample in a beaker and determining its density at various temperatures over a short temperature range. This data supplies the co-efficient of thermal expansion for the fluid, B, and is used to obtain the mechanical temperature compensation value by multiplying B by the average sample density from the temperature range investigated. This value was then set accordingly on the tension bar fitted to the U-tube and the unit closed up. The calibration data is included in Appendix 4.

3.6.4 Installation

The density meter was installed using the same sample lines and communications links used for the UV Analyser. The Measuring Cell was therefore mounted on the exterior wall of the tank farm, whilst the Electronics Unit, which was not intrinsically safe, was located in the Solvent Recovery Plant 1 Control Room. The instrument was supplied with power from the control room and the 0-10V analogue output from the Electronics Unit, which was proportional to the fluid density, was connected to a chart recorder on the SRP 1 control panel to provide a visual record of the density variation.

3.6.5 Results

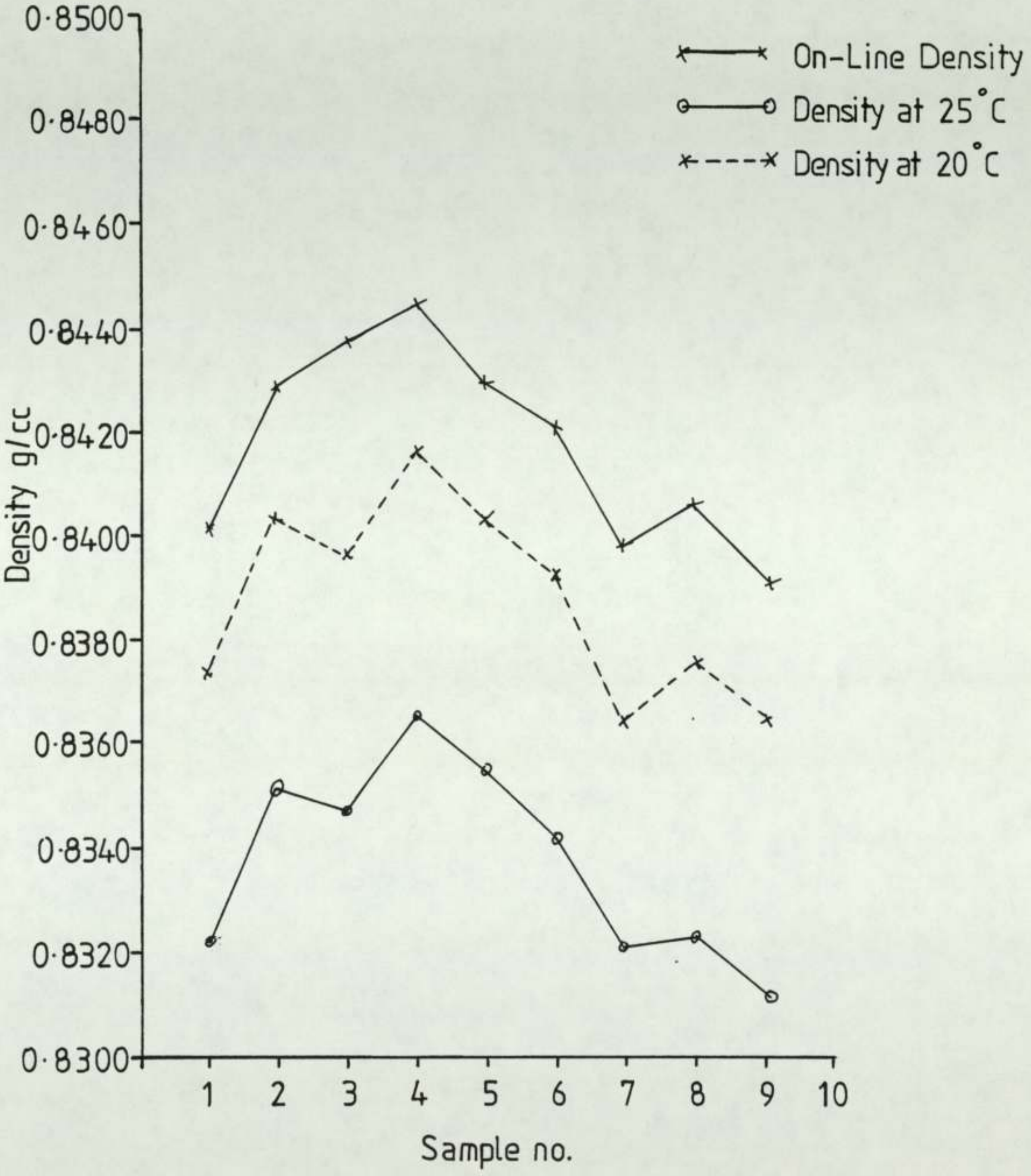
The initial trials of the measuring unit indicated that the resonant frequency of the U-tube and wet solvent was at the upper end of the pre-calibrated span and it did exceed this upper limit on one occasion. The span was then increased to allow for this variation and to enable the value to be indicated on the chart recorder. Altering the span setting does not have any effect on the resonant frequency obtained. It only affects the range over which the 0-10V output to the chart recorder is applicable.

The temperature compensation for these trials was carried out over the range 15 - 25^{o}C with density values being related to 20^{o}C. Initially to check the reproducibility of the in-line measured density, sample densities were checked in the control laboratory using the DMA 46, with a sample temperature of 25^{o}C. Referring to Table 3.3

Table 3.3 Results of On-line Density Meter Trials

Sample No.	Resonant Frequency (Hz)	Calculated In-Line Density (g/cc)	Measured Density @ 20°C (g/cc)	Measured Density @ 25°C (g/cc)
D1	255 219	0·8402	0·8374	0·8322
D2	255 356	0·8430	0·8404	0·8352
D3	255 396	0·8438	0·8397	0·8347
D4	255 432	0·8445	0·8417	0·8366
D5	255 358	0·8430	0·8404	0·8355
D6	255 314	0·8421	0·8393	0·8342
D7	255 164	0·8391	0·8365	0·8312
D8	255 241	0·8407	0·8376	0·8323
D9	255 168	0·8392	0·8365	0·8312

Figure 3·6 Comparison between On-Line Density Measurment and Measured Density at 20°C & 25°C



and Figure 3.6 it can be seen that the density values produced the same profile, indicating that the in-line unit was relating the density to a fixed temperature. On completing the in-line trials the densities of the samples were again checked, but this time the DMA 46 was re-set to give a sample temperature of 20°C, so that the accuracy of the measuring unit could be assessed. By again referring to Figure 3.6 it can be seen that these values correlate well with those obtained by the in-line measurement at the same temperature. The error in density obtained is ± 0.003 g/cc which is an error of about ± 0.35% in the density value obtained. The above error obtained is small but could be reduced further by using a more rigorous calibration to obtain the temperature compensation value. For these trials this calibration was conducted using a hand held density meter which only had a 3-decimal place accuracy. If a DMA 46 or more accurate unit was used this would provide a 4-decimal place or better accuracy which would provide a much better temperature compensation value. It should be pointed out that the mechanical temperature compensation on the trial unit has its own built in inaccuracy since it relies on the tensioning of the U-tube to the correct value by the calibrator which introduces human error into the system. A more advisable route would be to use a unit which was equipped with electronic temperature compensation to obtain a more accurate density value. The temperature range required for accurate calibration would be -10°C to 30°C.

3.6.6 Conclusion

It can be concluded that from these trials the accurate in-line determination of the wet solvent density is feasible, provided an accurate initial calibration is conducted along with the instrument being used in conjunction with an accurate method of providing temperature compensation. Therefore the use of in-line density measurement together with xylol analysis would provide a method of determining the Butanol, Water and Xylol composition in a wet solvent stream.

3.7. On-Line Viscometry

A method of monitoring the viscosity of the reactant mixture on-line was required in order to help prevent the reactors contents from gelling due to over polymerisation. The operational method for determining the viscosity is by an off-line test which can take up to twenty minutes for a result. This time delay can mean that the product resin could, at worst, gel, or be out of specification if the reaction conditions have been affected.

Works was carried out by P.A. Freeman(4) using a Bendix 1800 Viscometer on laboratory and pilot plant scale reactors to monitor the relative viscosity changes throughout the reaction cycle. This proved successful with the instrument detecting changes in the resin viscosity at the solvent addition points and during the acid reaction stage. This instrument was installed on the production reactor being used for the investigation work.

3.7.1 Equipment Description

This instrument consisted of two parts, the electronic and display module and the sensing head. The instrument operated on the principle of measuring the damping effect of the fluid on a reed in the sensing head which was excited in the longitudinal plane by the electronic unit. The electronics unit excited the oscillation of the reed, measured the damped oscillation to a fixed value and then sent another excitation to the reed. The frequency of excitation is proportional to the viscosity of the fluid and is displayed as the product of viscosity and density.

3.7.2 Instrument Installation

The instrument was calibrated using two standard oils covering the viscosity range of interest. The probe was then installed in the reactor so that it would be submerged in the fluid throughout the production cycle. This was connected by a coaxial cable to the electronics unit located in a safe area. In order that the output of the instrument could be monitored by the computer a 4 - 20 mA output card was installed in the unit and a 500Ω resistor connected in parallel with this to give a 2 - 10 V signal which was fed to the computer's analogue to digital converter. A graphics program was written so that the viscosity data could be presented in a trend display throughout the reaction cycle.

3.7.3 Discussion of Results

While this instrument was installed in the reactor, during the trial period, no useful results were obtained. This was due to the reed on the sensor assembly breaking off. The probe head was located in a different orientation but the reed snapped off again producing no useful feedback to the computer.

The cause of these failures was thought to be due to the increased severity of the agitation in the full scale reactor compared to the pilot scale vessel since all other factors in terms of materials and reaction conditions were the same.

3.7.4 Conclusion

It has been shown that the Bendix 1800 Viscometer is not a robust enough instrument in its present form to survive the conditions encountered in the full scale production vessels at BIP. Its usefullness has been shown(4,20) in pilot plant and laboratory trials and if a suitable method of baffling the agitated fluid could be found then the instrument may still prove itself, otherwise further investigation will need to be carried out to determine alternative methods for in-still viscosity measurement.

3.8 General Discussion and Conclusion

The results of this work show that an on-line system for determining the composition of the recovered solvent containing butanol, water and xylol is possible using an Ultra-Violet Photometer to determine the xylol composition. This would be used in conjunction with an on-line density measuring instrument which would provide the means to determine the butanol and water composition. Both instruments would be connected to the input devices of a computer so that their outputs could be accepted and analysed by the computer and the composition of the solvent calculated. The degree of accuracy to which the composition is obtained will depend on two factors. The first is the accuracy to which the initial calibration of the instruments is carried out with respect to the measurement to be made, together with the accuracy of the conversions carried out on the signal generated. The second factor is the method by which the computer software manipulates the two values to give the composition of the recovered solvent. This can either be through the use of an approximate linear relationship between composition and density or the inclusion of a series of density versus composition curves for differing xylol contents which would give the water to butanol ratio in the mixture. The second method would be the preferred option since it would be more accurate when dealing with this non ideal mixture.

The use of the above analytical techniques to determine the recovered solvent composition and then the use of these values to make appropriate adjustments to the pure solvent charges to the reactor enables more consistent production. It also allows the prediction of the amount of water to be removed during the reaction.

The Bendix 1800 Viscometer has proved not to be reliable enough for use in a full size production reactor even after successful pilot plant trials. It is therefore necessary to determine whether it is possible to industrially harden this instrument, or alternatively find a more suitable instrument.

4. COMPUTER CONTROL TRIALS AND SOFTWARE DEVELOPMENT

4.1 Introduction

Previous chapters have dealt with the development of the computer system. The software and hardware were discussed in Chapter 2 and the on-line instrumentation in Chapter 3. Both of these are needed to bring about effective control of the batch production process. This chapter is concerned with the application of a suitable control strategy to the full scale production plant.

A first series of trials were carried out to confirm the general philosophy regarding computer control for the production of butylated resins. These were carried out in conjunction with the tests of the on-line Ultra-Violet Analyser already described in Section 3.5. The trials were not complete computer control but generated prompts for control actions at the appropriate points. After completion of these trials, Digital to Analogue converters were installed on the computer system and additional software written to allow the control loop to be closed by passing signals to the plant and bring about full computer control of the process. Results are included which show the effects of signal integrity from certain plant instruments to the computers analogue to digital converter.

4.2 Computer Aided Plant Trials

These trials were conducted on the full scale production facility using the software and hardware described in

Chapter 2 and following the operating procedure given in Appendix 6. Three separate trials were carried out on types B and C resins using an initial wet charge of normal butanol as described in Chapter 1. This charge cannot be made up entirely from wet n-butanol and the difference was made up with dry n-butanol. These trials were conducted while the Ultra-Violet Analyser was installed on the wet n-butanol transfer line and a program was written to receive and convert the output from the analyser unit. This was described in Section 3.7.4.

4.2.1 Objectives

The series of Computer Aided Plant trials were carried out with the aim of meeting several objectives at the one time, which are outlined below.

1) The software written for the BBC and SEED micro-computers was tested to establish that the correct prompts for manual actions were being generated at the appropriate times during the process. The reliability of the software and the hardware over the length of a production run was also evaluated.

2) The n-butanol, xylol and water compositions of the wet solvent stream were determined using the U-V analyser and the DMA 46 density meter. These values were then used to calculate charge adjustments and the mass of aqueous distillate to be removed during each stage of the reaction.

3) The accuracy and consistency of the signals received by the SEED computer from the plant instrumentation

and their subsequent conversion to engineering units by the software was also evaluated.

The results obtained from these trials are given in Table 4.1 which includes a comparison with predictions for the water to be removed, using the results of the on-line analysis techniques, where available, and predictions based upon the work of Freeman(4), on resin of type A.

4.2.2 Results

For each of the objectives given in Section 4.2.1 for this series of trials the following results and observations can be made.

1) The operation of the computer software was successful. Appendix 5 contains copies of logsheets made during these trials. Figure 4.1 gives typical extracts from the logsheets for key stages in the process. From these it can be seen that the software carried out the appropriate actions and generated the correct prompts, based on the data received from the process and analytical instruments. The three trials carried out demonstrated that the computer and its peripheral devices were capable of operating satisfactorily during a complete production cycle which has a duration of about 24 hours. Since these trials were carried out on separate occasions they did not test the reliability of the computer system over longer periods.

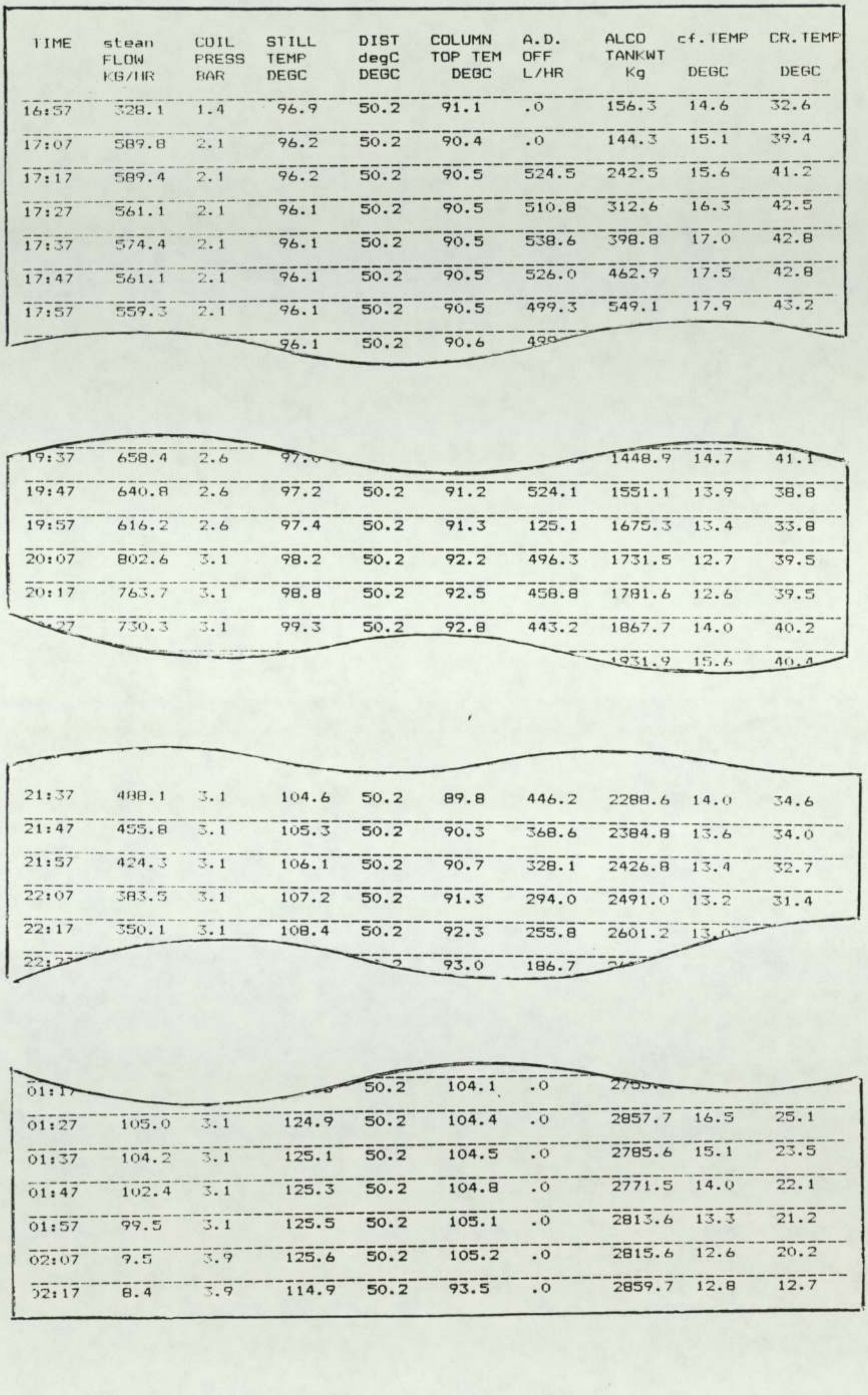
2) The on-line Ultra-Violet analyser was only partially

Table 4.1 Results of Trials

INPUT VALUES					PREDICTED VALUES			ACTUAL VALUES		
Date	Resin Type	Formalin Water Kg	Wet Solvent Water Kg	Water of Reaction (Condensation & Butylation) Dehydration Kg	Total AD Off During Dehydration Kg	Water of Formation Acid Reaction Kg	Total AD Off At End Of Acid Reaction Kg	Total AD Off at End Of Dehydration -Tare Kg	Total AD Off At End Of Acid Reaction -Tare Kg	Water of Reaction (Condensaton) & Butylation) Acid Reaction Kg
21.4.85	C	1626	124	350	2100	530	2630	2103	2612	509
30.4.85	B	1627	*88	350	2065	530 +	2595	2044	2383	339
1.5.85	B	1627	*123	350	2100	530 +	2596	2005	2397	392

+	Predicted value based on Type C Resin
*	Assumed value due to analyser failure
AD	Aqueous Distillate

Figure 4·1 Extract from Logsheet for Computer Aided Control Trials



TIME	steam FLOW KG/HR	COIL PRESS BAR	STILL TEMP DEGC	DIST degC DEGC	COLUMN TOP TEM DEGC	A.D. OFF L/HR	ALCO TANKWT Kg	cf.TEMP DEGC	CR.TEMP DEGC
16:57	328.1	1.4	96.9	50.2	91.1	.0	156.3	14.6	32.6
17:07	589.8	2.1	96.2	50.2	90.4	.0	144.3	15.1	39.4
17:17	589.4	2.1	96.2	50.2	90.5	524.5	242.5	15.6	41.2
17:27	561.1	2.1	96.1	50.2	90.5	510.8	312.6	16.3	42.5
17:37	574.4	2.1	96.1	50.2	90.5	538.6	398.8	17.0	42.8
17:47	561.1	2.1	96.1	50.2	90.5	526.0	462.9	17.5	42.8
17:57	559.3	2.1	96.1	50.2	90.5	499.3	549.1	17.9	43.2
[illegible]			96.1	50.2	90.6	[illegible]			
19:37	658.4	2.6	[illegible]			[illegible]	1448.9	14.7	41.1
19:47	640.8	2.6	97.2	50.2	91.2	524.1	1551.1	13.9	38.8
19:57	616.2	2.6	97.4	50.2	91.3	125.1	1675.3	13.4	33.8
20:07	802.6	3.1	98.2	50.2	92.2	496.3	1731.5	12.7	39.5
20:17	763.7	3.1	98.8	50.2	92.5	458.8	1781.6	12.6	39.5
[illegible]	730.3	3.1	99.3	50.2	92.8	443.2	1867.7	14.0	40.2
							1931.9	15.6	40.4
21:37	488.1	3.1	104.6	50.2	89.8	446.2	2288.6	14.0	34.6
21:47	455.8	3.1	105.3	50.2	90.3	368.6	2384.8	13.6	34.0
21:57	424.3	3.1	106.1	50.2	90.7	328.1	2426.8	13.4	32.7
22:07	383.5	3.1	107.2	50.2	91.3	294.0	2491.0	13.2	31.4
22:17	350.1	3.1	108.4	50.2	92.3	255.8	2601.2	[illegible]	
[illegible]				[illegible]	93.0	186.7	[illegible]		
[illegible]			[illegible]	50.2	104.1	.0	[illegible]		
01:27	105.0	3.1	124.9	50.2	104.4	.0	2857.7	16.5	25.1
01:37	104.2	3.1	125.1	50.2	104.5	.0	2785.6	15.1	23.5
01:47	102.4	3.1	125.3	50.2	104.8	.0	2771.5	14.0	22.1
01:57	99.5	3.1	125.5	50.2	105.1	.0	2813.6	13.3	21.2
02:07	9.5	3.9	125.6	50.2	105.2	.0	2815.6	12.6	20.2
02:17	8.4	3.9	114.9	50.2	93.5	.0	2859.7	12.8	12.7

successful in determining the n-butanol, water, xylol composition of the wet solvent stream during the computer control trials. The first trial carried out for a type C batch of resin used the results from the instrument to accurately predict the weight of aqueous distillate which was to be removed during the dehydration and acid reaction stages. The computer generated the appropriate prompts at these end points.

During the second and third trials the analyser indicated a xylol content that was outside the upper limit set on the unit. This value was inconsistent with the previously observed composition range and it was assumed that the analyser was at fault and an estimated value was used for these compositions based on previous observations. It was subsequently determined that the analyser was operating correctly and the deviation occured due to a change in the product mix on the production plant.

There is a deviation between the actual and predicted weights of aqueous distillate removed during the dehydration stage. An error in the estimated value for the wet solvent would account for this deviation. There is a much larger difference between actual and predicted values for the aqueous distillate removed during the acid reaction stage for the second and third trials. The predicted value for the water produced during the acid reaction stage was based on type A resins, and was used for types B and C, as the correct value was not available. Type B resins have a shorter acid reaction period.

3) The acquisition of the data and its subsequent conversion by the software into engineering units produced values comparable with those displayed on the main control panel, with the exception of two instruments. The comparison of values are reported on the logsheets in Appendix 5. The reactor temperature value reported was between 1 and 2 degrees centigrade higher on the control panel compared to the computer display. This was found to be due to an error in the displayed value on the control panels chart recorder which did not affect the computer read out, these values being derived independantly from a common source. This was subsequently corrected for the other trials. The other instrument for which differences were found was the aqueous distillate weight, which oscillated on the computer display by ±25 kg over the production range of 100kg to 2500kg. This oscillation is equivalent to a variation in the electrical signal received by the analogue to digital converter of ±31 mV on a full range of 4000kg and an output of 5 Volts. This variability is wholly explained by the limited precision of the pneumatic to electrical conversion. This oscillation was

reduced by the incorporation of a ten point running average routine into the software. This allowed a more precise determination of the value of the aqueous distillate weight.

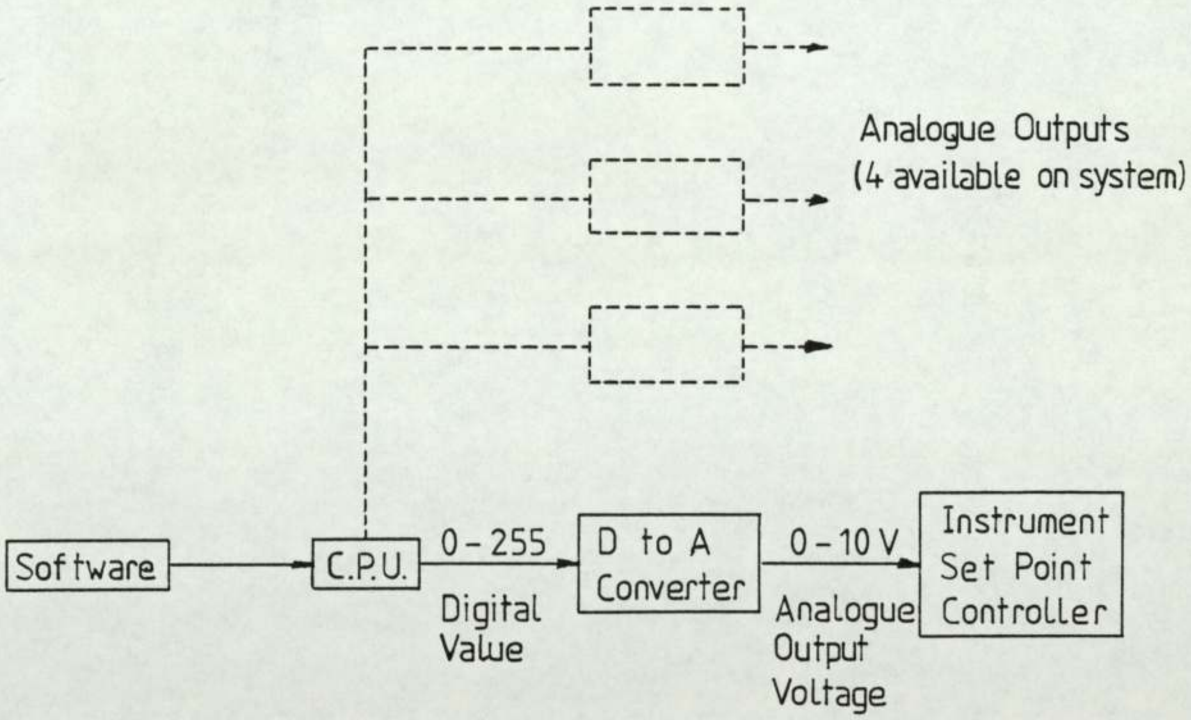
4.2.3 Conclusions

The computer aided control trials proved successful in meeting the objectives and demonstrating the reliability of the computer system. The software which had been written was shown to be reliable enough to be used in the development of a closed loop control system in conjunction with the installed control equipment. It was shown to be important to have a method of determining the wet solvent composition accurately so that the aqueous distillate to be removed could be calculated for each stage of the reaction. It is also necessary to know the extent of acid reaction for each particular resin. Finally it was shown that the integrity of the signals received and converted by the computer, except for the Aqueous Distillate Tank Weight, was sufficient to provide accurate control information for the process. Steps were taken to improve the accuracy and precision of measurements where discrepancies were detected.

4.3 Digital to Analogue Converters

In order to close the control loop, a means was required for sending the desired controller set point value from the computer to the plant instrument. This was achieved by installing two dual 8-bit digital to analogue converters on the SEED microcomputer. The configuration arrangements are given in Appendix 8. This allowed for a digital value, proportional to the required set point, in the range 0-255 to be sent from the computer software to the converter from which a proportional 0-10 V signal was generated to drive each instrument's set point. This is shown more clearly by Figure 4.2.

Figure 4.2 Digital to Analogue Converter Operation



In addition to the installation of this hardware, device drivers and descriptors were added to the operating system and user written procedures called these to gain access to the new hardware. The calibration procedure for these converters was similar to that for the analogue to digital converters described in Chapter 2, except in this case the digital value was generated by the computer and the response of the controllers' set point was noted. The installed converters had the capability of interfacing with four plant controllers. On this facility only three were required. Table 4.2 gives the controllers used with their ranges and the conversions used.

Table 4.2 Reactor Controllers with Ranges and Conversion Factors

			Conversion Factors	
Controller	Range	Units	M	C
Steam Pressure	0 - 4	barg	0·01569	-
Vacuum	0 -1000	mm Hg	0·255	-
Column Top Temp	55 - 105	^oC	0·1961	55

The conversion factors give the linear relationship between the digital value and the engineering value, the equation is of the form $E = mD + C$, where E is the engineering value and D is the digital value. It is important to note that the digital value takes only the integer values 0 - 255.

4.4 Additional Control Procedure

To make use of the signal to the plant controllers some additional software modules were required which were added to the software packages already written. The Spec 200 Set Point Controllers have their own in-built 3 - term control algorithims and so a loop control program did not require to be written into the extra software, which needed only to provide a set point - Figure 4.3, 4.4 and 4.5 show how these additional procedures and their flag files linked into the software structure. Each of the procedures and their function is now described.

4.4.1 Procedure Steamset

This procedure was used to adjust the steam pressure applied to the jacket and coils of the reaction vessel during the reaction. The required steam pressure was taken from the flag file 'steamon' as a digital value. The digital values were determined from the standard processing conditions for each stage in the reaction. This value was then set to the digital to analogue converter which output the appropriate voltage to drive the supervisory controllers set point to the desired position. This procedure periodically scanned the flag file at one minute intervals to detect if a change in steam pressure was required.

4.4.2 Procedure Coltop

This procedure was similar in operation to 'steamset' except that it obtained a value from the flag file 'columnt'.

Figure 4·3 Procedure/Flag File Interaction for Logging and Display Package

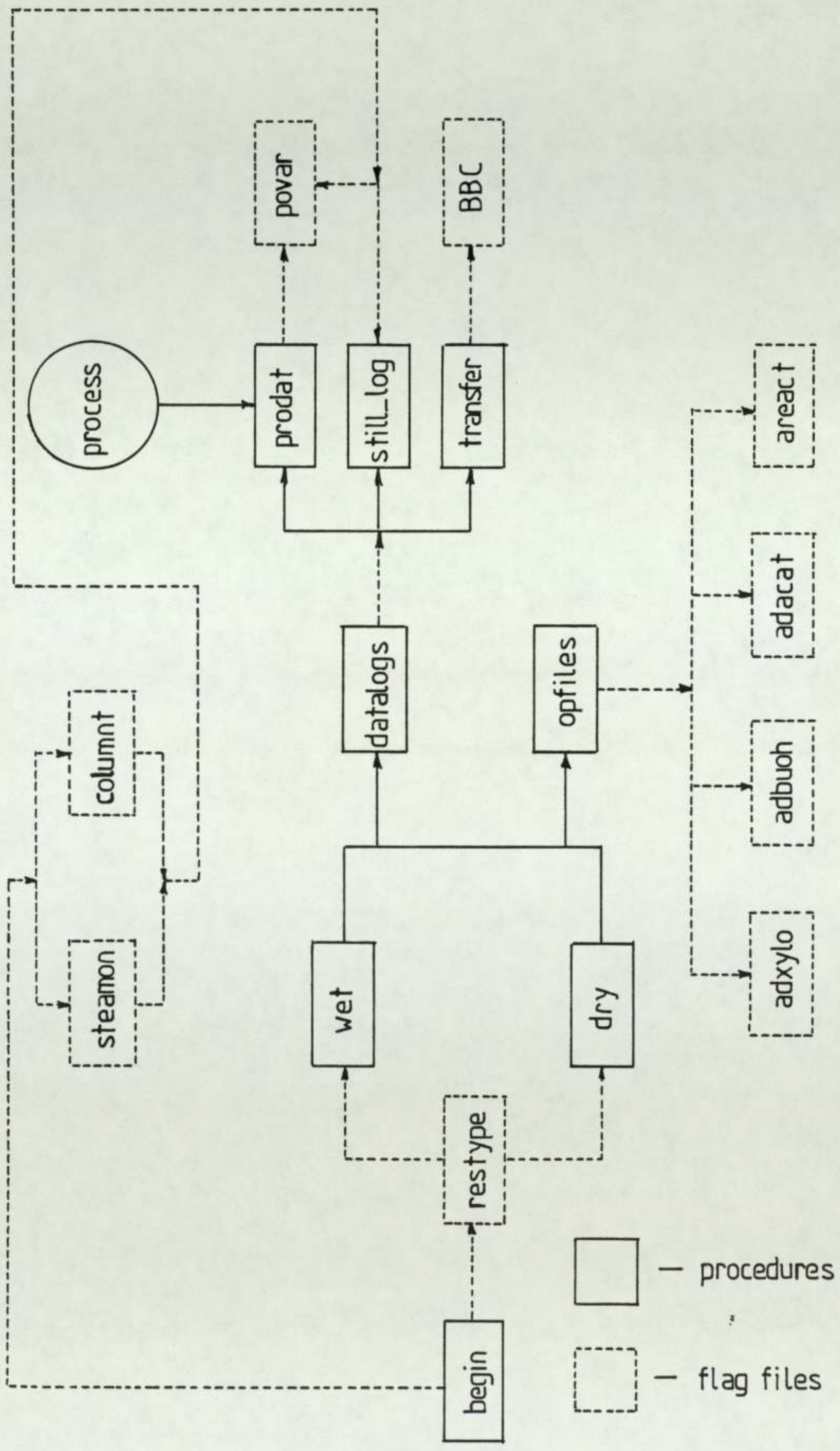


Figure 4·4 Procedure/Flag File Interaction for Monitoring and Control Package

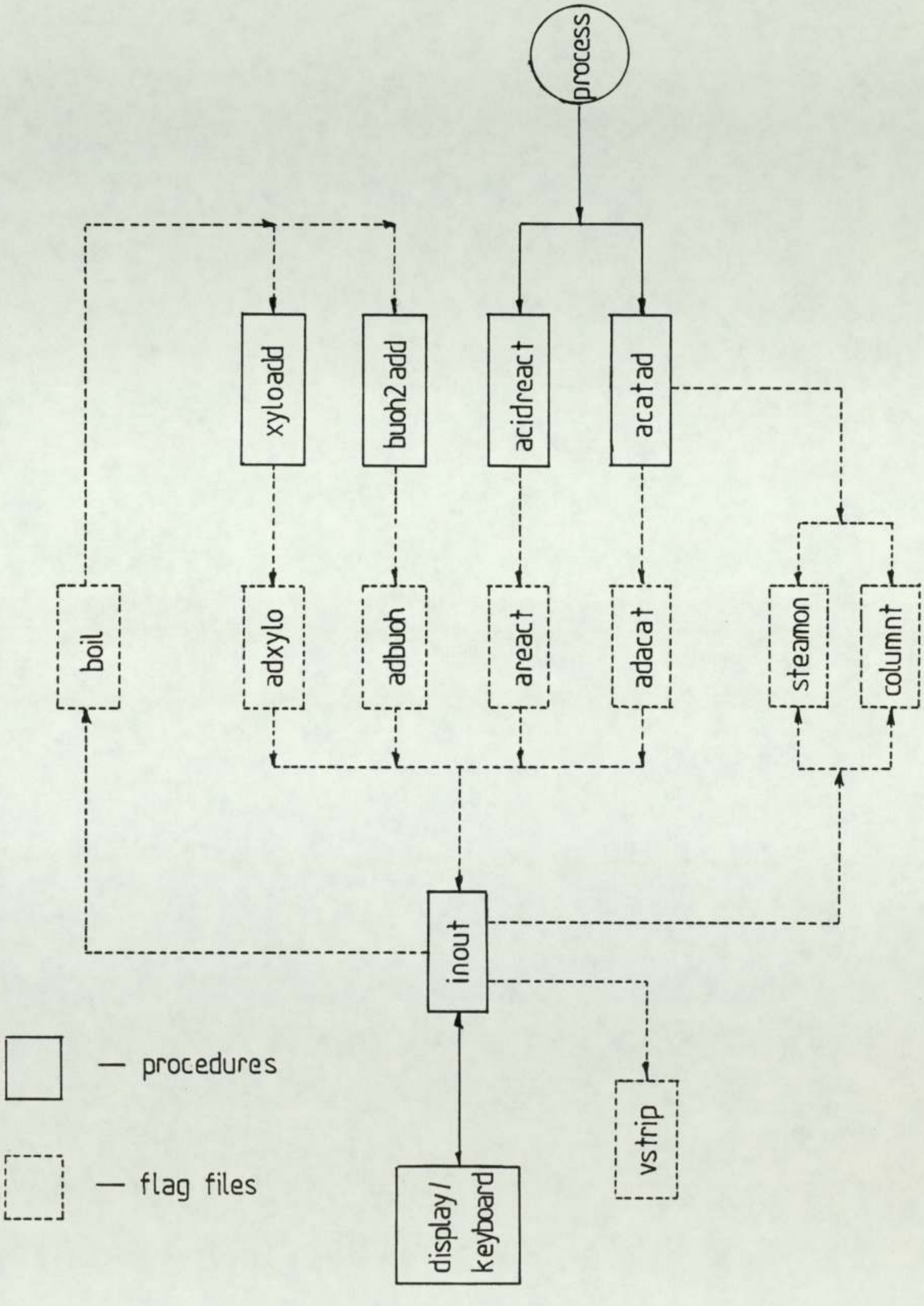
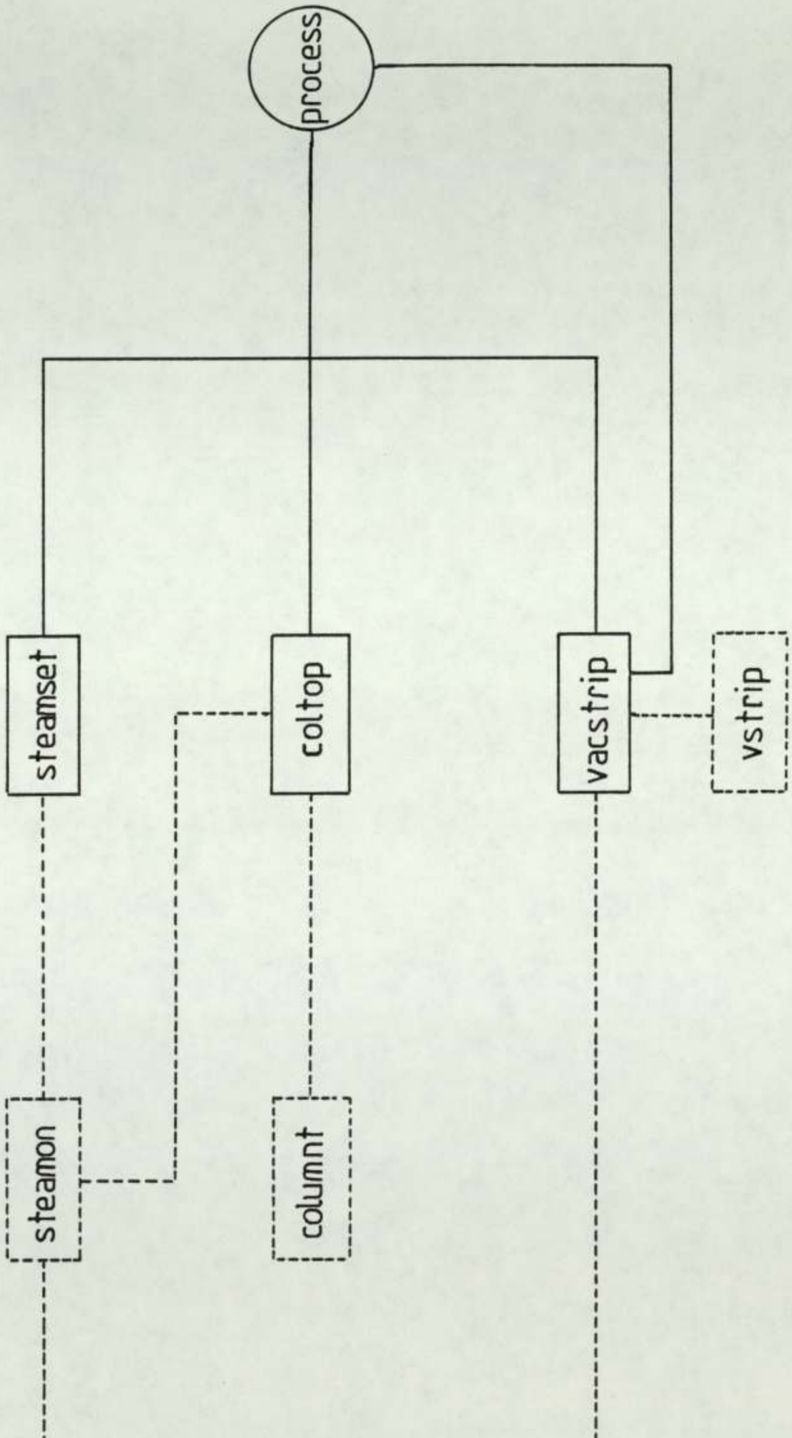


Figure 4·5 Procedure/Flag File Interaction for Monitoring and Control Package (cont.)



The digital values were again determined from the standard processing conditions. The value obtained was then used to adjust the supervisory set-point on the column top temperature 3 - term controller to the desired value at that particular time.

4.4.3 Procedure VacStrip

This procedure operated only during vacuum stripping. The purpose of this procedure was to control the removal rate of the wet solvent at reaction temperature by controlling the vacuum. It also controlled the total volume removed. The basis for this procedure was provided by trials carried out at BIP(19) on vacuum stripping the wet solvent under manual control at the reaction temperatures on a full scale plant. This work generated a set of time versus vacuum steps which were used by the procedure. The data was stored in a data file which was accessed by the procedure and the information used to send a series of values to the digital analogue converter. This increased the vacuum in steps over a period of time. While the vacuum was being increased, the procedure monitored the distillate temperature and wet solvent flowrate. The control action was that if these were found to drop then the steam was switched on at a low pressure, and therefore flow, to bring the distillate temperature and flowrate back up. If the distillate temperature increased above a certain value, then the vacuum was held, delaying the next step until the temperature fell back into the required range.

This procedure operated until the required amount of wet solvent was removed. The use of this procedure will save time and energy vacuum stripping the resin since it will not require to be cooled down and then re-heated during this process.

Appendix 7 gives a table of the steps of time and vacuum for the vacuum stripping stage.

4.5 Computer Control - Plant Trials

The trials were carried out in two stages. The first was to test and debug the software including the new control procedures in a real time environment. The second stage was to conduct full control trials to verify the control strategy and computer system in production situations.

4.5.1 Software Testing and Debugging

A series of proving trials were carried out on batches of resin being manufactured on the test reactor. These trials provided similar temperatures and aqueous distillate weights to those of the resin family being investigated. Throughout these trials the set point controllers were set to local, which meant that the plant operators were in control. This did not affect the adjustment of the remote set point values by the computer and these were observed throughout the production cycle to check that the computer was taking the correct action at the desired points, but without the signal being used to control the plant. The local control loops were observed to be well tuned and responded to manual changes in set points.

During these trials several minor errors were detected in the software at various stages in the production cycle. These were easily corrected. One major problem found at this stage concerned a conflict between procedures during disc accessing operations to read flag files. This was progressively resolved by re-setting program priorities on the less critical procedures and this reduced the frequency with which the discs were accessed.

Once these problems had been corrected a final test was carried out which proved that the software and hardware operated correctly at the various desired points in the production cycle.

4.5.2 Computer Control Trials

The next stage after completing the software trials was to carry out a series of full computer control trials. To prove the system, two trials were conducted on the reactor system using dry butanol batch charges for the resin family under investigation. This simplified the problem of water analysis.

The first trial was carried out on a type C resin. The set point controllers were set to remote so that the computer was controlling the plant on-line. Once material charging was completed and acknowledged the computer proceeded to monitor the reaction and make adjustments to the set points, whenever these were required during the reaction. The local loops tracked these changes correctly. The actions taken by the software were all correct based on the values received through the analogue to digital converter and the program logic. One problem occured during the acid reaction stage of the process in which spurious electrical noise affected the aqueous tank weight value, due to the signal passing through an industrial area of high electrical noise. The software interpreted this as a genuine signal, and assumed that the end of this stage had been achieved, and initiated the shutdown procedures needed prior to vacuum stripping. The fault of such erroneous input had not been previously experienced.

The cause of this signal noise was found to have been a bad earth connection on the cable shielding. This was rectified and tested before the second trial. The software was also modified to incorporate a routine to detect any spurious peaks in the digital value from this instrument and smooth the value. The software for this is given in the appropriate sections of Appendix 3.

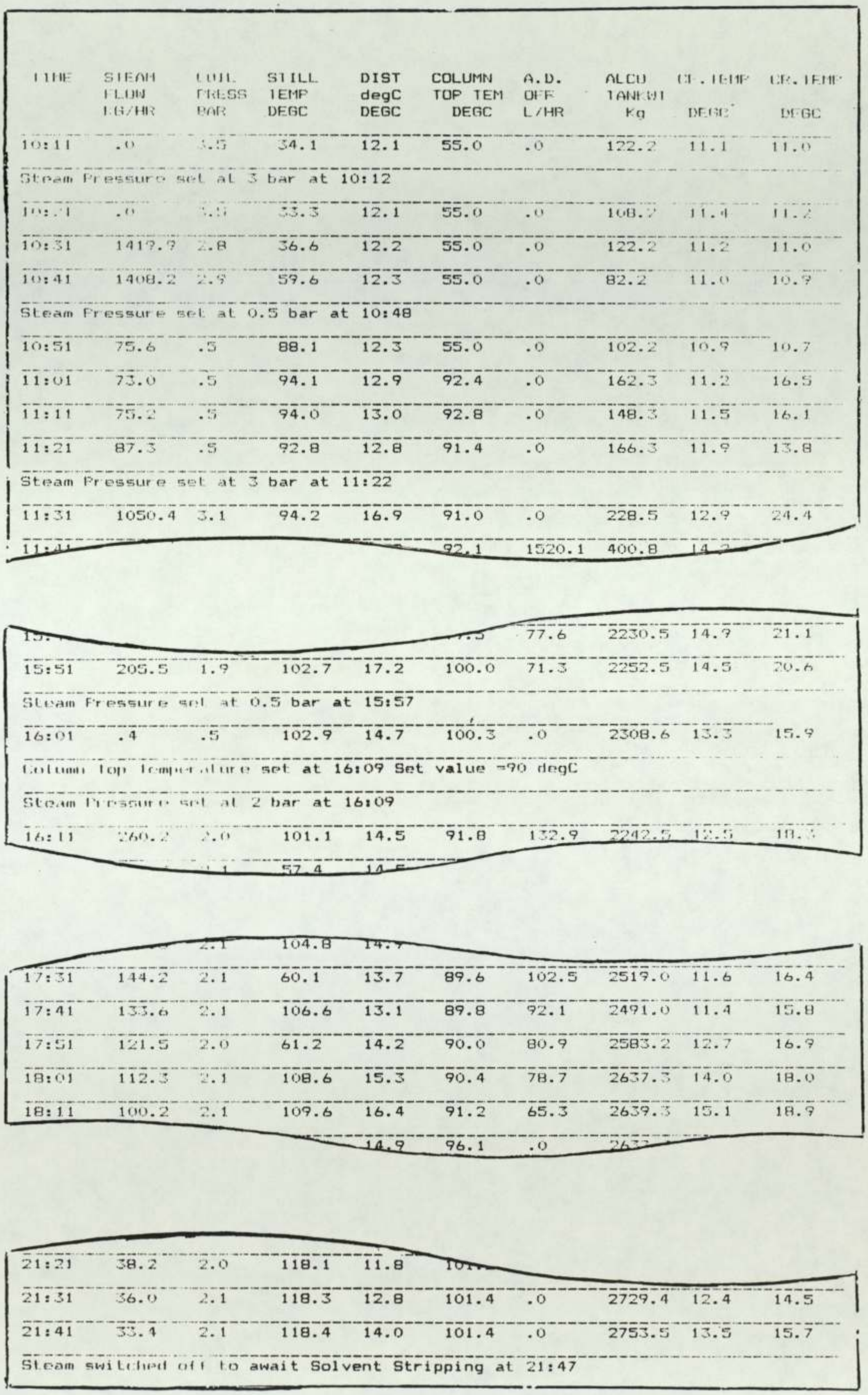
The second trial was carried out on a type B resin. The process proceeded correctly and the software operated successfully all the way through to the end of the acid reaction stage which marked the successful completion of the production cycle. This was in contrast to the previous trial which failed after the dehydration stage. Log-sheets for these two trials are given in Appendix 5 and extracts are given in Figure 4.6

The vacuum stripping of the resin under computer control was not carried out during these trials due to a failure of the distillate temperature recorder. The value from this instrument was required to prevent boil over occurring in the reactor and therefore no trials on this could be conducted while this was out of commission.

4.6 Discussion of Computer Control Trials

A series of control trials have been carried out using the computer system in conjunction with analyser units. These have shown that the control parameters which have been identified can be used to determine the end points at various stages in the production cycle. For this the computer must receive information in an accurate and repeatable form from the plant instruments and analysers. The importance of accuracy and reliability of this information was shown on three occasions during these trials. The first case concerned the accuracy of the aqueous distillate weight being received by the computer. The value eventually received by the software had passed through two conversions, pneumatic to electrical and then electrical to digital. Smoothing routines based on a ten point running average were installed in the software to improve the precision of this value. This could not eliminate the problem of an inaccurate value due to earthing failure. This was overcome by further logical analysis of the digital value

Figure 4·6 Extracts from Logsheet for Computer Control Trial



```
TIME   STEAM    COIL    STILL    DIST   COLUMN   A.D.     ALCU    CF.TEMP  CR.TEMP
       FLOW     PRESS   TEMP     degC   TOP TEM  OFF      TANKWT
       KG/HR    BAR     DEGC     DEGC     DEGC   L/HR      Kg      DEGC     DEGC
-----------------------------------------------------------------------------------
10:11   .0      3.5     34.1     12.1   55.0     .0       122.2   11.1     11.0
-----------------------------------------------------------------------------------
Steam Pressure set at 3 bar at 10:12
-----------------------------------------------------------------------------------
10:21   .0      3.5     33.3     12.1   55.0     .0       108.2   11.4     11.2
-----------------------------------------------------------------------------------
10:31   1419.9  2.8     36.6     12.2   55.0     .0       122.2   11.2     11.0
-----------------------------------------------------------------------------------
10:41   1408.2  2.9     59.6     12.3   55.0     .0       82.2    11.0     10.9
-----------------------------------------------------------------------------------
Steam Pressure set at 0.5 bar at 10:48
-----------------------------------------------------------------------------------
10:51   75.6    .5      88.1     12.3   55.0     .0       102.2   10.9     10.7
-----------------------------------------------------------------------------------
11:01   73.0    .5      94.1     12.9   92.4     .0       162.3   11.2     16.5
-----------------------------------------------------------------------------------
11:11   75.2    .5      94.0     13.0   92.8     .0       148.3   11.5     16.1
-----------------------------------------------------------------------------------
11:21   87.3    .5      92.8     12.8   91.4     .0       166.3   11.9     13.8
-----------------------------------------------------------------------------------
Steam Pressure set at 3 bar at 11:22
-----------------------------------------------------------------------------------
11:31   1050.4  3.1     94.2     16.9   91.0     .0       228.5   12.9     24.4
-----------------------------------------------------------------------------------
11:4                                    92.1     1520.1   400.8   14.
```

```
15:                                              77.6     2230.5  14.9     21.1
-----------------------------------------------------------------------------------
15:51   205.5   1.9     102.7    17.2   100.0    71.3     2252.5  14.5     20.6
-----------------------------------------------------------------------------------
Steam Pressure set at 0.5 bar at 15:57
-----------------------------------------------------------------------------------
16:01   .4      .5      102.9    14.7   100.3    .0       2308.6  13.3     15.9
-----------------------------------------------------------------------------------
Column Top Temperature set at 16:09 Set value =90 degC
-----------------------------------------------------------------------------------
Steam Pressure set at 2 bar at 16:09
-----------------------------------------------------------------------------------
16:11   260.2   2.0     101.1    14.5   91.8     132.9    2242.5  12.5     18.3
-----------------------------------------------------------------------------------
                2.1     57.4     14.
```

```
                2.1     104.8    14.
-----------------------------------------------------------------------------------
17:31   144.2   2.1     60.1     13.7   89.6     102.5    2519.0  11.6     16.4
-----------------------------------------------------------------------------------
17:41   133.6   2.1     106.6    13.1   89.8     92.1     2491.0  11.4     15.8
-----------------------------------------------------------------------------------
17:51   121.5   2.0     61.2     14.2   90.0     80.9     2583.2  12.7     16.9
-----------------------------------------------------------------------------------
18:01   112.3   2.1     108.6    15.3   90.4     78.7     2637.3  14.0     18.0
-----------------------------------------------------------------------------------
18:11   100.2   2.1     109.6    16.4   91.2     65.3     2639.3  15.1     18.9
-----------------------------------------------------------------------------------
                                 14.9   96.1     .0       263
```

```
-----------------------------------------------------------------------------------
21:21   38.2    2.0     118.1    11.8   101.
-----------------------------------------------------------------------------------
21:31   36.0    2.1     118.3    12.8   101.4    .0       2729.4  12.4     14.5
-----------------------------------------------------------------------------------
21:41   33.4    2.1     118.4    14.0   101.4    .0       2753.5  13.5     15.7
-----------------------------------------------------------------------------------
Steam switched off to await Solvent Stripping at 21:47
```

within the software. It would have been preferable to have information which was both precise and accurate. This could be achieved with a sensor which was electrically based, generating either a direct or digital signal or a voltage value, and connected to the computer.

The other two instances concerned the robustness of plant instruments and in particular concerned the Bendix 1800 Viscometer, Section 3.10, and the distillate temperature recorder. In both cases certain operations were unable to be carried out because of the failure of these instruments to perform to requirements. In the case of the viscometer, it was intended that this would be used to provide a trend display available throughout the production cycle so that any sudden rise in viscosity could be detected. Although not a control parameter, it would have provided a safety feedback to the operator and allowed for manual testing to be eliminated.

The failure of the distillate temperature recorder prevented the final stage of the resin adjustment from being carried out under computer control. This would have been based on the series of stepwise reductions in vacuum previously determined manually.

All three cases have shown the need for instruments not only to be reliable but also to be in a form in which the instrument can be easily interfaced to external sources so that it sends an accurate and precise value to the computers' input devices. Precision can be lost passing through a series of conversions.

4.7 Conclusion of Computer Trials

It can be concluded from this series of computer control trials that the production of butylated melamine formaldehyde resins can be controlled by a computer using the parameters of temperature, time and mass of aqueous distillate removed. For this it is necessary that the data from the relevant plant instruments should be reliable accurate and reproducible. The trials have also shown the need for instruments which are to be installed in a reactor be of a suitably robust nature.

5. GENERAL DISCUSSION AND CONCLUSION

5.1 General Discussion

The work reported in this thesis covers the analysis of recovered solvents and process control of a resin production plant. These provide insights for the development of a comprehensive strategy for control of the production of synthetic resins. A possible strategy and the benefits of introducing it are discussed in detail in this chapter.

5.2 Recovered Solvent Analysis

In Chapter 3 several methods were investigated for carrying out the on-line analysis of the recovered solvents. The most practical methods were determined to be a dual system comprising of an Ultra-Violet Analyser and a Density Measuring Unit. The installation of this system would provide the basis for the implementation of an accurate computer and process control strategy. These instruments were demonstrated to function correctly, but were not available during the process control investigation.

5.3 Process Control

The trials conducted on the plant have shown that monitoring the parameters of time, reactor temperature and mass of aqueous distillate removed from the system provides a means of accurately controlling the overall reaction to the desired end point. For this it is necessary to know the mass of aqueous distillate to be removed. This can be either by the use of pure solvent charges or using an accurate method for determining the recovered solvent composition, as described in Chapter 3. In the use of the above parameters, it has been found that the most important factor is the reliability and accuracy of the instruments used.

The next requirement is to transfer these values to the computer. This was carried out via an analogue to digital converter by the computer system installed on the plant. The converted value for the aqueous distillate mass became imprecise due to the series of conversions that the value passed through, with a high precision analogue to digital converter sampling a lower precision pneumatic to electrical converter. To eliminate this, instruments should be selected which generate an electrical analogue signal which can easily be converted by the computer into a digital signal. A better method would be to use an instrument which generated a digital signal which can be sent directly to the computer, therefore eliminating any conversion errors.

Once this information was received by the computer in a usable form it was required to be stored on floppy discs which had associated problems of accessing time. This could be improved or eliminated by using other technology for data storage such as hard discs or a Virtual RAM driver which allows a portion of the systems memory to be used as a disc drive.

The processing of control information by the software was required in time for control action to be taken. It was necessary to pay attention to the scheduling of the procedures due to the time sharing of the central processor by the operating system, to ensure correct action. It is important that the operating system chosen allows these priorities to be changed while the control system is commissioned and tested. Higher capacity could be obtained by the application of additional hardware such as a second processor to handle the input and output operations for the computer. This along with the use of more sophisticated software would also improve the work load of the processor within the OS-9 multi-tasking operating system. Undoubtedly, advances in available computer hardware mean that a more powerful system for these tasks could easily be assembled.

The presentation of the processed information was made to the operator through a second computer running a graphics program. This did not allow for the effective interaction between the graphics displays and the main monitoring and control software on the SEED computer. A more effective configuration would have been a system which supported its own graphics, eliminating the need for a dual terminal system. Again with the advances in computer graphics displays, a suitable system could be found and installed.

5.4 Software

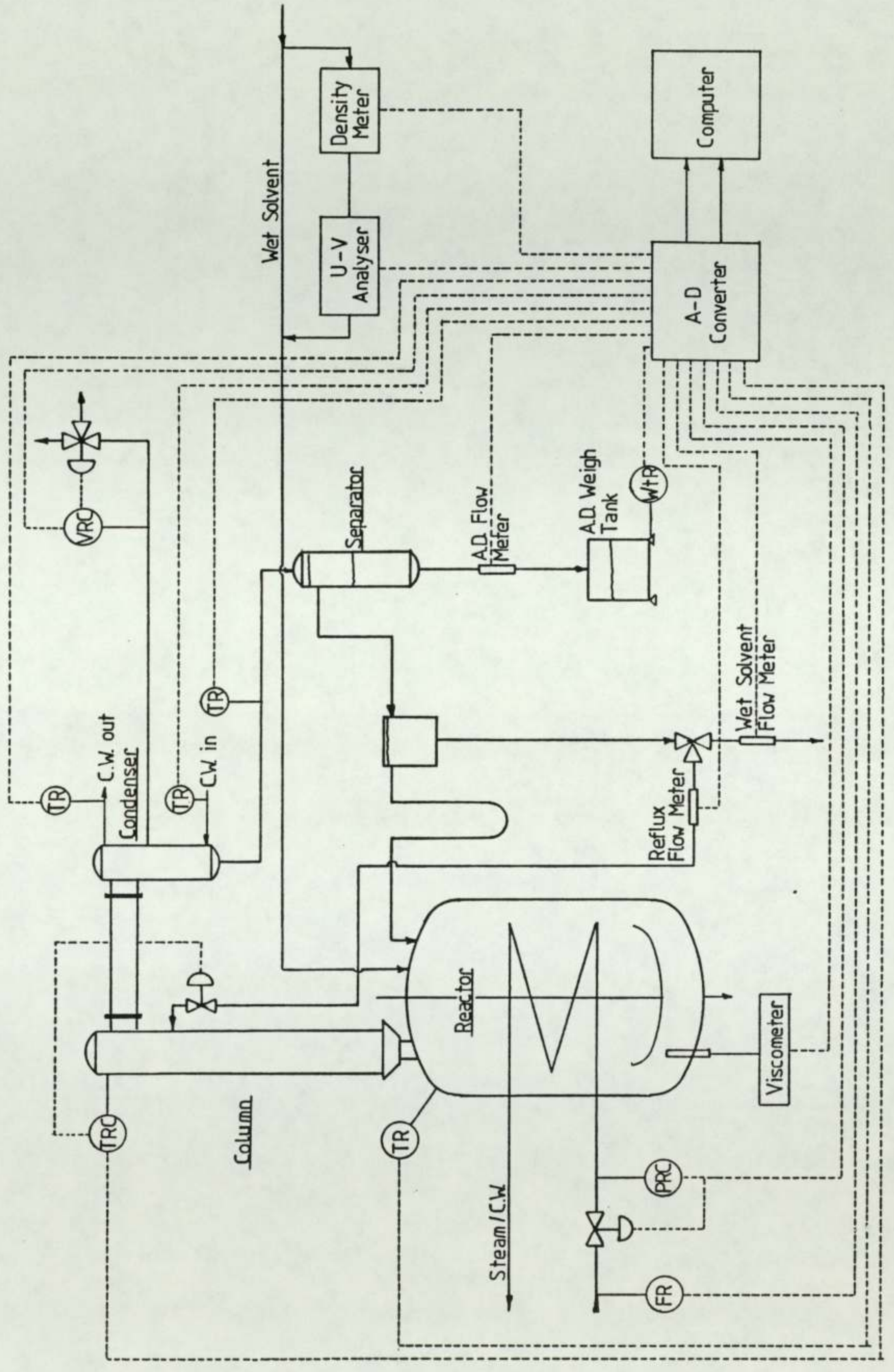
The use of a modular format for the control software proved to be flexible and allowed for the progresseive development of the control strategy. As new instrumentation and computer hardware was installed, software could be written and operated by the system so that it could be finely tuned without affecting the procedures already in operation.

The software written for use with this system required a degree of operator interaction to inform the software when certain actions had been taken such as materials charging, the switching on of heating or cooling, or the results of solvent analysis. In a more fully automated system these actions would be taken and monitored by the computer with the operator being notified of these through a suitable output device. The operator would be notified of any actions required should an unusual condition or emergency occur.

5.5 System Outline

The work discussed in this thesis is best illustrated by Figure 5.1 which shows in general terms the essential requirements for bringing about computer control for the production of synthetic resins. This requires the analysis of the recovered solvents being fed to the reactor so that the quantitites of extra water and xylol being added are known. Another requirement is the monitoring of the key control parameters of reactor temperatures and mass of aqueous distillate with reaction time. There is also a need to monitor the product viscosity in order to prevent over condensation. This requires a more dependable instrument than that currently evaluated becoming available. As well as these requirements, in order to make the system more automated it is necessary

Figure 5·1 Plant Instrument Modifications



for the computer to operate valve actuators and receive feedback from proximity switches indicating the valve position. This would mean a larger input capacity on the computer system than that installed for the trials. It also means that the instrumentation available on the plant must be upgraded to meet the more stringent demands which would be made by overall computer control of this process. This is in order for the computer software to base its control actions on precise information received from the plant sensors.

5.6 Computer Control Benefits

The benefits of computer control can be summarised by the following; the implementation of a control system would lead to an improvement in the consistency of the resins produced, reducing the batch quality variations by accurately determining the reaction end points using the new control parameters. Currently resin batches fall outside the specification range, which then requires a rework procedure to be carried out. The estimated benefits can be up to 10% of the production costs if the need for rework is reduced or removed altogether. The use of a computer system removes the need for offline sampling and testing which requires a degree of subjective judgement in determining the results by the testers and causes delays. It would allow for more frequent monitoring of plant instruments in order to detect any variations which may affect the final product, essentially the control of the process will be more definitive. There would also be the facility for providing more management information especially in the cases where abnormal conditions arise during processing.

5.7 Conclusions and Recommendations for Further Work

1) The measurement of the parameters; reactor temperature, mass of aqueous distillate removal and time have been shown to be a practical means of determining the end points in the reactions which produce butylated resins on a full scale production facility.

2) The use of a computer system to monitor and control the production of synthetic resins using the new control parameters has been proved to be practical by the use of an experimental system. A suitable Computer Control System could be installed on the batch reactors using these control parameters to monitor the process. The implementation should be decided on the basis of an economic evaluation.

3) Two methods have been determined which would give the butanol, water and xylol mixture compositions in the recovered solvents. The first is Gas-Liquid Chromatography which would give a complete breakdown of the solvent streams composition but only for one sample during the solvent charging operation. The second method is a dual system comprising of an Ultra-Violet analyser giving the xylol content of the stream and operating in conjunction with a density meter from which the water and butanol compositions

can be determined. This dual method would provide a continuous analysis of the solvent stream. Investigations should be continued to determine if there are any further methods available for determining the composition of the recovered solvents with a view to implementation after an economic evaluation.

4) The Bendix 1800 Viscometer has proved not to be a durable and reliable instrument when installed in a full scale production reactor where the agitation conditions are more severe. Further investigations and trials should be made using this viscometer to determine if there are any means by which it can be made more robust and reliable. Alternatively, other methods for determining the product viscosity in the reactor should be investigated.

5) Investigations should be made into the possibility of incorporating a mathematical model into the control system in order to provide an on-going check against the ideal conditions for a particular stage in the reaction. This will depend upon other work to understand the reaction mechanisms more exactly.

APPENDIX 1

The Teaching Company Scheme

TEACHING COMPANY SCHEME

This Scheme aims to link Universities and Polytechnics with manufacturing industry in order to raise industrial performance and use academic knowledge and capacity more effectively. Advanced technology developed in Universities can be used to improve manufacturing and industry methods. Selected able graduates can be trained for industrial careers. Existing company and academic staff can be re-trained and their skills refreshed by personal contact with the academics. Conversely academic staff gain by the direct association with industry finding ideas for research and an updated background for their teaching.

The Scheme operates through Teaching Company Programmes in which a University or Polytechnic participates with an Industrial Company in an individually structured programme. The company management should be committed to change and welcome academic collaboration in all technical and management activities at all levels.

The permanent academic staff joining the Scheme are assisted by high calibre graduates recruited in consultation with the company for two year appointments as Teaching Company Associates. The associates are normally based full time with the company and work in collaboration with company and academic staff on tasks within the programme. The University or Polytechnic arranges induction and continuing tuition courses according to personal and Programme needs. Associate appointments may lead to higher degrees but the main aim is that they should lead to posts in industry with substantial responsibility and reward.

The Schemes are funded partly by the Science and Engineering Research Council and the Department of Trade and Industry and partly by the industrial partner. Further support may be available through other DTI and SERC channels.

The benefits of the Scheme to the various parties involved are as follows :-

The Associates will learn by experience during their work with senior academic and industrial staff on real commercial tasks. This prolonged industrial experience will hopefully lead to improvements in the company's operations and to a greater confidence in the performance of the Associate. This will often allow the company to delegate a far greater responsibility with all the concurrent benefits to a future industrial career.

The Company benefits from the Academics' and Associates' contribution in applying advanced technology to its operational problems and from the recruitment of able Teaching Company Associates.

Finally, the Universities and Polytechnics can extend their research and post-graduate and post-experience teaching beyond the classroom and laboratory to operating companies with manufacturing facilities. The Schemes may lead to future consultancies or joint appointment responsibilities.

APPENDIX 2

Analogue - Digital Converters - Seed 19 Analogue Inputs
- Instrument Calibration Curves

SEED ANALOGUE INPUTS

CONNECTOR A 25 Pin Male 'D' Connector

Channel	Variable	Pin No.
0	Reactor temperature	1 +ve I/P 2 Grd
1	Alcoform Tank Wt	3 +ve I/P 4 Gnd
2	-	5 +ve I/P 6 Gnd
3	Steam Flow	7 +ve I/P 8 Gnd
4	Coil Pressure	9 +ve I/P 10 Grd
C	Wet Solvent Flow	17 +ve I/P 18 Grd
D	-	19 +ve I/P 20 Grd
E	-	21 +ve I/P 22 Grd
F	-	23 +ve I/P 24 Grd

+ve I/P - Positive Input

Grd - Ground

SEED ANALOGUE INPUTS

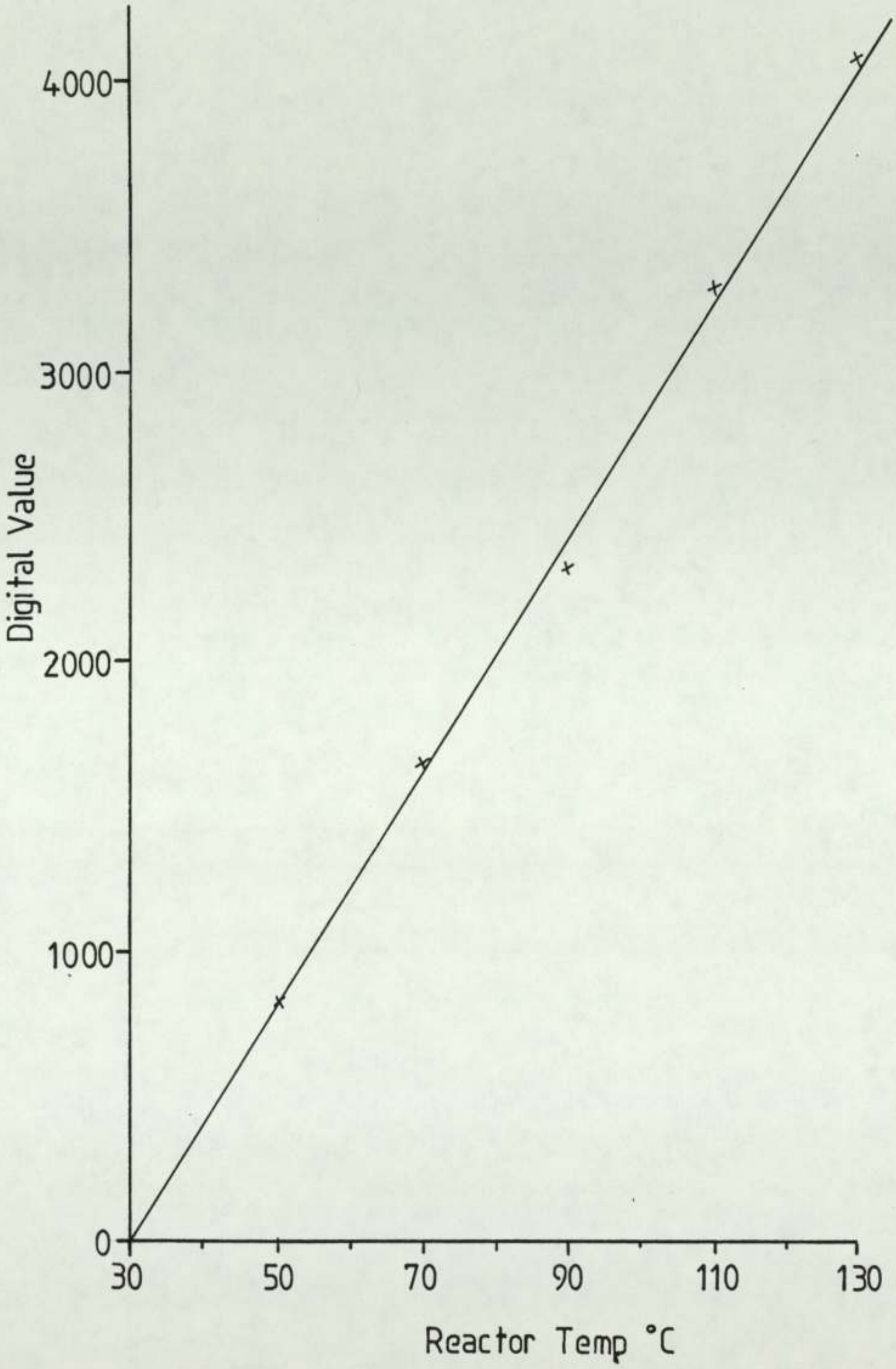
CONNECTOR B 25 Pin Male 'D' Connector

Channel	Variable	Pin No.
5	Cooling Water return Temperature	11 +ve I/P 12 Grd
6	Cooling Water Feed Temperature	13 +ve I/P 14 Grd
7	Vacuum	15 +ve I/P 16 Grd
8	Column Top Temperature	17 +ve I/P 18 Grd
9	Distillate Temperature	19 +ve I/P 20 Grd
A	Aqueous Distillate Flow	21 +ve I/P 22 Grd
B	Reflux Flow	23 +ve I/P 24 Grd

+ve I/P - Positive Input

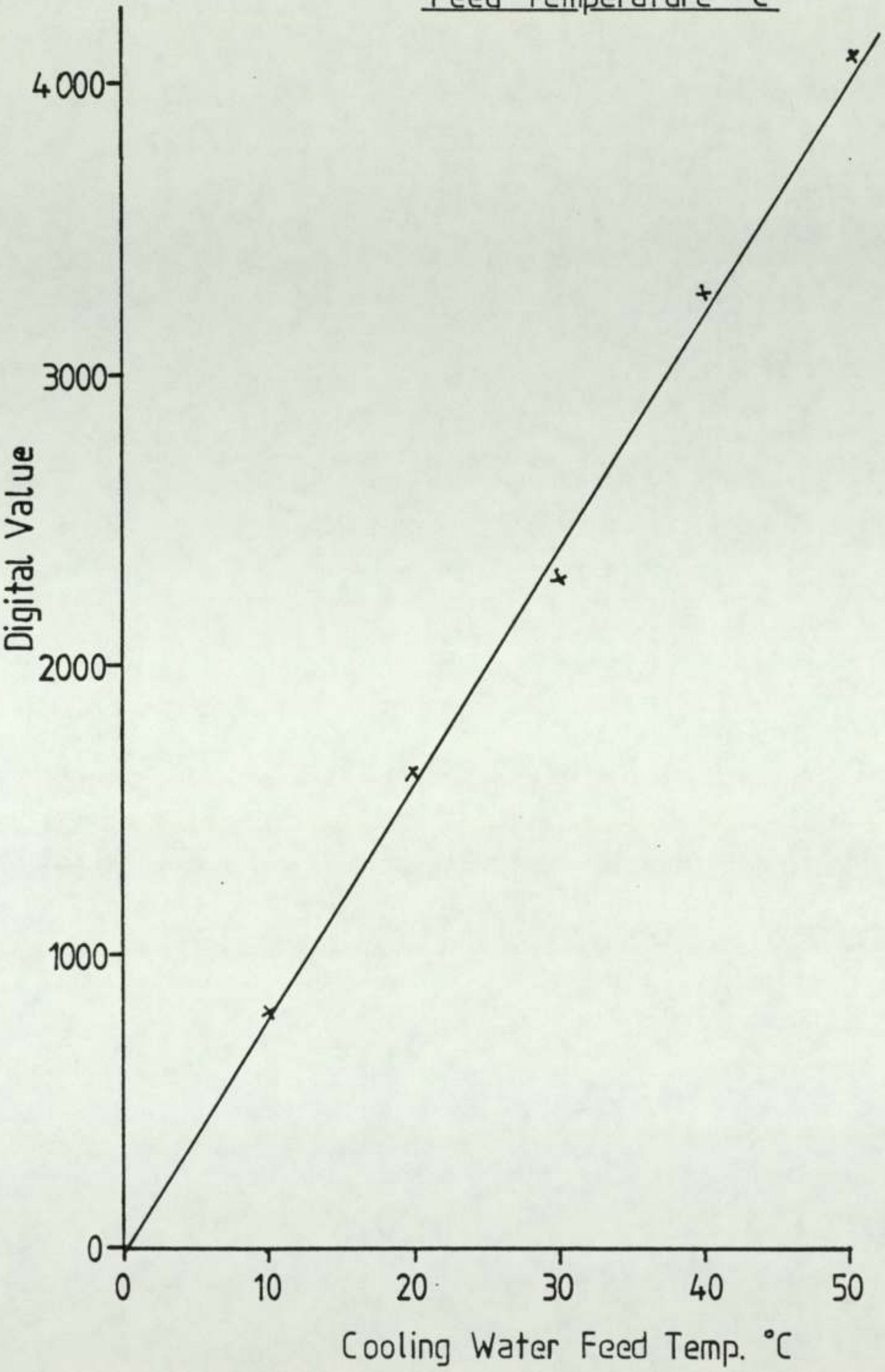
Grd - Ground

Figure A2·1 Calibration Curve for Reactor Temp. °C



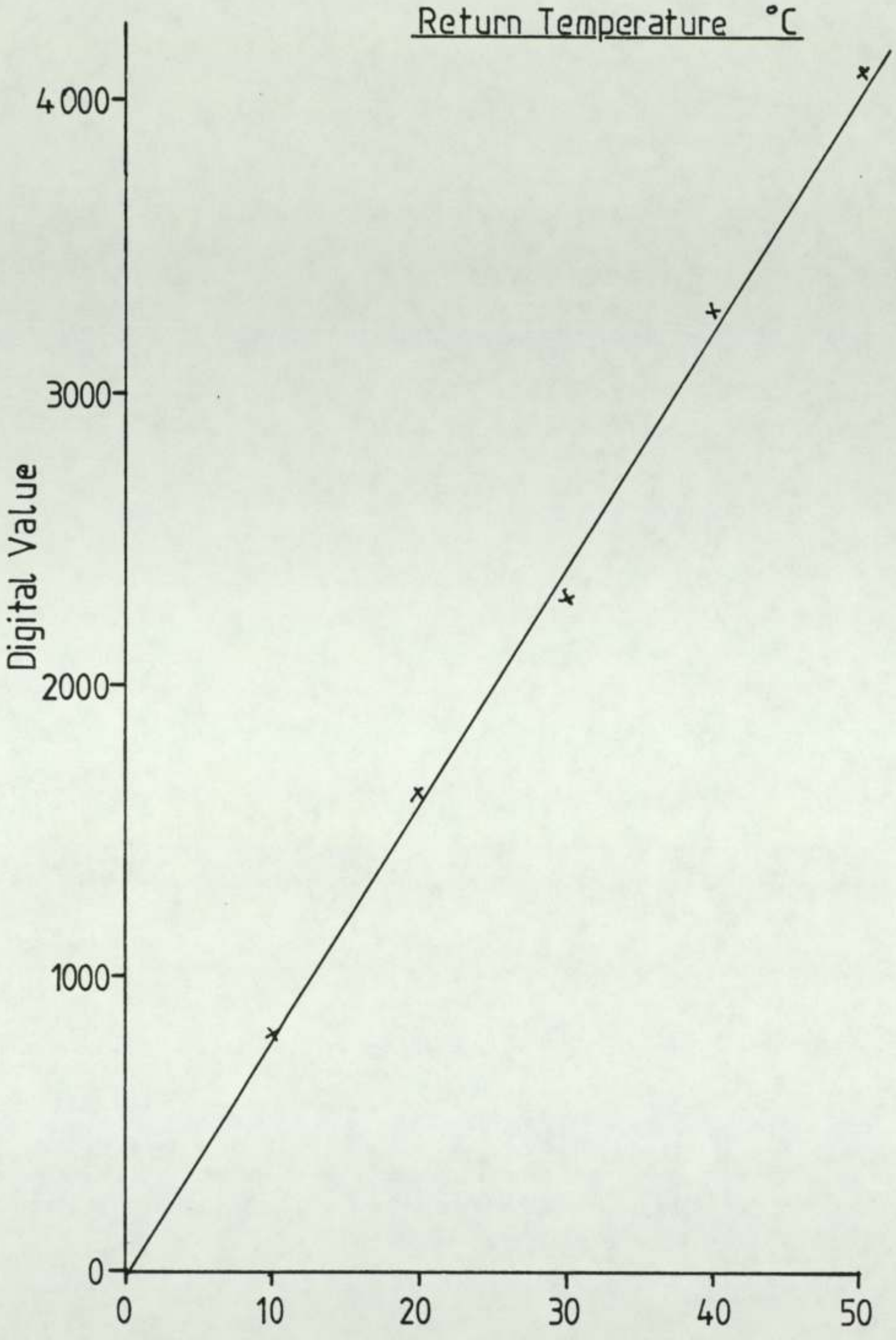
1/Gradient = $2{\cdot}452 \times 10^{-2}$

Figure A2·2 Calibration Curve for Cooling Water Feed Temperature °C



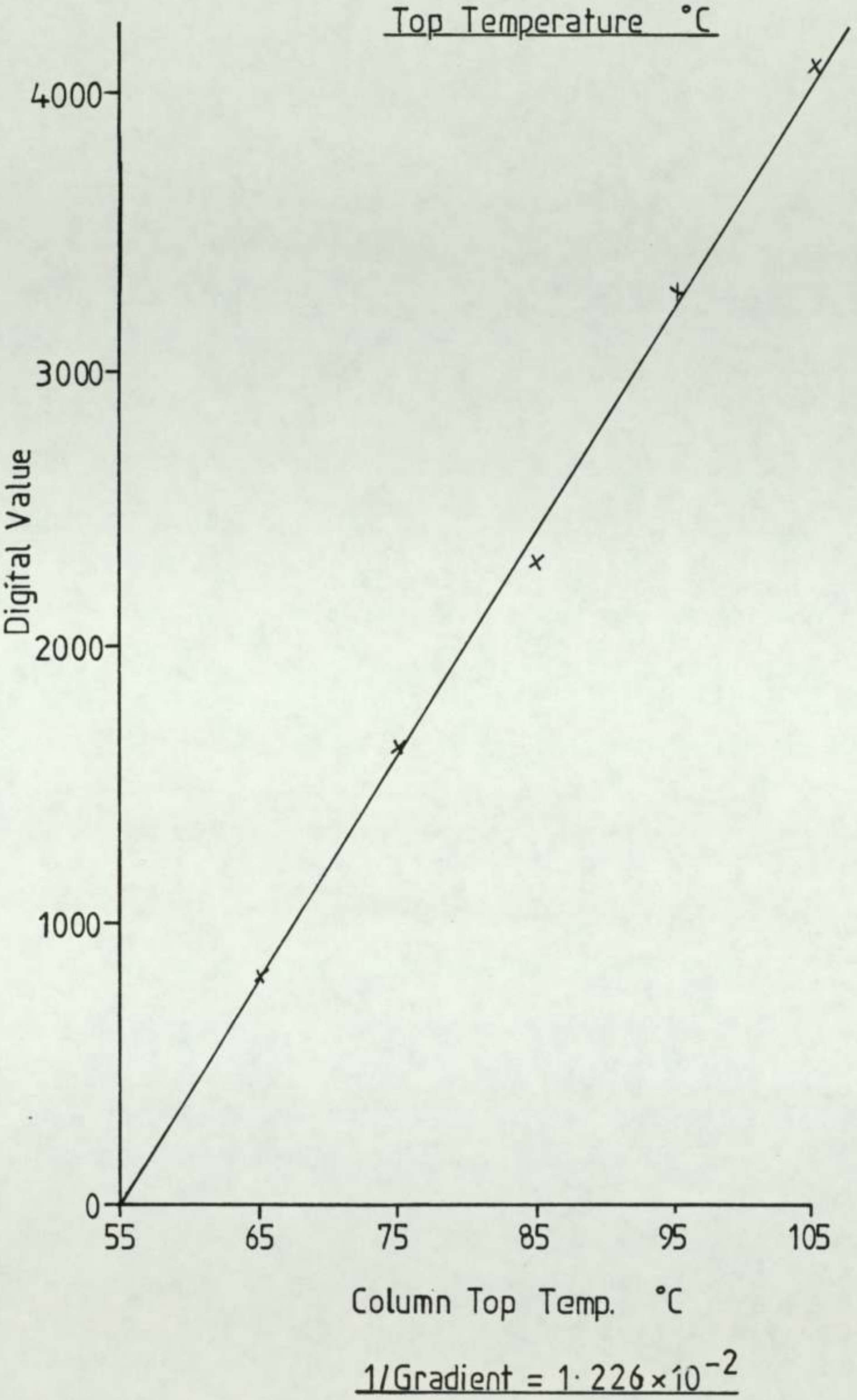
1/Gradient = $1 \cdot 226 \times 10^{-2}$

Figure A2·3 Calibration Curve for Cooling Water Return Temperature °C



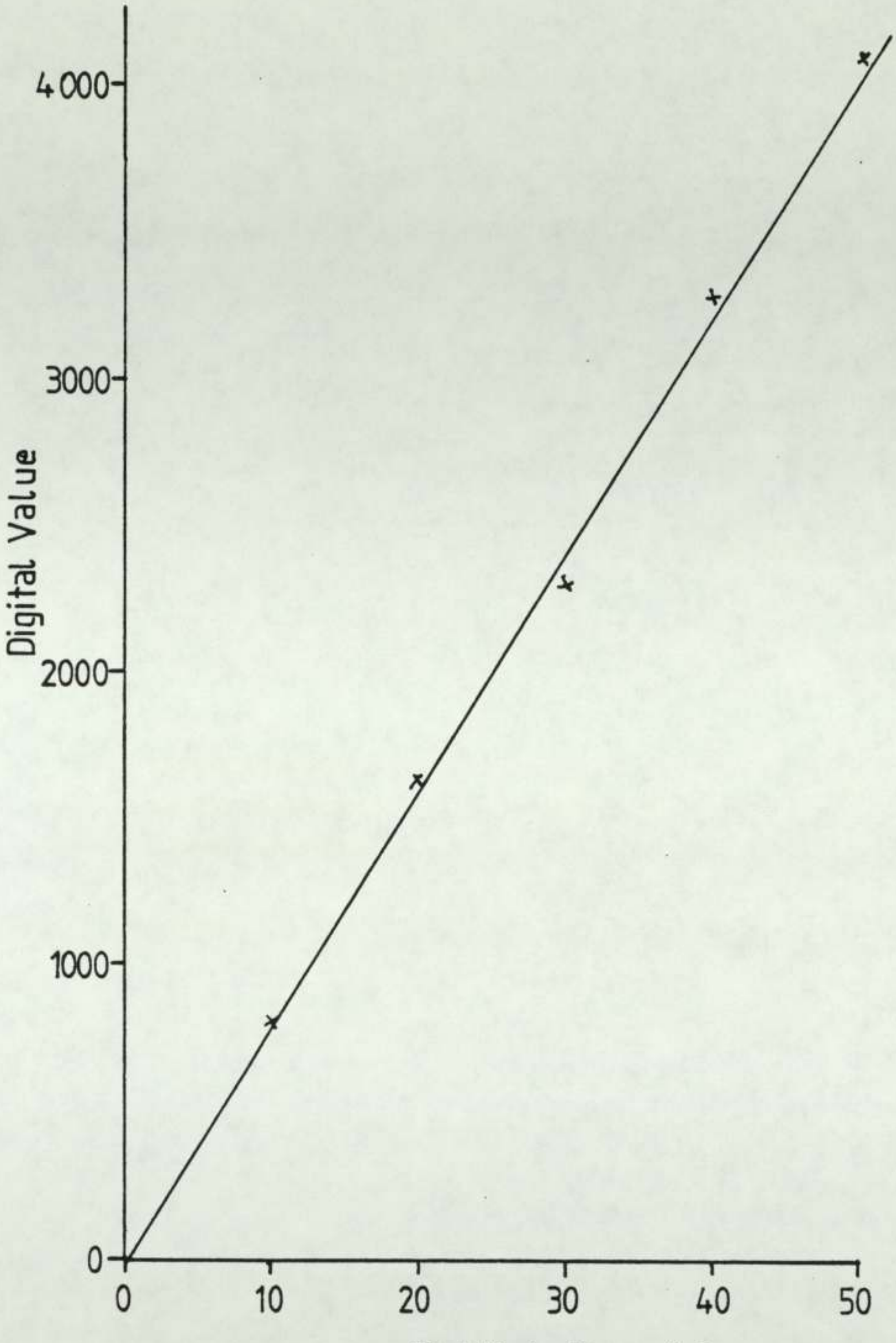
1/Gradient = 1·226×10^{-2}

Figure A2·4 Calibration Curve for Column Top Temperature °C



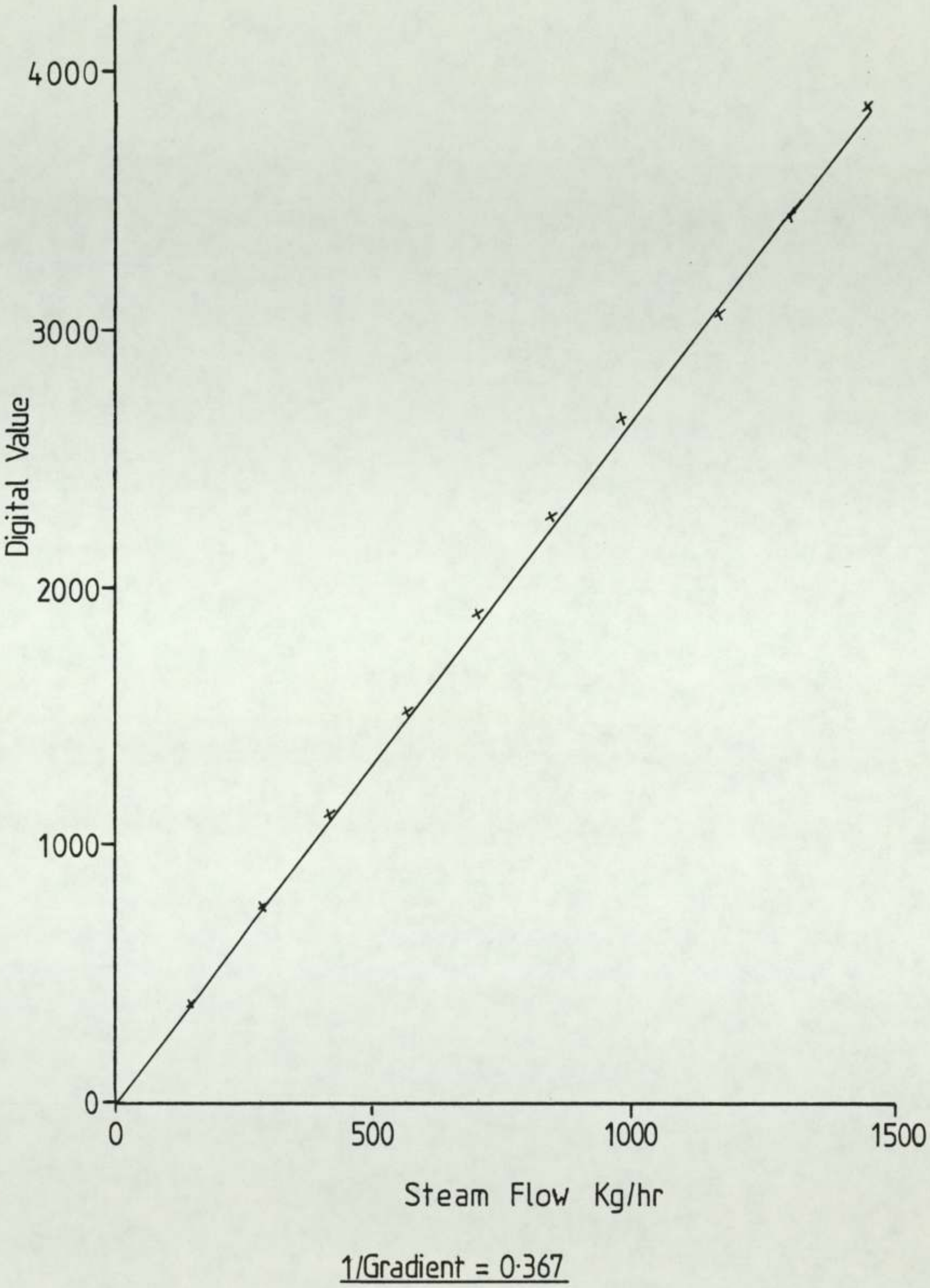
1/Gradient = $1\cdot 226 \times 10^{-2}$

Figure A2·5 Calibration Curve for Distillate Temperature °C



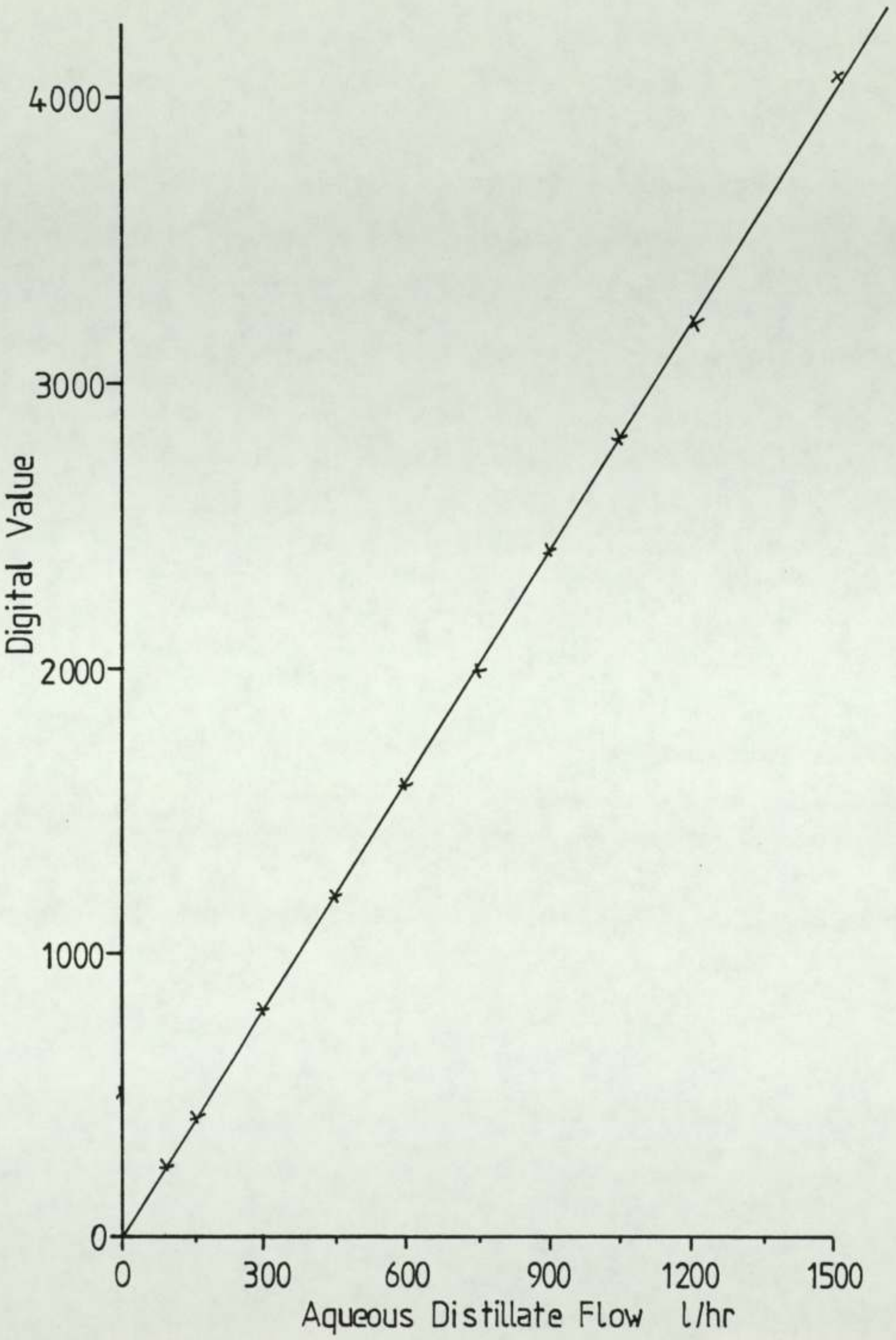
1/Gradient = $1{\cdot}226 \times 10^{-2}$

Figure A2·6 Calibration Curve for Steam Flow



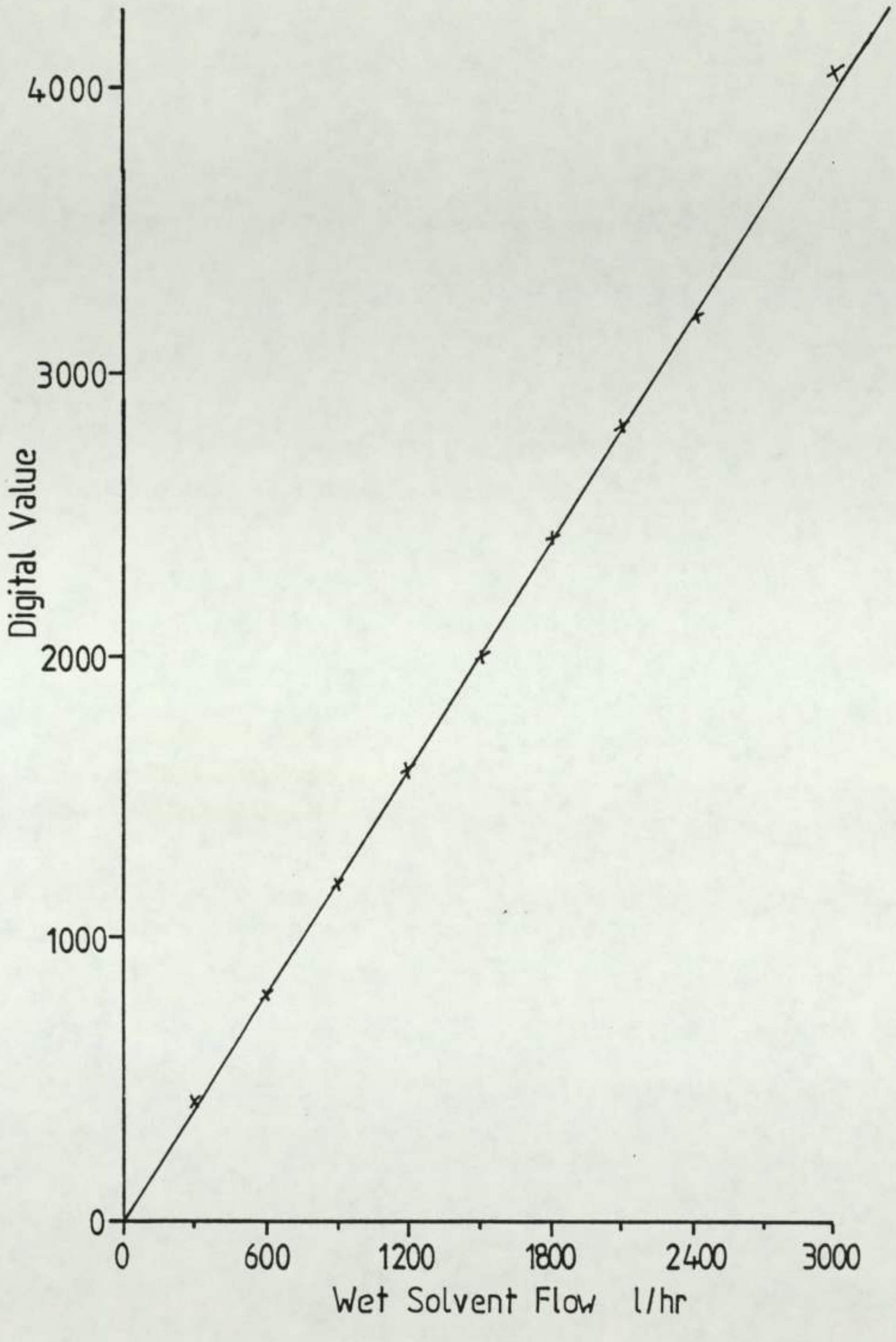
1/Gradient = 0·367

Figure A2·7 Calibration Curve for Aqueous Distillate Flow



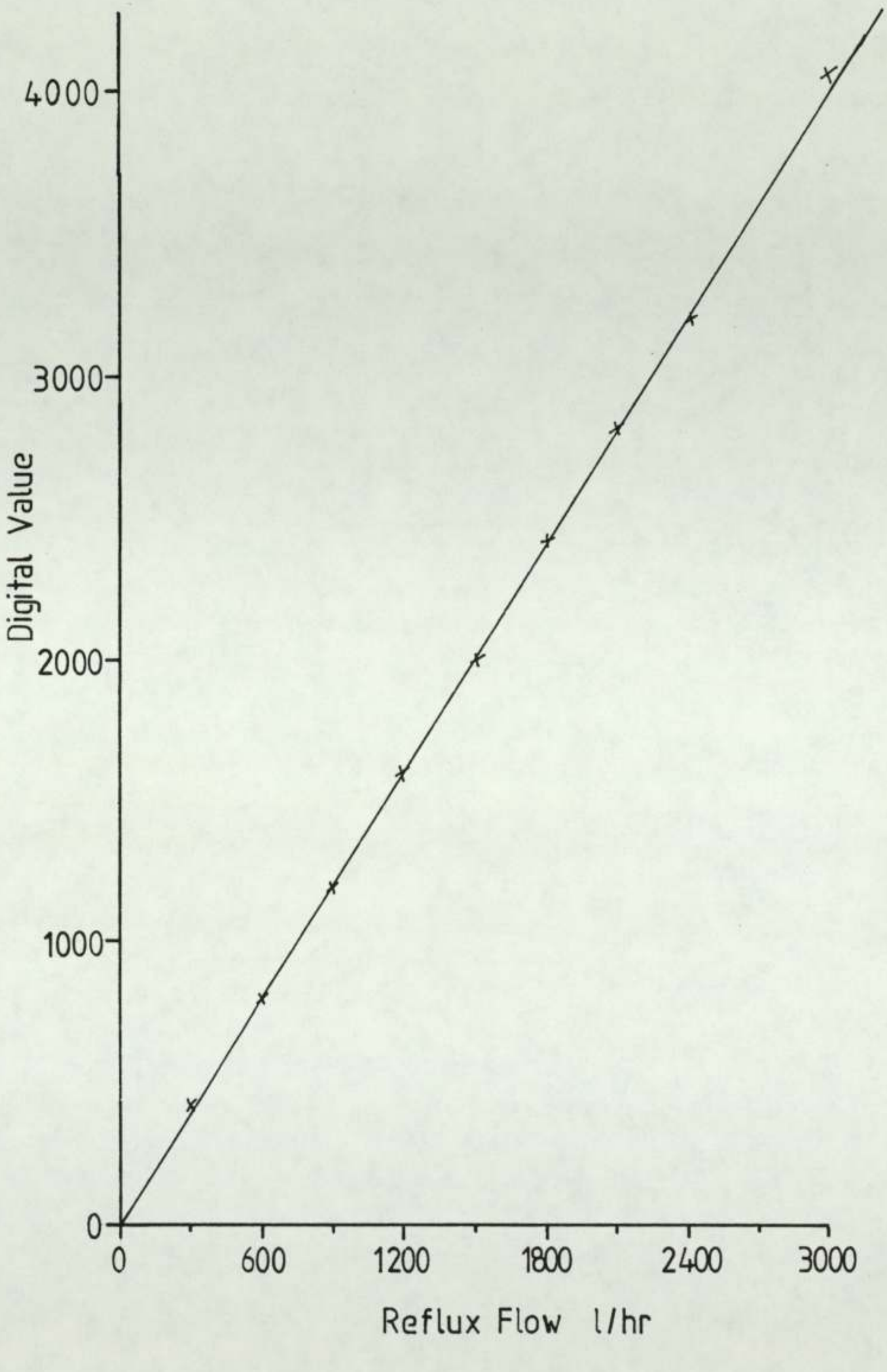
1/Gradient = 0·3712

Figure A2·8 Calibration Curve for Wet Solvent Flow



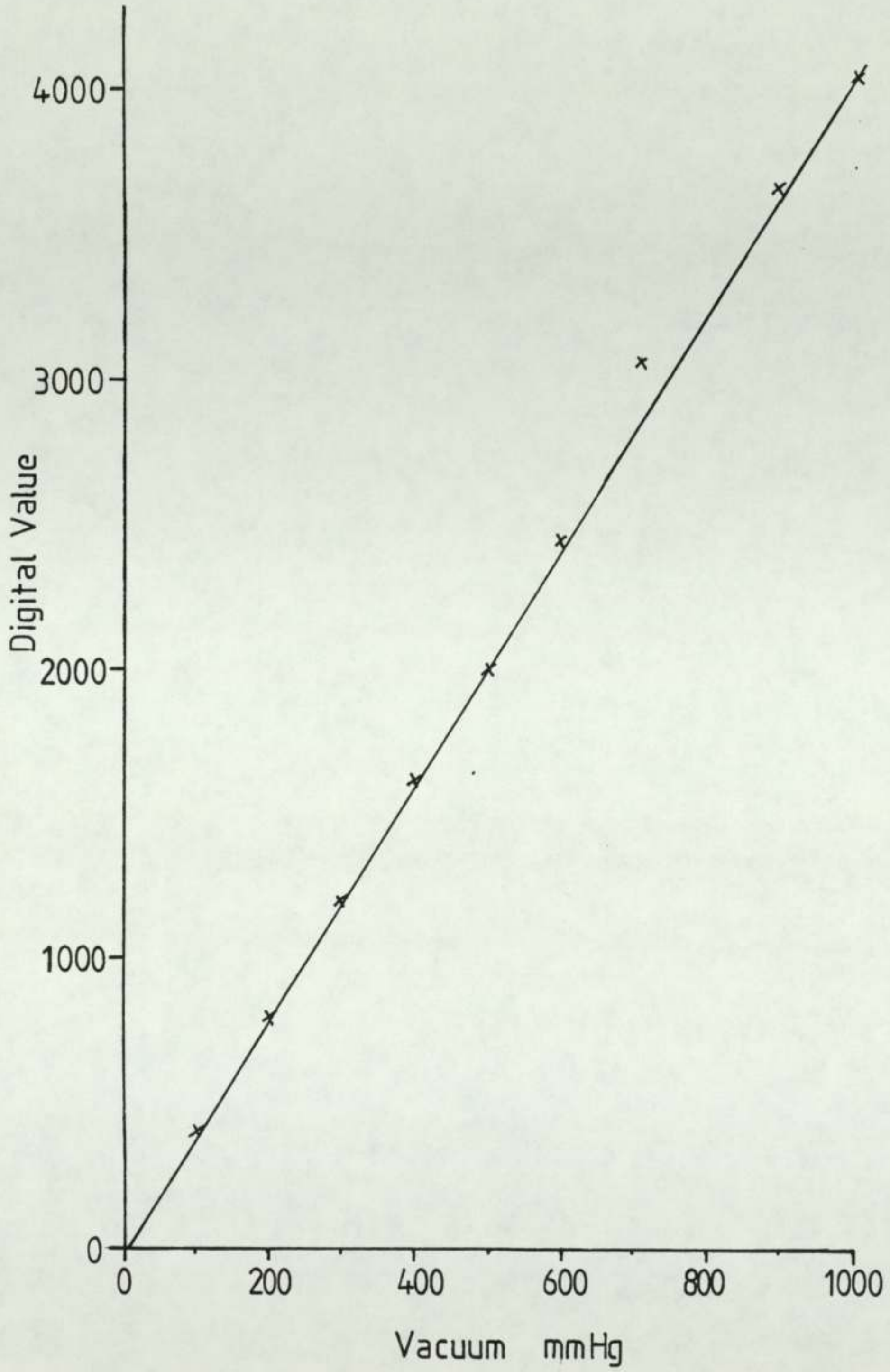
1/Gradient = 0·7424

Figure A2·9 Calibration Curve for Reflux Flow



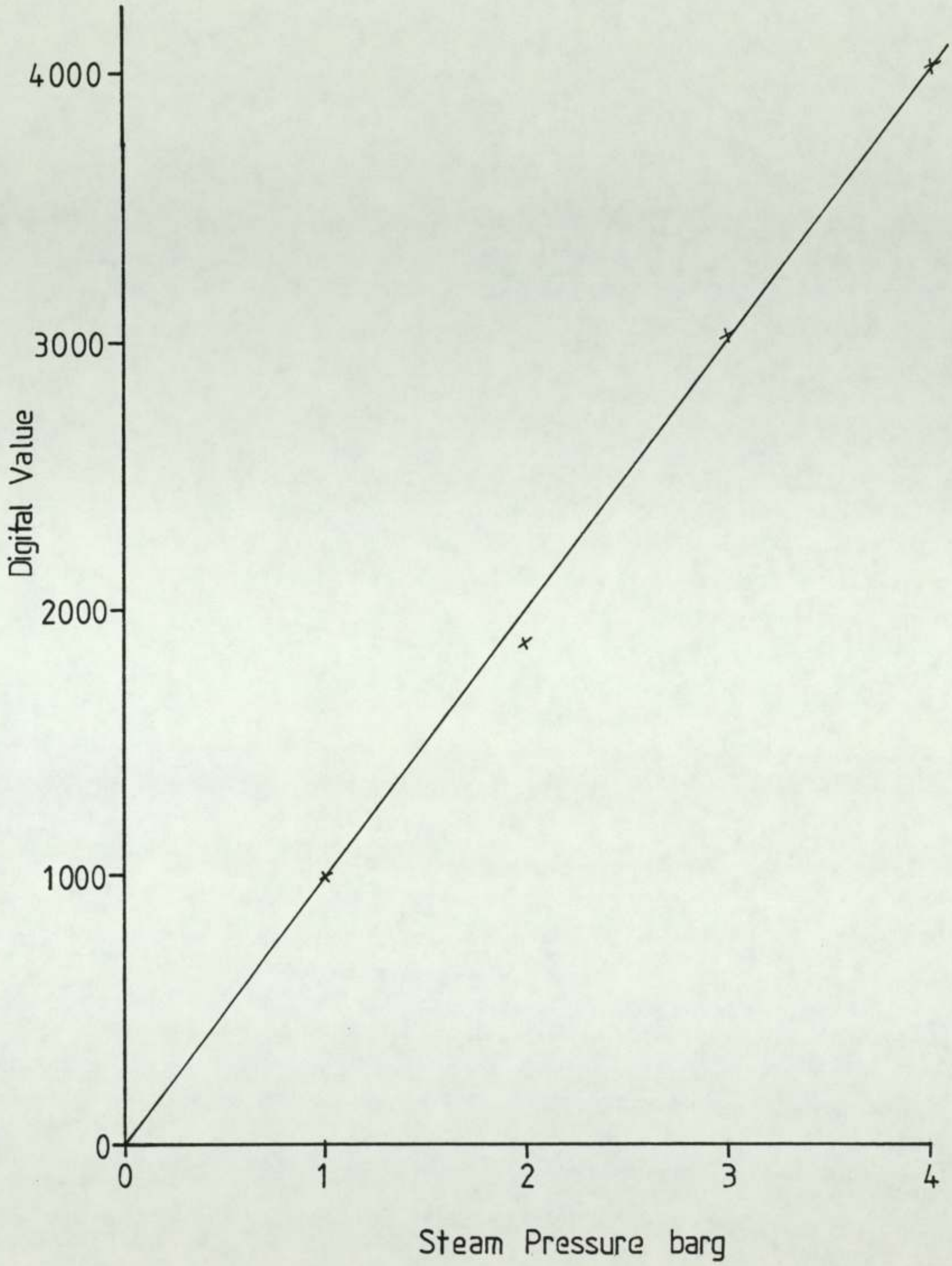
1/Gradient = 0·7424

Figure A2·10 Calibration Curve for Vacuum



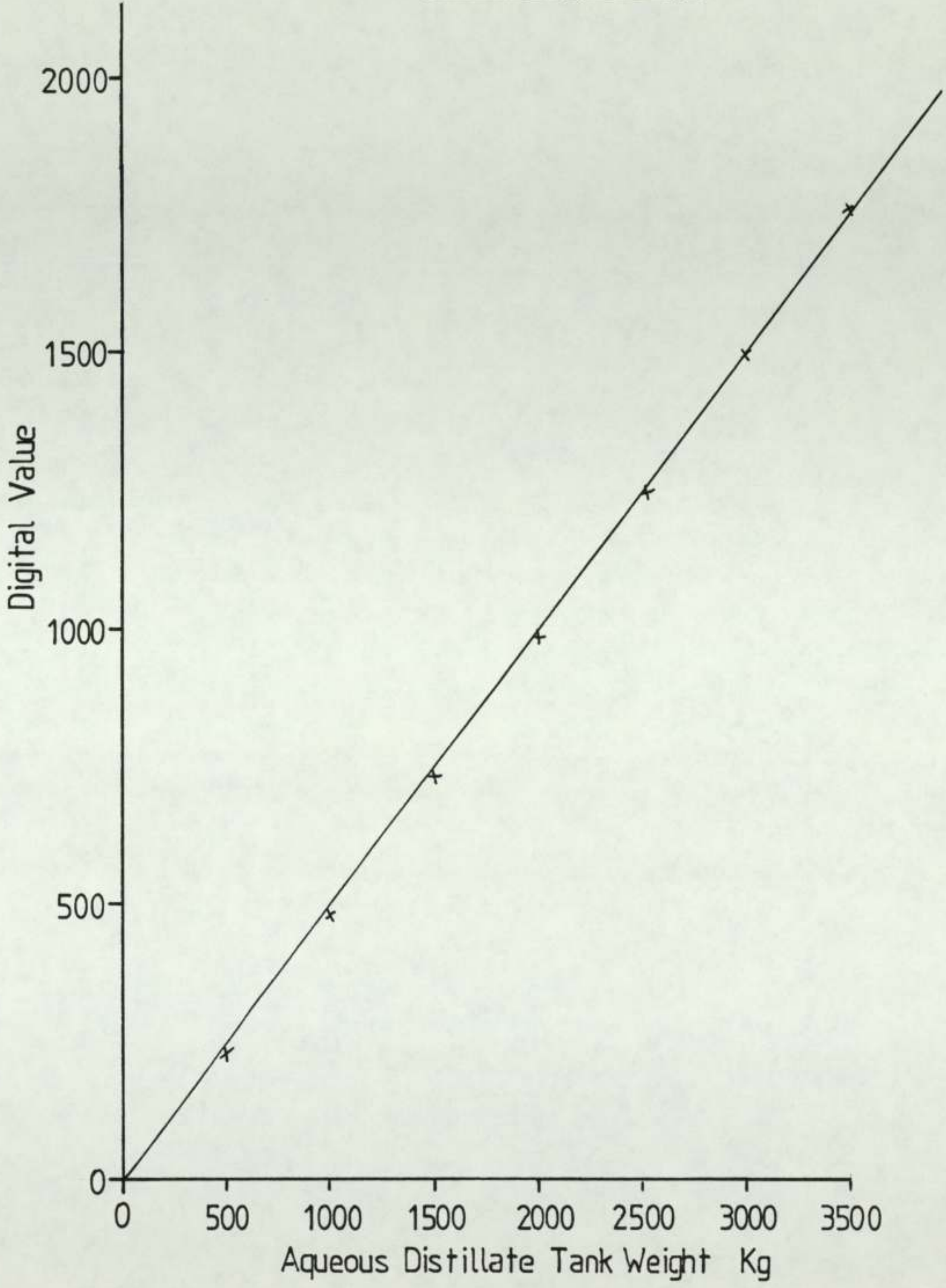
1/Gradient = 0·2466

Figure A2·11 Calibration Curve for Steam Pressure



1/Gradient = $9{\cdot}997\times10^{-4}$

Figure A2·12 Calibration Curve for Aqueous Distillate Tank Weight Kg

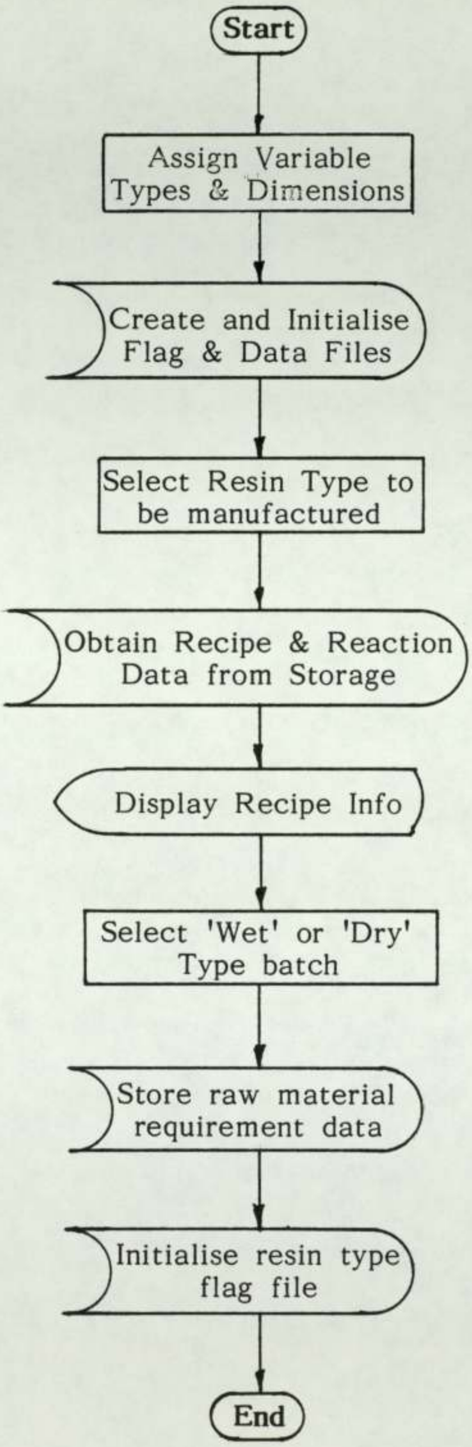


1/Gradient = 2·004

APPENDIX 3

Software - Flowcharts and Program Listings for the SEED 19 and BBC Computer

FIGURE A3.1 Procedure 'begin'



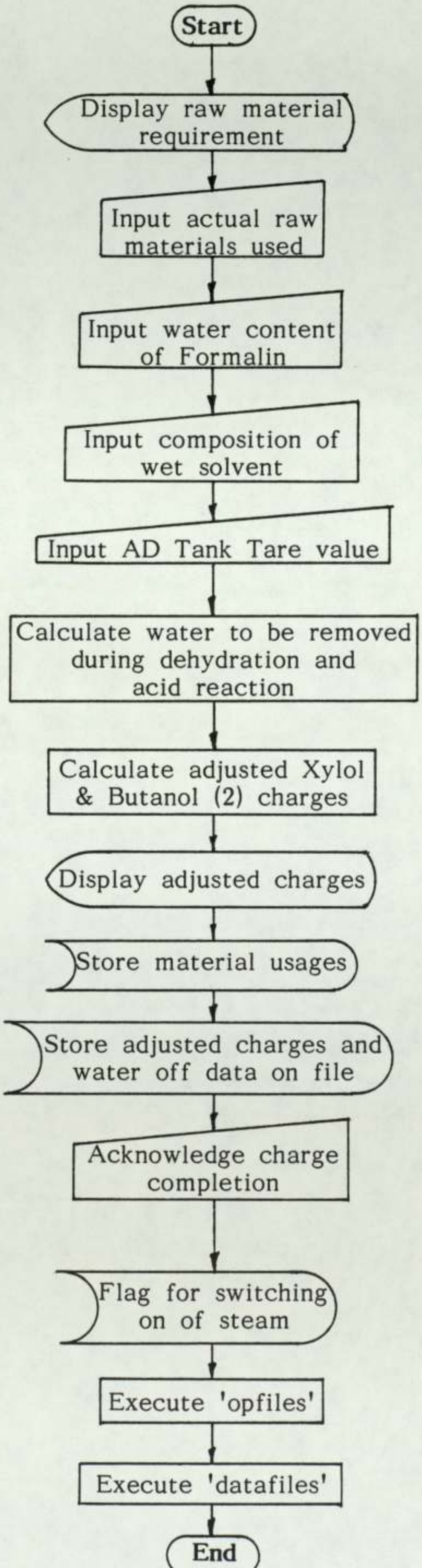
```
PROCEDURE begin
REM *************************************************************
REM *                                                           *
REM *                  Procedure 'begin'                        *
REM *                                                           *
REM *       Procedure to:                                       *
REM *                                                           *
REM *      (i) Select Resin Type and display Recipes            *
REM *                                                           *
REM *      (ii) Create processing flag files                    *
REM *                                                           *
REM *************************************************************
REM
REM
REM
REM Assigning of data types and array dimensioning
REM
DIM forma,melam,acat,afoam,faid,caus:REAL
DIM buoh1(3),buoh2(3),wbuoh1(3),wbuoh2(3),ibuoh1(3),ibuoh2(3),wibuoh1

(3),wibuoh2(3),xylo1(3):REAL
DIM path,prodrec1:BYTE
DIM typebu(2),ans(1),as(1):STRING
DIM typeres(6):STRING
DIM finish1,boil1,testdat1,restype1:BYTE
DIM adxylo1,adbuoh1,adacat1,areact1:BYTE
DIM steamon1,column_on1:BYTE
DIM colval,stval:INTEGER
ON ERROR GOTO 65
REM
REM Creation of Flag Files for end point determination and Resin Type selection
REM
CREATE £boil1,"/d1/boil"
CREATE £adxylo1,"/d1/adxylo"
CREATE £adbuoh1,"/d1/adbuoh"
CREATE £adacat1,"/d1/adacat"
CREATE £areact1,"/d1/areact"
CREATE £restype1,"/d1/restype"
REM
REM Creation and initialisation of Controller Set Point Files
REM
stval=0
CREATE £steamon1,"/d1/steamon"
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
colval=0
CREATE £column_on1,"/d1/columnt"
OPEN £column_on1,"/d1/columnt"
WRITE £column_on1,colval
CLOSE £column_on1
REM
REM Loading and Operation of Set Point Controller Procedures
REM
SHELL "steamset&"
SHELL "coltop&"
PRINT "Procedure startuup has begun"
PRINT
REM Selection of Resin Type to be produced
REM
```

```
PRINT "Which resin type is to be made, enter below"
PRINT
5 INPUT "RESIN TYPE    BE-",typeres(6)
PRINT
PRINT "Do you wish to change this value, enter y(yes), n(no)"
PRINT
INPUT "Answer is  -",ans(1)
IF ans(1)="y" THEN 5
PRINT
PRINT
REM
REM Retrieval of selected resin data form disc files
REM
10 OPEN £path,"/d1/"+typeres(6)
READ £path,forma,urea,melam,acat,afoam,faid,caus
BASE 1
FOR i=1 TO 3
READ £path,buoh1(i),buoh2(i),ibuoh1(i),ibuoh2(i),xylo1(i)
NEXT i
FOR i=1 TO 2
READ £path,wbuoh1(i),wbuoh2(i),wibuoh1(i),wibuoh2(i)
NEXT i
READ £path,reactwat,dehwat
CLOSE £path
REM
REM Table of resin data on VDU
REM
PRINT "The standard charges for resin "; typeres(6); " are as follows :-"
PRINT
IF melam=0 THEN 20
PRINT TAB(18); "Melamine"; TAB(31); melam; " kg"
PRINT
IF urea=0 THEN 25
20 PRINT TAB(18); "Urea"; TAB(31); urea; " kg"
PRINT
25 PRINT TAB(18); "Formalin"; TAB(31); forma; " litres"
PRINT
PRINT "Raw Materials"; TAB(25); "DRY"; TAB(42); "WET"; TAB(59); "ALL WET"
PRINT "-------------------------------------------------------------------------
--"
IF buoh1(1)=0 THEN 26
PRINT "n-Butanol(1)"; TAB(25); buoh1(1); TAB(42); buoh1(2); TAB(

60); buoh1(3)
PRINT
PRINT "n-Butanol(2)"; TAB(25); buoh2(1); TAB(42); buoh2(2); TAB(

60); buoh2(3)
PRINT
IF ibuoh1(1)=0 THEN 27
PRINT
26 PRINT "Iso-Butanol(1)"; TAB(25); ibuoh1(1); TAB(42); ibuoh1(2

); TAB(60); ibuoh1(3)
PRINT
PRINT "Iso-Butanol(2)"; TAB(25); ibuoh2(1); TAB(42); ibuoh2(2); TAB

(60); ibuoh2(3)
PRINT
27 PRINT "Wet n-Butanol(1)"; TAB(42); wbuoh1(1); TAB(60); wbuoh1
```

```
(2)
PRINT
PRINT "Wet n-Butanol(2)"; TAB(42); wbuoh2(1); TAB(60); wbuoh2(2)


PRINT
IF ibuoh1(1)=0 THEN 28
PRINT "Wet Iso-Butanol(1)"; TAB(42); wibuoh1(1); TAB(60); wibuoh1

(2)
PRINT
PRINT "Wet Iso-Butanol(2)"; TAB(42); wibuoh2(1); TAB(60); wibuoh2

(2)
PRINT
28 PRINT "Xylol"; TAB(25); xylol(1); TAB(42); xylol(2); TAB(60); xylol

(3)
PRINT "-------------------------------------------------------------------------
----"
PRINT TAB(18); "Acid Catalyst"; TAB(46); acat; " l"
PRINT
PRINT TAB(18); "Antifoam soln"; TAB(46); afoam; " l"
PRINT
PRINT TAB(18); "Caustic Soda soln"; TAB(46); caus; " ml"
PRINT
PRINT TAB(18); "J100 Filter Aid"; TAB(46); faid; " kg"
PRINT TAB(18); "-----------------------------------"
REM
REM Selection of Wet or Dry batch of resin
REM
PRINT "Is this to be a wet, dry or all wet butanol type resin"
PRINT
PRINT "Enter w(wet), d(dry), xw(all wet) below"
PRINT
50 INPUT "Answer is - ",typebu(2)
PRINT
PRINT "Do you wish to change this answer"
PRINT
PRINT "Enter y(yes),n(no)"
PRINT
REM
REM Creation of file containing the specific Raw Material requirements
REM for the Resin and batch type chosen
REM
CREATE £prodrec1,"/d1/prodrec"
INPUT "Answer is - ",as(1)
IF as(1)="y" THEN 50
REM
REM File for 'Dry' batch charge
REM
55 IF typebu(2)="d" THEN
OPEN £prodrec1,"/d1/prodrec"
WRITE £prodrec1,forma,urea,melam,acat,afoam,faid,caus,buoh1(1),buoh2

(1),ibuoh1(1),ibuoh2(1),xylol(1),reactwat,dehwat
CLOSE £prodrec1
ENDIF
REM
```

```
REM File for 'Wet' batch charge
IF typebu(2)="w" THEN
OPEN £prodrec1,"/d1/prodrec"
WRITE £prodrec1,forma,urea,melam,acat,afoam,faid,caus,buoh1(2),buoh2

(2),wbuoh1(2),wbuoh2(2),ibuoh1(2),ibuoh2(2),wibuoh1(1),wibuoh2(2)

,xylol(2),reactwat,dehwat
CLOSE £prodrec1
ENDIF
GOTO 70
REM
REM Error trap
REM
65 errnum=ERR
IF errnum=218 THEN
SHELL "sleep 60"
ENDIF
GOTO 55
REM
REM Initialisation of file with selected batch type
REM
70 OPEN £restype1,"/d1/restype"
WRITE £restype1,typebu(2)
CLOSE £restype1
END
```

FIGURE A3.2 Procedure 'wet'



```
PROCEDURE wet
REM ***************************************************************
REM *                                                             *
REM *                     Procedure 'wet'                         *
REM *                                                             *
REM *       Procedure to:                                         *
REM *                                                             *
REM *      (i) Display and request material additions for a       *
REM *           'wet' batch of resin                              *
REM *                                                             *
REM *      (ii) Calculate mass of water to be removed during      *
REM *           Dehydration and Acid Reaction Stages              *
REM *                                                             *
REM ***************************************************************
REM
REM
REM
REM Assignment of data types and array dimensions
REM
DIM prodrec1,adjust1,resdat1,finish1,restype1,steamon1,ad1,p1:BYTE
DIM typr,typebu(2),ctime(6),ans(1):STRING
DIM resin(6),batch(4),shift(2):STRING
DIM stval,chan,dat:INTEGER
REM
REM Accessing of File to determine which batch type is to be
REM manufactured
REM
OPEN £restype1,"/d1/restype"
READ £restype1,typebu(2)
CLOSE £restype1
IF typebu(2)="d" THEN 70
REM Acquisition of 'wet' batch data from disc
REM
OPEN £prodrec1,"/d1/prodrec"
READ £prodrec1,forma,urea,melam,acat,afoam,faid,caus,buoh1,buoh2

,wbuoh1,wbuoh2,ibuoh1,ibuoh2,wibuoh1,wibuoh2,xylo1,reactwat,dehwat
CLOSE £prodrec1
REM
REM Entering of information for Logsheet generation
REM
INPUT "Resin Type, BEXXXX  - ",resin(6)
PRINT
INPUT "Batch no.  - ",batch(4)
PRINT
INPUT "Shift  - ",shift(2)
1 PRINT "Has charging commenced ?, y(yes),n(no)"
INPUT "Answer is  - ",ans(1)
IF ans(1)="y" THEN
ctime(6)=MID$(DATE$,10,5)
REM
REM Generation of first section of the logsheet
REM
OPEN £p1,"/pS":WRITE
PRINT £p1,CHR$(12)
PRINT £p1,"Resin Type - "; resin(6); "   Batch no. - "; batch(4)

; "   Shift - "; shift(2)
PRINT £p1
PRINT £p1
```

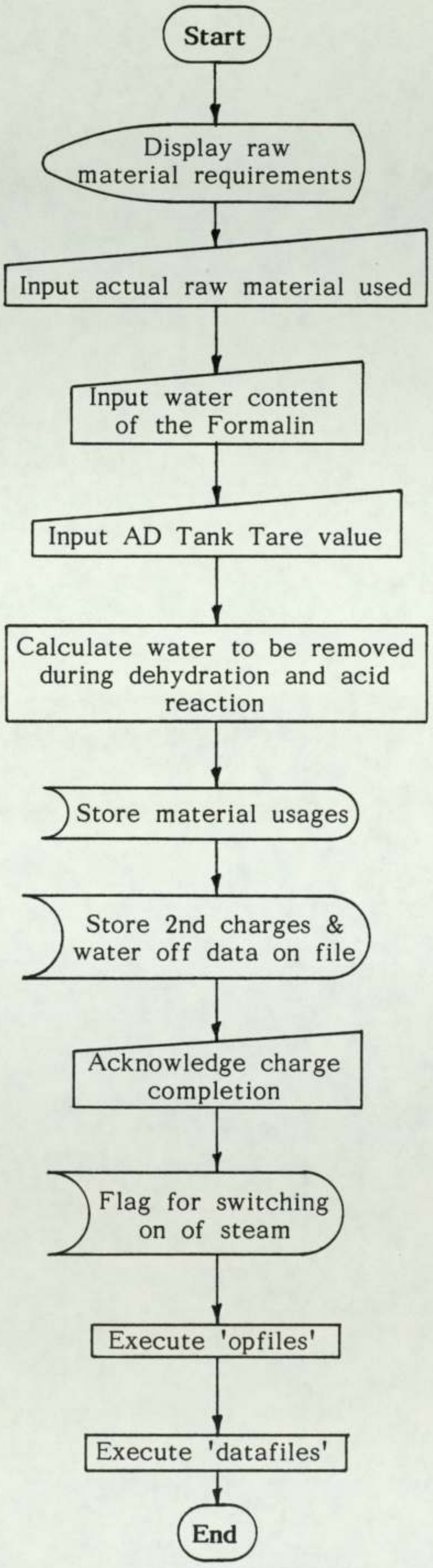
```
PRINT £p1
PRINT £p1,"Charging commenced at  - "; ctime(6)
PRINT £p1
CLOSE £p1
ELSE
GOTO 1
ENDIF
PRINT "Enter the actual raw material quanties used below"
PRINT
REM
REM Entry of Formalin composition
REM
PRINT "Standard Formalin charge - "; TAB(50); forma
PRINT
INPUT "Formalin used  - ",forma
PRINT
INPUT "Water content of the formalin  -  ",formw
PRINT
INPUT "Density of the formalin  -  ",formdens
PRINT
PRINT "Wait while the AD Tank Tare value is calculated"
PRINT
chan=1
taread=.0
REM
REM Calculation of the A.D. Weigh Tank Tare value
REM
OPEN £ad1,"/anlg"
FOR i=1 TO 10
PUT £ad1,chan
GET £ad1,dat
taread=taread+FLOAT(dat)
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=6
NEXT i
CLOSE £ad1
tare=taread/10*2.004
PRINT
PRINT "Tare value is - "; tare
REM Calculation of the mass of water charged in the Formalin
REM
forwat=forma*formdens*formw
IF wibuoh1=0 THEN 5
REM
REM Entry of raw material quantities charged
REM
PRINT "Standard Iso-Butanol(1) charge - "; TAB(50); ibuoh1
PRINT
INPUT "Iso-Butanol(1) used",ibuoh1
GOTO 10
PRINT
5 PRINT "Standard n-Butanol(1) charge - "; TAB(50); buoh1
PRINT
INPUT "n-Butanol(1) used  -  ",buoh1
10 PRINT
```

```
INPUT "Anti - foam soln used  -",afoam
PRINT
PRINT "The pH of the still contents requires adjustment to a value between .8 -
6.3"
PRINT
PRINT "The standard caustic soln should be used to make this adjustment"
PRINT
INPUT "Caustic soda soln used  -",caus
IF wibuoh1=0 THEN 15
PRINT
PRINT "Standard Wet Iso-Butanol(1) charge - "; TAB(50); wibuoh1
PRINT
INPUT "Wet Iso-Butanol(1) used  -",wibuoh1
GOTO 20
15 PRINT
PRINT "Standard Wet n-Butanol(1) charge - "; TAB(50); wbuoh1
PRINT
INPUT "Wet n-Butanol(1)  used    -",wbuoh1
20 PRINT
IF melam=0 THEN 25
PRINT "Standard Melamine charge - "; TAB(50); melam
PRINT
INPUT "Melamine used  -",melam
PRINT
GOTO 30
25 PRINT "Standard Urea charge - "; TAB(50); urea
PRINT
INPUT "Urea used - ",urea
30 PRINT
REM
REM Entry of the Wet Solvent composition
REM
PRINT "Enter the percentages of xylol and water by weight of the wet solvent"
PRINT
INPUT "Percentage of xylol in the wet solvent  -  ",perxylo
PRINT
INPUT "Percentage of water in the wet solvent  -  ",perwat
PRINT
INPUT "Enter the DENSITY of the wet solvent stream  -  ",dens
IF wbuoh1=0 THEN 35
REM
REM Calculation of water content of the Wet Solvent, the Water to be
REM removed during each stage and the Butanol(2) adjusted charge
REM
charmass=wbuoh1*dens
xymass=perxylo*charmass/100
wmass=perwat*charmass/100
stxymass=415.*.861
xylo=(stxymass-xymass)/.861
IF xymass>stxymass THEN xylo=.0
ENDIF
totwat=forwat+reactwat+wmass+dehwat+tare
rbuoh=(buoh1+buoh2)*.81
buohrem=(totwat-tare)*.08
totbuoh=(buoh1+buoh2)*.81+charmass*(100-perwat-perxylo)*.01
buohleft=totbuoh-buohrem
IF buohleft-rbuoh<0 THEN
buoh2=(rbuoh-buohleft)/.81+buoh2
ENDIF
ibuoh2=.0
```

```
REM
REM Display of adjusted charges and water to be removed masses
REM
PRINT
PRINT "Charge mass  "; charmass
PRINT "Water mass   "; wmass
PRINT "Xylol wet solvent   "; xylo
PRINT "Total water off     "; totwat
PRINT "Total buoh added    "; totbuoh
PRINT "Butanol(2) charge   "; buoh2
PRINT
GOTO 40
35 charmass=wibuoh1*dens
xymass=perxylo*charmass/100
wmass=perwat*charmass/100
stxymass=415.*.861
xylo=(stxymass-xymass)/.861
IF xymass>stxymass THEN xylo=.0
ENDIF
totwat=forwat+reactwat+wmass+dehwat+tare
rbuoh=(ibuoh1+ibuoh2)*.805
buohrem=(totwat-tare)*.08
totbuoh=(ibuoh1+ibuoh2)*.805+charmass*(100-perwat-perxylo)*.01
buohleft=totbuoh-buohrem
IF buohleft-rbuoh<0 THEN
ibuoh2=(rbuoh-buohleft)/.805+ibuoh2
ENDIF
buoh2=0
40 PRINT
typr="wet"
REM Water to be removed in 3hr boil-up
addwater=wmass+forwat+dehwat+tare
REM File containing Raw Material adjustments
CREATE £adjust1,"/d1/adjust"
OPEN £adjust1,"/d1/adjust"
WRITE £adjust1,xylo,ibuoh2,buoh2,typr,addwater,totwat
CLOSE £adjust1
PRINT "Water off during dehydration  - "; addwater
PRINT "Water off at end of acid reaction  - "; totwat
REM File containing Raw Material additions
CREATE £resdat1,"/d1/resdat"
OPEN £resdat1,"/d1/resdat"
WRITE £resdat1,forma,buoh1,ibuoh1,wbuoh1,wibuoh1,afoam,melam,urea

,caus
CLOSE £resdat1
PRINT "To acknowledge charge completion and the turning on off the steam"
PRINT
PRINT "hit any numeric key"
PRINT
INPUT "Answer is  -  ",carry
REM
REM Accessing of flag file to indicate steam switched on
REM
stval=1
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
REM
REM Print out on the logsheet all the values which have been
```

```
REM entered and calculated in this procedure
REM
OPEN £p1,"/pS":WRITE
PRINT £p1,"Formalin  - "; forma; " litres"
PRINT £p1,"Formalin water content  - "; formw
PRINT £p1,"Density of Formalin  - "; formdens
PRINT £p1
PRINT £p1,"A.D. Tank Tare Value  - "; tare; " Kg"
PRINT £p1
IF buoh1=.0 THEN 50
PRINT £p1,"n-Butanol(1) charged  - "; buoh1; " litres"
GOTO 51
50 PRINT £p1,"Iso-Butanol(1) charged  - "; ibuoh1; " litres"
51 IF buoh1=.0 THEN 52
PRINT £p1,"Wet n-Butanol(1) charged  - "; wbuoh1; " litres"
GOTO 53
52 PRINT £p1,"Wet Iso-Butanol(1) charged  - "; wibuoh1; " litres"
53 PRINT £p1
IF melam=0 THEN 54
PRINT £p1
PRINT £p1,"Melamine charged  - "; melam; " Kg"
GOTO 55
54 PRINT £p1
PRINT £p1,"Urea charged  - "; urea; " Kg"
55 PRINT £p1
PRINT £p1,"Water content of the Wet Solvent  - "; perwat; "%"
PRINT £p1,"Xylol content of the Wet Solvent  - "; perxylo; "%"
PRINT £p1,"Density of Wet Solvent  - "; dens; " Kg/M3"
PRINT £p1
PRINT £p1,"Water content of charge  - "; wmass; " Kg"
PRINT £p1,"Adjusted Xylol charge  - "; xylo; " litres"
PRINT £p1
PRINT £p1,"Total Butanol added  - "; totbuoh; " litres"
PRINT £p1,"Adjusted Butanol(2) charge  - "; buoh2; " litres"
PRINT £p1,"Water off during dehydration  - "; addwater; " Kg"
PRINT £p1,"Water off during Acid Reaction  - "; totwat; " Kg"
PRINT £p1
PRINT £p1
PRINT £p1
PRINT £p1
PRINT £p1,CHR$(12)
CLOSE £p1
REM
REM Operation of procedure to initalise all the flag files
REM
SHELL "opfiles&"
70 END
```

FIGURE A3.3 Procedure 'dry'



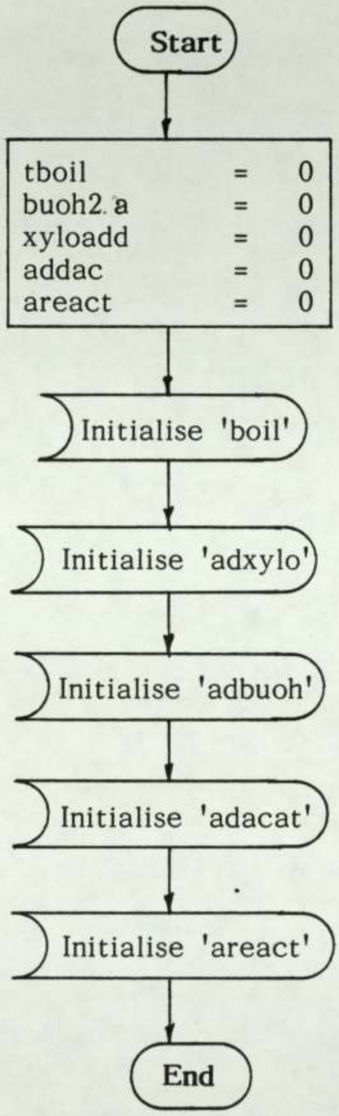
```
PROCEDURE dry
REM ****************************************************************
REM *                                                              *
REM *                    Procedure 'dry'                           *
REM *                                                              *
REM *     Procedure to:                                            *
REM *                                                              *
REM *     (i) Display and request material additions for a         *
REM *         'dry' batch of resin                                 *
REM *                                                              *
REM *     (ii) Calculate mass of water to be removed during        *
REM *          Dehydration and Acid Reaction Stages                *
REM *                                                              *
REM ****************************************************************
REM
REM
REM
REM Assignment of data types and array dimensions
REM
DIM prodrec1,resdat1,adjust1,finish1,restype1,steamon1,ad1,p1:BYTE
DIM typr,typebu(2),ans(1):STRING
DIM resin(6),batch(4),shift(2):STRING
DIM stval,dat,chan:INTEGER
DIM taread:REAL
DIM ctime(6):STRING
REM
REM Accessing of File to determine which batch type is to be
REM manufactured
REM
OPEN £restype1,"/d1/restype"
READ £restype1,typebu(2)
CLOSE £restype1
IF typebu(2)="w" THEN 70
REM
REM Acquisition of 'dry' batch data from disc
OPEN £prodrec1,"/d1/prodrec"
READ £prodrec1,forma,urea,melam,acat,afoam,faid,caus,buoh1,buoh2

,ibuoh1,ibuoh2,xylo1,reactwat,dehwat
CLOSE £prodrec1
REM
REM Entering of information for Logsheet generation
REM
INPUT "Resin Type, BEXXXX  - ",resin(6)
PRINT
INPUT "Batch no.  - ",batch(4)
PRINT
INPUT "Shift  - ",shift(2)
1 PRINT "Has charging commenced ?, y(yes), n(no)"
PRINT
INPUT "Answer is  - ",ans(1)
IF ans(1)="y" THEN
ctime(6)=MID$(DATE$,10,5)
REM
REM Generation of first section of the logsheet
REM
OPEN £p1,"/pS":WRITE
PRINT £p1,CHR$(12)
PRINT £p1,"Resin Type - "; resin(6); "   Batch no. - "; batch(4)
```

```
; "   Shift - "; shift(2)
PRINT £p1
PRINT £p1
PRINT £p1
PRINT £p1,"Charging commenced at  - "; ctime(6)
PRINT £p1
CLOSE £p1
ELSE
GOTO 1
ENDIF
PRINT "Enter the actual raw material quanties used below"
PRINT
REM
REM Entry of Formalin composition
REM
PRINT "Standard Formalin charge -"; TAB(50); forma
PRINT
INPUT "Formalin used   -",forma
PRINT
INPUT "Water content of the formalin  -  ",formw
PRINT
INPUT "Density of the formalin - ",formdens
REM
REM Calculation of the water added in the Formalin and the total water to
REM be removed during each stage of the reaction
REM
PRINT
PRINT "Wait while the AD Tare weight is calculated"
PRINT
chan=1
taread=.0
REM
REM Calculation of the A.D. Weigh Tank Tare value
REM
OPEN £ad1,"/anlg"
FOR i=1 TO 10
PUT £ad1,chan
GET £ad1,dat
taread=taread+FLOAT(dat)
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=6
NEXT i
CLOSE £ad1
tare=taread/10*2.004
PRINT "Tare value is - "; tare
REM
REM Calculation of water to be removed during each reaction stage
REM
formwat=formw*forma*formdens
totwat=reactwat+formwat+dehwat+tare
addwat=formwat+dehwat+tare
PRINT
REM
REM Entry of raw material quantities charged
REM
```

```
IF ibuoh1=0 THEN 5
PRINT "Standard Isobutanol charge -"; TAB(50); ibuoh1
PRINT
INPUT "Iso-Butanol(1) used  -",ibuoh1
GOTO 10
5 PRINT
PRINT "Standard n-Butanol charge -"; TAB(50); buoh1
PRINT
INPUT "n-Butamol used   -",buoh1
10 PRINT
INPUT "Anti-foam soln used  -",afoam
PRINT
PRINT "The pH of the still contents  should be in the range 5.8 - 6.3"
PRINT
PRINT "To adjust the pH the standard caustic soln should be used "
PRINT
INPUT "Caustic soda soln used  -",caus
IF melam=0 THEN 25
PRINT
PRINT "Standard Melamine charge -"; TAB(50); melam
PRINT
INPUT "Melamine used  - ",melam
urea=0
GOTO 30
25 PRINT
PRINT "Standard Urea charge -"; TAB(50); urea
PRINT
INPUT "Urea used   -",urea
melam=0
30 PRINT
typr="dry"
wbuoh1=.0
REM
REM Creation of file containing the actual Raw Material usages for generation
REM on a Logsheet
REM
CREATE £resdat1,"/d1/resdat"
OPEN £resdat1,"/d1/resdat"
WRITE £resdat1,forma,buoh1,ibuoh1,wbuoh1,wibuoh1,afoam,melam,urea

,caus
CLOSE £resdat1
REM
REM Creation of a file containing the adjusted pure charges
REM
CREATE £adjust1,"/d1/adjust"
OPEN £adjust1,"/d1/adjust"
WRITE £adjust1,xylo1,ibuoh2,buoh2,typr,addwat,totwat
CLOSE £adjust1
PRINT
PRINT "Water off during dehydration  - "; addwat
PRINT "Water removed at the end of acid reaction  - "; totwat
PRINT
PRINT
PRINT "To acknowledge charge completion and turn on of steam hit any numeric key
 "
PRINT
INPUT "Acknowledge - ",carry
REM
REM Accessing flag file to indicate steam switched on
```

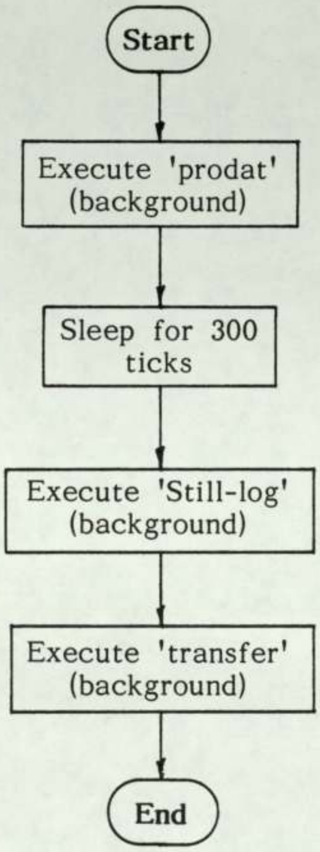
```
REM
stval=1
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
REM
REM Print out on the logsheet all the values which have been
REM entered and calculated
REM
OPEN £p1,"/pS":WRITE
PRINT £p1,"Formalin  - "; forma; " litres"
PRINT £p1,"Formalin water content  - "; formw
PRINT £p1,"Density of Formalin  - "; formdens; " Kg/M3"
PRINT £p1
PRINT £p1,"A.D. Tank Tare Value  - "; tare; " Kg"
IF buoh1=.0 THEN 50
PRINT £p1,"n-Butanol(1) charged  - "; buoh1; " litres"
PRINT £p1
GOTO 51
50 PRINT £p1,"Iso-Butanol(1) charged  - "; ibuoh1; " litres"
PRINT £p1
51 IF melam=.0 THEN 54
PRINT £p1,"Melamine charged  - "; melam; " Kg"
PRINT £p1
GOTO 55
54 PRINT £p1,"Urea charged  - "; urea; " Kg"
PRINT £p1
55 PRINT £p1,"Water content of charge  - "; formwat; " Kg"
PRINT £p1
PRINT £p1,"Water off during dehydration  - "; addwat; " Kg"
PRINT £p1,"Water off during Acid Reaction  - "; totwat; " Kg"
PRINT £p1
PRINT £p1
PRINT £p1
PRINT £p1
PRINT £p1,CHR$(12)
CLOSE £p1
REM
REM Operation of procedure to initialise all the flag files
REM
SHELL "opfiles&"
70 END
```

FIGURE A3.4 Procedure 'opfiles'



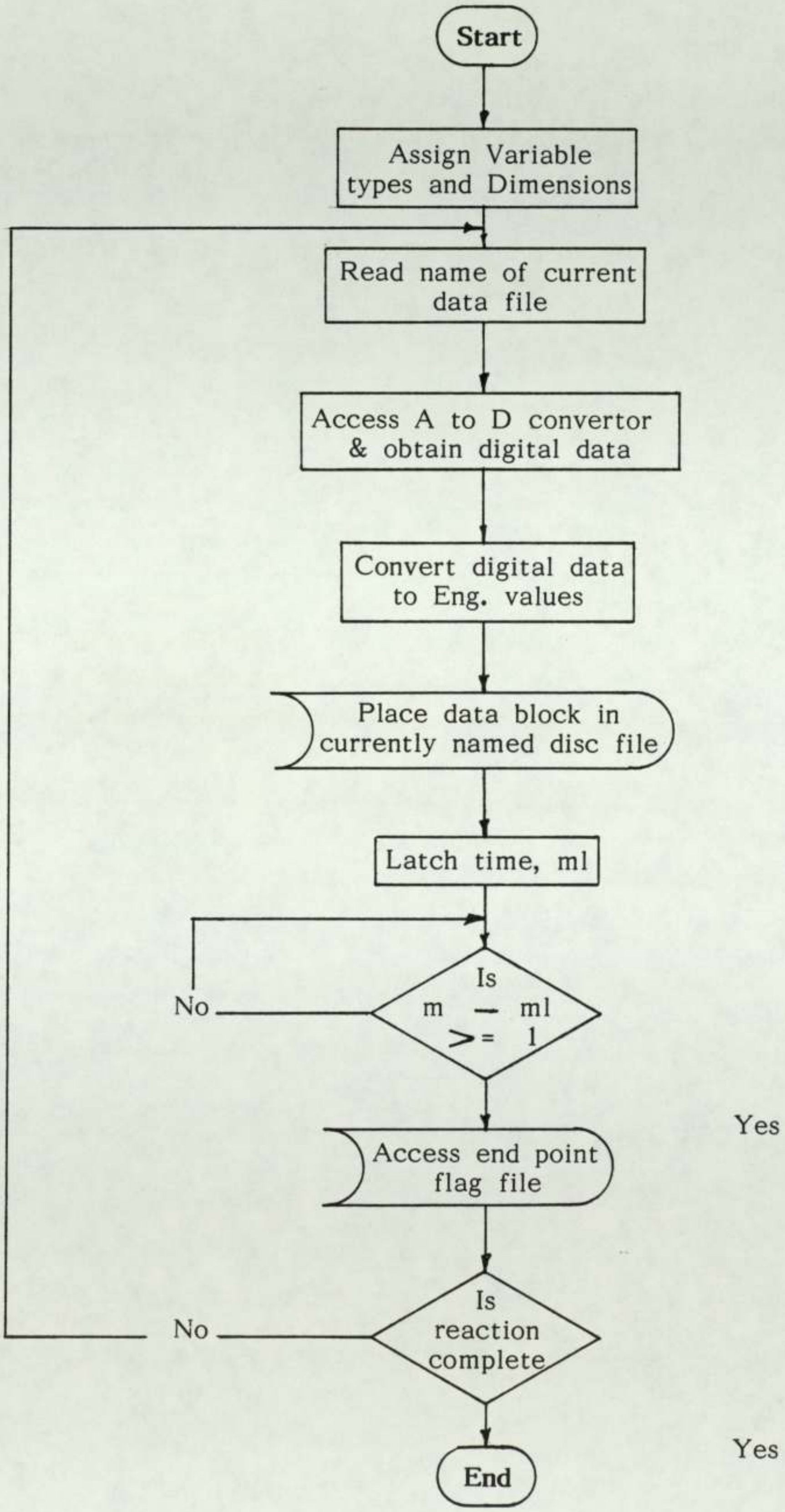
```
PROCEDURE opfiles
REM
REM Procedure to set initial values in process flag files
REM
DIM boil1,adxylo1,adbuoh1,adacat1,areact1:BYTE
tboil=0
buoh2a=0
xyloadd=0
addac=0
areact=0
REM Flag file to indicate boiling commenced
OPEN £boil1,"/d1/boil"
WRITE £boil1,tboil
CLOSE £boil1
REM Flag file for Xylol addition
OPEN £adxylo1,"/d1/adxylo"
WRITE £adxylo1,xyloadd
CLOSE £adxylo1
REM Flag file for Butanol(2) addition
OPEN £adbuoh1,"/d1/adbuoh"
WRITE £adbuoh1,buoh2a
CLOSE £adbuoh1
REM Flag file for Acid Catalyst addition
OPEN £adacat1,"/d1/adacat"
WRITE £adacat1,addac
CLOSE £adacat1
REM Flag file to indicate the end of the acid reaction
OPEN £areact1,"/d1/areact"
WRITE £areact1,areact
CLOSE £areact1
END
```

FIGURE A3.5 Procedure 'datalogs'



```
PROCEDURE datalogs
REM
REM Procedure to begin operation of logging routines
REM
SHELL "prodat&"
SHELL "sleep 400"
SHELL "still_log&"
SHELL "transfer&"
END
```

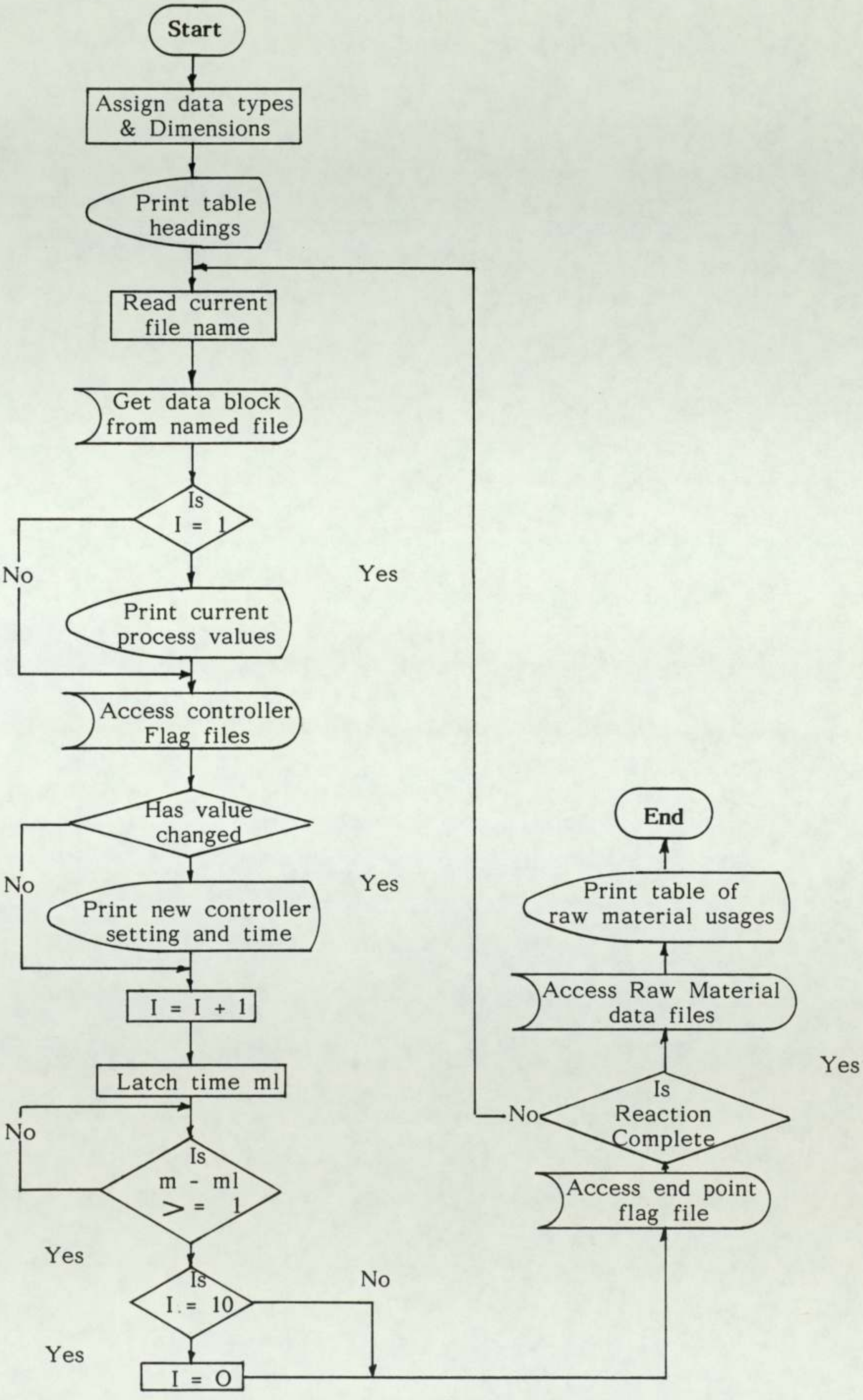
FIGURE A3.6 Procedure 'prodat'



```
PROCEDURE prodat
REM **************************************************
REM *                                                *
REM *     Procedure to convert Analogue inputs into  *
REM *                                                *
REM *     Digital values for further conversion to   *
REM *                                                *
REM *     Engineering values                         *
REM *                                                *
REM **************************************************
REM
REM
REM Definition of variables and arrays
DIM rtime(6):STRING
DIM data1,ad1,process1,finish1:BYTE
DIM value(12),avad(10),b(10):REAL
DIM a(16):REAL
DIM chan,dat,i,dat1:INTEGER
DIM start(6),stime(6):STRING
DIM name:STRING[2]
ON ERROR GOTO 15
REM
REM File names for intermediate data storage
DATA "1","2","3","4","5","6","7","8","9","10"
k=0
GOTO 5
3 i=i+1
5 READ name
6 jk=1
REM
REM Aquiring data from A-D Convertor
REM
OPEN £ad1,"/anlg"
BASE 0
FOR chan=0 TO 12
PUT £ad1,chan
GET £ad1,dat
a(chan)=dat
NEXT chan
CLOSE £ad1
REM
REM Conversion of digital values to Eng values
REM
value(0)=.0242*a(0)+30.
value(1)=2.004*a(1)
value(2)=.367*a(3)
value(3)=.0009997*a(4)
value(4)=.01326*a(5)
value(5)=.01226*a(6)
value(6)=.2466*a(7)
value(7)=.01226*a(8)+55
value(8)=.01226*a(9)
value(9)=.37121*a(10)
value(10)=.7424*a(11)
value(11)=.7424*a(12)
BASE 1
rtime(6)=MID$(DATE$,10,5)
jk=2
REM
REM Placing of Eng. values in data files
```

```
REM
10 OPEN £process1,"/d0/POVAR/dat"+name
PUT £process1,rtime(6)
PUT £process1,value
CLOSE £process1
REM
REM Time loop of one minute
REM
m1=VAL(MID$(DATE$,13,2))
REPEAT
m=VAL(MID$(DATE$,13,2))
IF m<m1 THEN
m=m+60
ENDIF
UNTIL m-m1>=1
REM
REM Return to step 3 to access A-D converter
REM
GOTO 3
REM Error trap
15 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+1
ENDIF
UNTIL s-s1>=5
REM
REM File accessing error returns to appropriate point
REM
IF jk=1 THEN 6
IF jk=2 THEN 10
20 END
```

FIGURE A3.7 Procedure 'Still-log'



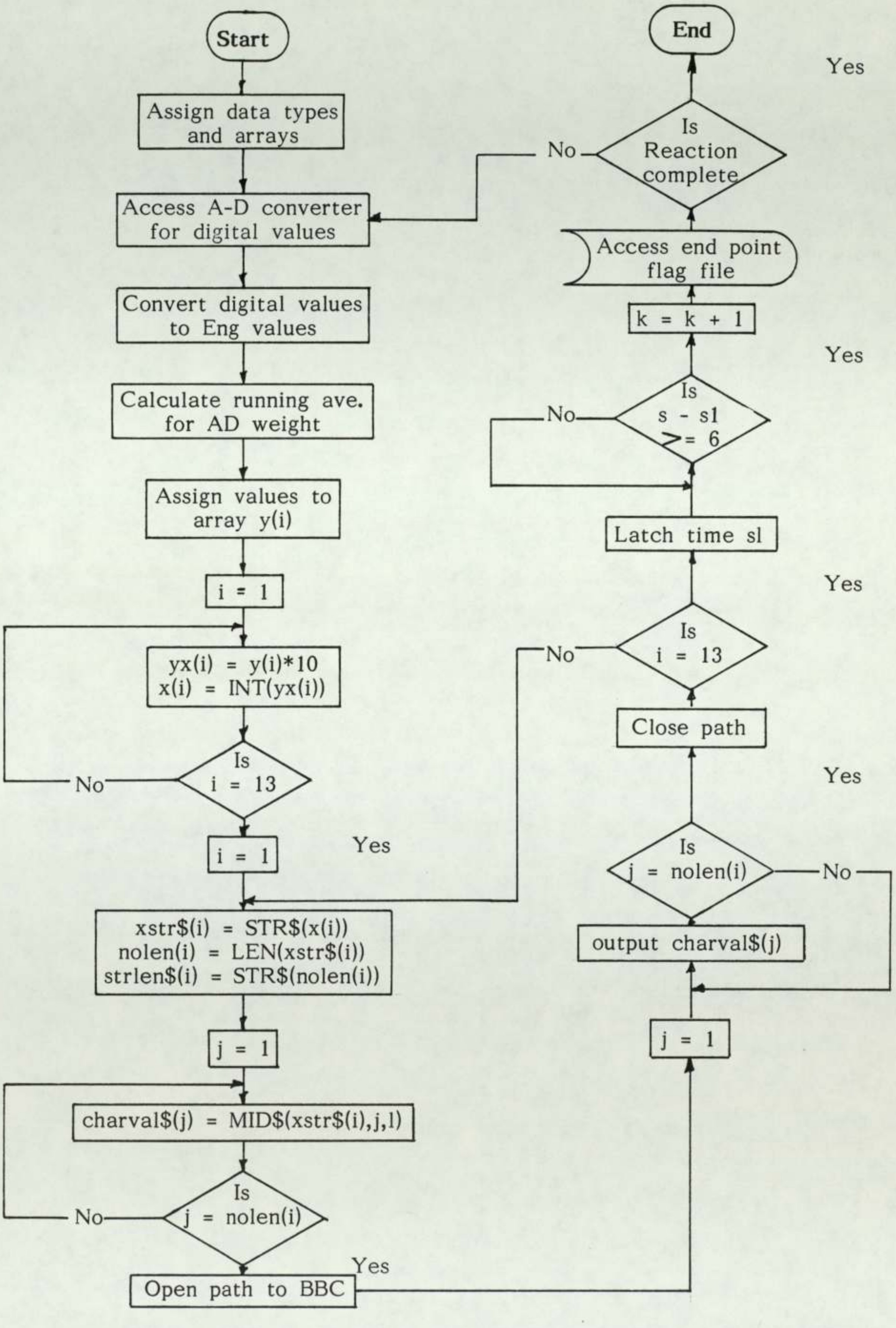
```
PROCEDURE still_log
REM ********************************************************************
REM *                                                                  *
REM * Procedure which generates a Logsheet containing process values   *
REM *                                                                  *
REM ********************************************************************
REM
REM Assigning of data types and dimensioning of variables
REM
DIM p1,process1,steamon1,column_on1:BYTE
DIM testdat1,resdat1,additions1:BYTE
DIM stval,colval,stval1,colval1:INTEGER
DIM rtime(6):STRING
DIM name:STRING[2]
DIM value(12):REAL
DIM i:INTEGER
DIM dtime(6):STRING
stval1=0
colval1=0
ON ERROR GOTO 10
REM
REM File names for intermediate data storage
REM
DATA "1","2","3","4","5","6","7","8","9","10"
SHELL "sleep 200"
OPEN £p1,"/pS":WRITE
REM Printing of log sheet headings
PRINT £p1; TAB(2); "TIME"; TAB(9); "STEAM"; TAB(18); "COIL"; TAB

(25); "STILL"; TAB(34); "DIST"; TAB(41); "COLUMN"; TAB(50); "A.D."

; TAB(58); "ALCO"; TAB(65); "CF.TEMP"; TAB(74); "CR.TEMP"
PRINT £p1; TAB(9); "FLOW"; TAB(18); "PRESS"; TAB(25); "TEMP"; TAB

(34); "degC"; TAB(41); "TOP TEM"; TAB(50); "OFF"; TAB(58): "TANKWT"
PRINT £p1; TAB(9); "KG/HR"; TAB(18); "BAR"; TAB(25); "DEGC"; TAB

(34); "DEGC"; TAB(43); "DEGC"; TAB(50); "L/HR"; TAB(60); "Kg"; TAB

(66); "DEGC"; TAB(75); "DEGC"
PRINT £p1,"-------------------------------------------------------------------
----------"
i=1
5 READ name
REM Acquisition of data from process data files
6 OPEN £process1,"/d0/POVAR/dat"+name
GET £process1,rtime(6)
GET £process1,value
CLOSE £process1
REM Assigning data values to variable names
valu1=value(1)
valu2=value(2)
valu3=value(3)
valu4=value(4)
valu5=value(5)
valu6=value(6)
valu7=value(7)
valu8=value(8)
valu9=value(9)
valu10=value(10)
```

```
valu11=value(11)
valu12=value(12)
IF i=1 THEN
REM Logging of specific process values on a hardcopy
PRINT £p1 USING "s7,X1,9(r8.1)",rtime(6),valu3,valu4,valu1,valu9

,valu8,valu10,valu2,valu6,valu5
PRINT £p1,"_-----------------------------------------------------------------
-----------"
ENDIF
REM Accessing controller flag files to determine current set points
OPEN £steamon1,"/d1/steamon"
READ £steamon1,stval
CLOSE £steamon1
OPEN £column_on1,"/d1/columnt"
READ £column_on1,colval
CLOSE £column_on1
IF colval=colval1 THEN 7
IF colval=1 THEN
REM Logging of change in Column Set point and time of change
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Column Top Temperature set at "; dtime(6); " Set value =90 degC"
PRINT £p1,"_-----------------------------------------------------------------
-----------"
ENDIF
IF colval=0 THEN
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Column Top Temperature Controller switched off at "; dtime

(6)
PRINT £p1,"_-----------------------------------------------------------------
-----------"
ENDIF
7 colval1=colval
IF stval=stval1 THEN 8
IF stval=1 THEN
REM Logging of change in Steam pressure
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Steam Pressure set at 3 bar at "; dtime(6)
PRINT £p1,"_-----------------------------------------------------------------
-----------"
ENDIF
IF stval=2 THEN
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Steam Pressure set at 2 bar at "; dtime(6)
PRINT £p1,"_-----------------------------------------------------------------
-----------"
ENDIF
IF stval=3 THEN
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Steam switched of at "; dtime(6)
PRINT £p1,"_-----------------------------------------------------------------
-----------"
ENDIF
IF stval=10 THEN
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Steam switched off to await Solvent Stripping at "; dtime

(6)
PRINT £p1,"_-----------------------------------------------------------------
```

```
------------"
ENDIF
IF stval=5 THEN
dtime(6)=MID$(DATE$,10,5)
PRINT £p1,"Steam Pressure set at 0.5 bar at "; dtime(6)
PRINT £p1,"-----------------------------------------------------------------------
-----"
ENDIF
8 stval1=stval
REM Timing loop of one minute
m1=VAL(MID$(DATE$,13,2))
REPEAT
m=VAL(MID$(DATE$,13,2))
IF m<m1 THEN
m=m+60
ENDIF
UNTIL m-m1>=1
IF i=10 THEN
i=0
ENDIF
i=i+1
IF stval=10 THEN 20
REM return to step 5 and access next data file
GOTO 5
10 errnum=ERR
REM Error trap of 5 secs
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=5
GOTO 6
20 PRINT £p1
REM Accessing of files containing Raw Material usages
OPEN £resdat1,"/d1/resdat"
READ £resdat1,forma,buoh1,ibuoh1,wbuoh1,wibuoh1,afoam,melam,urea

,caus
CLOSE £resdat1
REM Accessing of files for secondary material additions
OPEN £additions1,"/d1/additions"
READ £additions1,buoh2,ibuoh2,acat,xylol
CLOSE £additions1
REM Generation of Raw Materials used on the Log Sheet
PRINT £p1,CHR$(12)
PRINT £p1 USING "'Formalin(litres)',X5,R10.1,X2",forma
PRINT £p1
PRINT £p1 USING "'Butanol(1) (litres)',x5,r10.1,x2",buoh1
PRINT £p1
PRINT £p1 USING "'Butanol(2) (litres)',x5,r10.1,x2",buoh2
PRINT £p1
PRINT £p1 USING "'Iso-Butanol(1) (litres)',x5,r10.1,x2",ibuoh1
PRINT £p1
PRINT £p1 USING "'Iso-Butanol(2) (litres)',x5,r10.1,x2",ibuoh2
PRINT £p1
PRINT £p1 USING "'Wet Butanol(1) (litres)',x5,r10.1,x2",wbuoh1
PRINT £p1
PRINT £p1 USING "'Wet Iso-Butanol(1) (litres)',x5,r10.1,x2",wibuoh1
```

```
PRINT £p1
PRINT £p1 USING "'Xylol (litres)',x5,r10.1,x2",xylol
PRINT £p1
PRINT £p1 USING "'Acid Catalyst (litres)',x5,r10.1,x2",acat
PRINT £p1
PRINT £p1 USING "'Antifoam Soln (ml)',x5,r10.1,x2",afoam
PRINT £p1
PRINT £p1 USING "'Melamine (Kg)',x5,r10.1,x2",melam
PRINT £p1
PRINT £p1 USING "'Urea (Kg)',x5,r10.1,x2",urea
40 CLOSE £p1
END
```

FIGURE A3.8 Procedure 'transfer'



```
PROCEDURE transfer
REM ***************************************************************
REM *                                                              *
REM * Procedure which transmits process values to the BBC computer *
REM *                                                              *
REM *                                                              *
REM * for display in the graphics mode                             *
REM *                                                              *
REM ***************************************************************
REM
REM Assigning data types and variable arrays
REM
DIM data1,process1,ad1,pq1,finish1:BYTE
DIM rtime(9):STRING
DIM x(12),nolen(12):INTEGER
DIM y(12),a(16),avad(10),b(10):REAL
DIM yx(12):REAL
DIM xstr$(12),strlen$(12),charval$(5)
DIM chan,dat,i,dat1:INTEGER
ON ERROR GOTO 55
k=1
vad=1
d1=0
REM Data acquisition from the A-D convertor
5 OPEN £ad1,"/anlg"
BASE 0
FOR chan=0 TO 15
PUT £ad1,chan
GET £ad1,dat
a(chan)=dat
NEXT chan
CLOSE £ad1
REM Conversion of data into eng units
stillt=.0242*a(0)+30.
steamf=.367*a(3)
coilp=.0009997*a(4)
cowatr=.01326*a(5)
cowatf=.01226*a(6)
vacu=.2466*a(7)
cttemp=.01226*a(8)+55.
distt=.01226*a(9)
adflow=.37121*a(10)
refluxr=.7424*a(11)
wetsolr=.7424*a(12)
REM Averaging routine for A.D. tank weight value
IF k<=10 THEN
avad(k-1)=a(1)
ELSE
FOR j=0 TO 8
b(j)=avad(j+1)
NEXT j
FOR j=0 TO 8
avad(j)=b(j)
NEXT j
avad(9)=a(1)
ENDIF
IF k<9 THEN
ad=0
FOR j=0 TO k-1
ad=ad+avad(j)
```

```
NEXT j
adrem=ad/k
ENDIF
IF k>=9 THEN
ad=0
FOR j=0 TO 9
ad=ad+avad(j)
NEXT j
adrem=ad/10
ENDIF
14 BASE 1
REM Assigning values to an array
y(1)=stillt
y(2)=adrem
y(3)=steamf
y(4)=coilp
y(5)=cowatr
y(6)=cowatf
y(7)=vacu
y(8)=cttemp
y(9)=distt
y(10)=adflow
y(11)=refluxr
y(12)=wetsolr
REM Converting process values to integers
FOR i=1 TO 12
yx(i)=y(i)*10
x(i)=INT(yx(i))
NEXT i
IF yx(2)>17465. THEN
x(2)=17465
ENDIF
REM Breakdown of Process values into Individual characters
FOR i=1 TO 12
xstr$(i)=STR$(x(i))
nolen(i)=LEN(xstr$(i))
strlen$(i)=STR$(nolen(i))
FOR j=1 TO nolen(i)
charval$(j)=MID$(xstr$(i),j,1)
NEXT j
REM Transmission of characters to display computer
OPEN £data1,"/dtr":WRITE
PRINT £data1,strlen$(i)
FOR j=1 TO nolen(i)
PRINT £data1,charval$(j)
NEXT j
CLOSE £data1
NEXT i
REM Timing loop for ten second delay
15 s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=10
k=k+1
GOTO 5
20 BASE 1
rtime(9)=MID$(DATE$,10,8)
```

```
GOTO 60
REM 5 second error trap for accessing delays
55 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+1
ENDIF
UNTIL s-s1>=5
GOTO 5
60 END
```

FIGURE A3.9 Procedure 'inout'

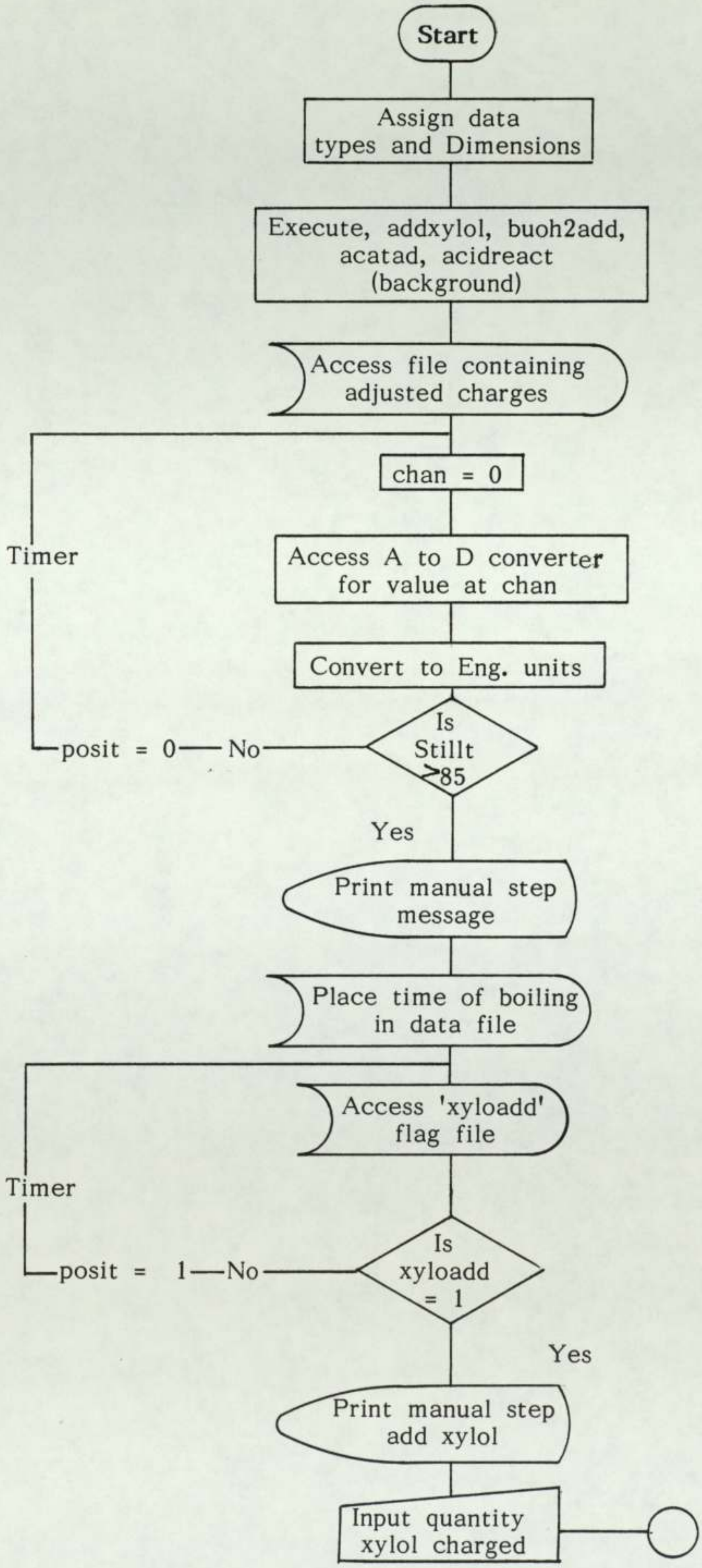


FIGURE A3.9 Procedure 'inout' (Continued)

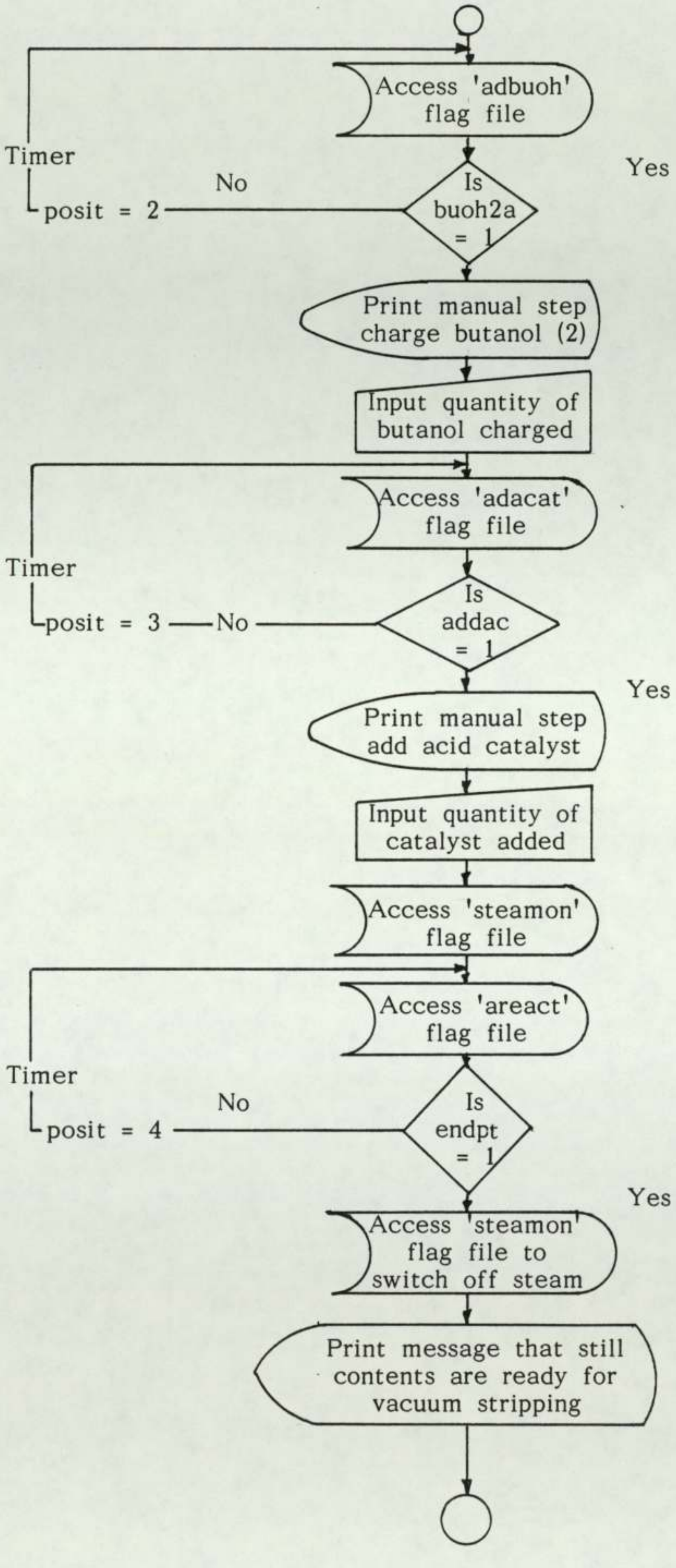
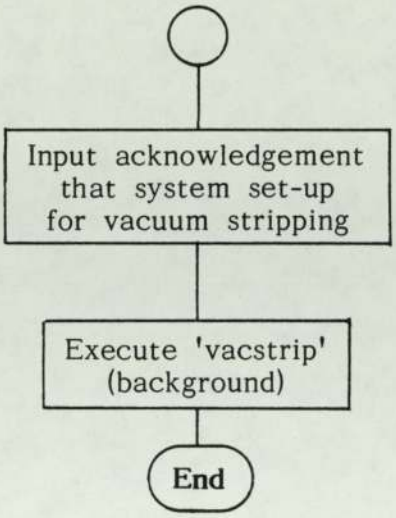


FIGURE A3.9 Procedure 'inout' (C'td)



```
PROCEDURE inout
REM **********************************************************
REM *                                                        *
REM * Procedure to monitor the flag files and provide the    *
REM *                                                        *
REM * Operator interface with the system by generating       *
REM *                                                        *
REM * messages and prompts                                   *
REM *                                                        *
REM **********************************************************
REM
REM
REM Definition of variables and arrays
DIM boil1,ad1,adacat1,adjust1,areact1,additions1:BYTE
DIM adbuoh1,adxylo1,steamon1,column_on1:BYTE
DIM ans(1),an(1):STRING
DIM typr(3):STRING
DIM chan,dat,dat1:INTEGER
DIM stval,toptemp:INTEGER
ON ERROR GOTO 300
REM Data retrieval for material additions and water off quantities
OPEN £adjust1,"/d1/adjust"
READ £adjust1,xylo1,ibuoh2,buoh2,typr(3),addwater,reactwat
CLOSE £adjust1
CREATE £additions1,"/d1/additions"
REM Initiation of procedure to indicate point for Xylol addition
SHELL "addxylol&"
5 chan=0
posit=0
REM Access A-D convertor channel 0
OPEN £ad1,"/anlg"
BASE 0
PUT £ad1,chan
GET £ad1,dat
CLOSE £ad1
BASE 1
REM Convert digital value to Still Temp
stillt=.0242*dat+30.
REM Test to see if still temp > 80 degC
IF stillt>80. THEN
stval=5
REM If condition met steam pressure flag file accessed
REM and pressure dropped to 0.5 bar
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
REM Prompt for manual check to determine contents of still boiling
PRINT
PRINT "Check the still contents to see if boiling has started"
PRINT
PRINT "When the contents are boiling, enter b(boiling)"
PRINT
REM Acknowledgement of boiling commencement
6 INPUT "Answer is    -    ",ans(1)
PRINT
IF ans(1)="b" THEN
REM Methylolation stage, 15 min reflux
PRINT "The contents should now be boiled for 15 mins, with the reflux"
PRINT
PRINT "being returned to the still"
```

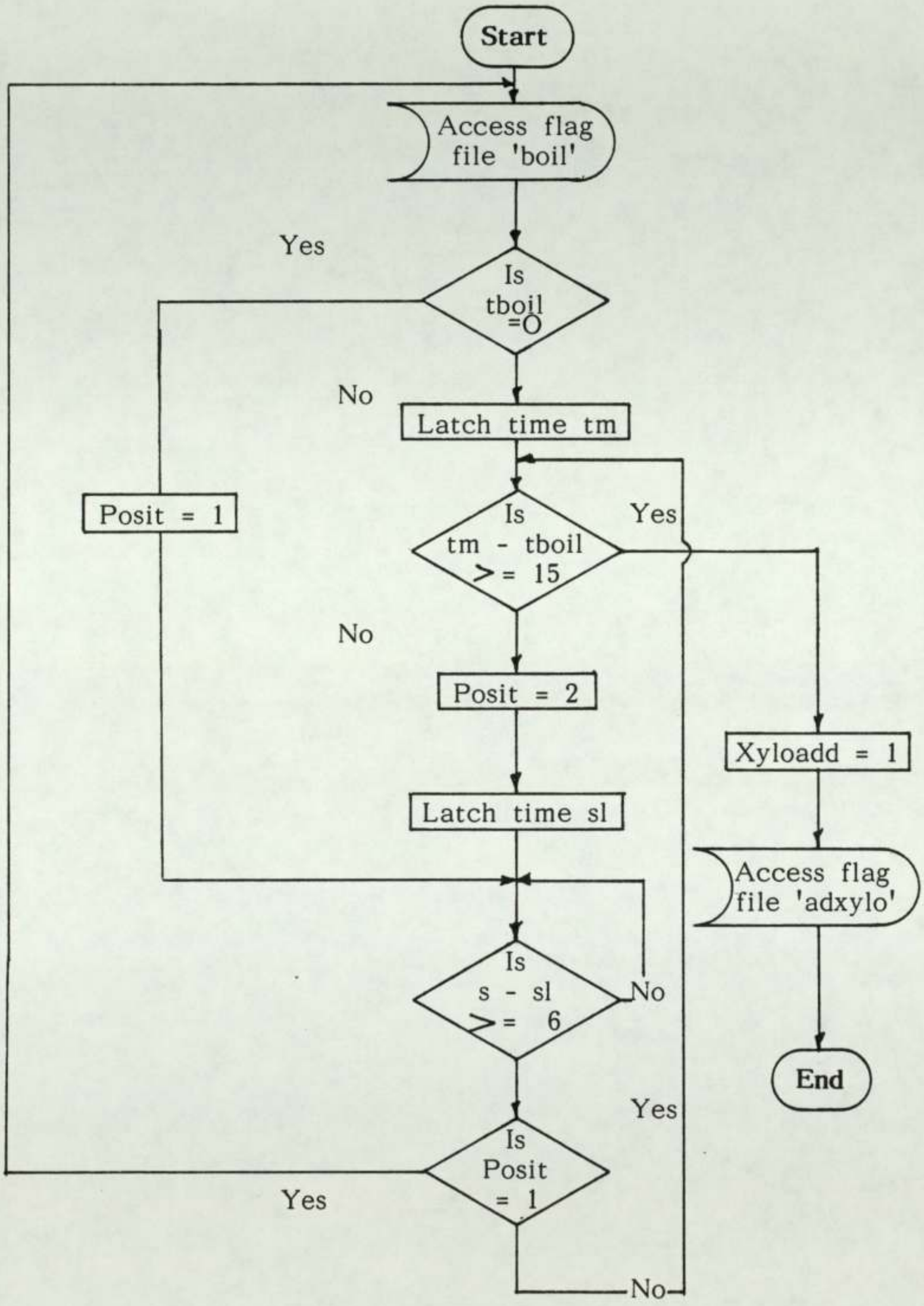
```
ELSE
GOTO 6
ENDIF
REM Time of boiling placed in data file
rm=VAL(MID$(DATE$,13,2))
rmb=VAL(MID$(DATE$,10,2))*60
tboil=rm+rmb
OPEN £boil1,"/d1/boil"
WRITE £boil1,tboil
CLOSE £boil1
REM Move to next stage of process
GOTO 10
ENDIF
REM Process position marker for returning to correct point
REM after entering timing loop
posit=0
GOTO 100
10 posit=1
REM Access Xylol addition flag file
OPEN £adxylo1,"/d1/adxylo"
READ £adxylo1,xyloadd
CLOSE £adxylo1
IF xyloadd=1 THEN
REM When value has changed prompt generated for material addition
PRINT "The still contents have been boiling for 15 mins, the Xylol should now be
 added"
PRINT
PRINT "The adjusted Xylol charge is  -"; TAB(50); xylol; "  Litres"
PRINT
REM Acknowledge charge by entering quantity
15 INPUT "Xylol used  -  ",xylol
PRINT
PRINT "Do you wish to change this value"
PRINT
PRINT "Enter y(yes),n(no)"
PRINT
INPUT "Answer is  -  ",ans(1)
IF ans(1)="y" THEN 15
REM Steam pressure increased to proceed reaction
stval=1
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
REM Initiate Butanol(2) addition procedure
SHELL "buoh2add&"
GOTO 20
ELSE
posit=1
GOTO 100
ENDIF
REM Access Butanol(2) addition flag file
20 posit=2
OPEN £adbuoh1,"/d1/adbuoh"
READ £adbuoh1,buoh2a
CLOSE £adbuoh1
REM Generate prompt for Butanol(2) addition
IF buoh2a=1 THEN
PRINT
PRINT "The still contents have been boiling for 3 hrs"
PRINT
```

```
PRINT "It is now time for the butanol(2) addition to the still"
PRINT
IF ibuoh2>0 THEN 25
PRINT
PRINT "The adjusted n-Buoh(2) charge is   -  "; buoh2
PRINT
REM Enter quantity of Butanol charged
24 INPUT "n-Butanol(2) added  -  ",buoh2
PRINT
PRINT "Do you wish to change this value"
PRINT
PRINT "Enter y(yes),n(no)"
PRINT
INPUT "Answer is  -  ",ans(1)
IF ans(1)="y" THEN 24
ibuoh2=.0
GOTO 28
REM Enter quantity of Butanol charged
25 PRINT "The adjusted iso-Buoh(2) charge is  -  "; ibuoh2
PRINT
26 INPUT "Iso-Butanol(2) used   -  ",ibuoh2
PRINT
PRINT "Do you wish to change this value"
PRINT
PRINT "Enter y(yes), n(no)"
PRINT
INPUT "Answer is  -  ",ans(1)
PRINT
IF ans(1)="y" THEN 26
buoh2=.0
REM Initiate Acid Catalyst addition procedure
28 SHELL "acatad&"
GOTO 30
ELSE
posit=2
GOTO 100
ENDIF
30 posit=3
REM Access Acid Catalyst addition flag file
OPEN £adacat1,"/d1/adacat"
READ £adacat1,addac
CLOSE £adacat1
IF addac=1 THEN
REM Generate prompt for addition of acid catalyst
PRINT
PRINT "The still contents have reached 102 C and the water removed has attained
the required amount"
PRINT
PRINT "The Acid Catalyst should now be added"
PRINT
PRINT "When the catalyst has been added enter the amount used below"
PRINT
REM Acknowledgement of catalyst charged
35 INPUT "Acid Catalyst used  -  ",acat
PRINT
PRINT "Do you wish to change this value"
PRINT
PRINT "Enter y(yes), n(no)"
PRINT
INPUT "Answer is  -  ",ans(1)
```

```
IF ans(1)="y" THEN 35
stval=2
REM Adjustment of steam pressure for next stage of process
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
toptemp=1
REM Setting of column top temp. controller value
OPEN £column_on1,"/d1/columnt"
WRITE £column_on1,toptemp
CLOSE £column_on1
REM Initiation of acid reaction monitoring procedure
SHELL "acidreact&"
OPEN £additions1,"/d1/additions"
WRITE £additions1,buoh2,ibuoh2,acat,xylo1
CLOSE £additions1
GOTO 40
ELSE
posit=3
GOTO 100
ENDIF
40 posit=4
REM Accessing of acid reaction stage flag file
OPEN £areact1,"/d1/areact"
READ £areact1,endpt
CLOSE £areact1
IF endpt=1 THEN
REM Generation of message at end of acid reaction stage
PRINT
PRINT "The required amount of Aqueous Distillate has been removed"
PRINT
PRINT "during the Acid Reaction"
PRINT
PRINT "The Still contents should now be Vacuum Stripped"
GOTO 200
ELSE
posit=4
GOTO 100
ENDIF
REM Timing routine of one minute interval
100 s1=VAL(MID$(DATE$,13,2))
REPEAT
s=VAL(MID$(DATE$,13,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=1
IF posit=0 THEN 5
IF posit=1 THEN 10
IF posit=2 THEN 20
IF posit=3 THEN 30
IF posit=4 THEN 40
200 PRINT
REM Adjustment of steam pressure before Vacuum Stripping resin
stval=10
PRINT "When Vacuum Stripping has commenced acknowledge by entering v(vac)"
PRINT
REM Commencement of Vacuum Stripping
INPUT "Vacuum Stripping started  -  ",an(1)
PRINT "Vacuum stripping commenced"
```

```
PRINT
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
GOTO 350
REM Error trap routine for accessing errors
300 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=5
IF posit=0 THEN 5
IF posit=1 THEN 10
IF posit=2 THEN 20
IF posit=3 THEN 30
IF posit=4 THEN 40
350 END
```

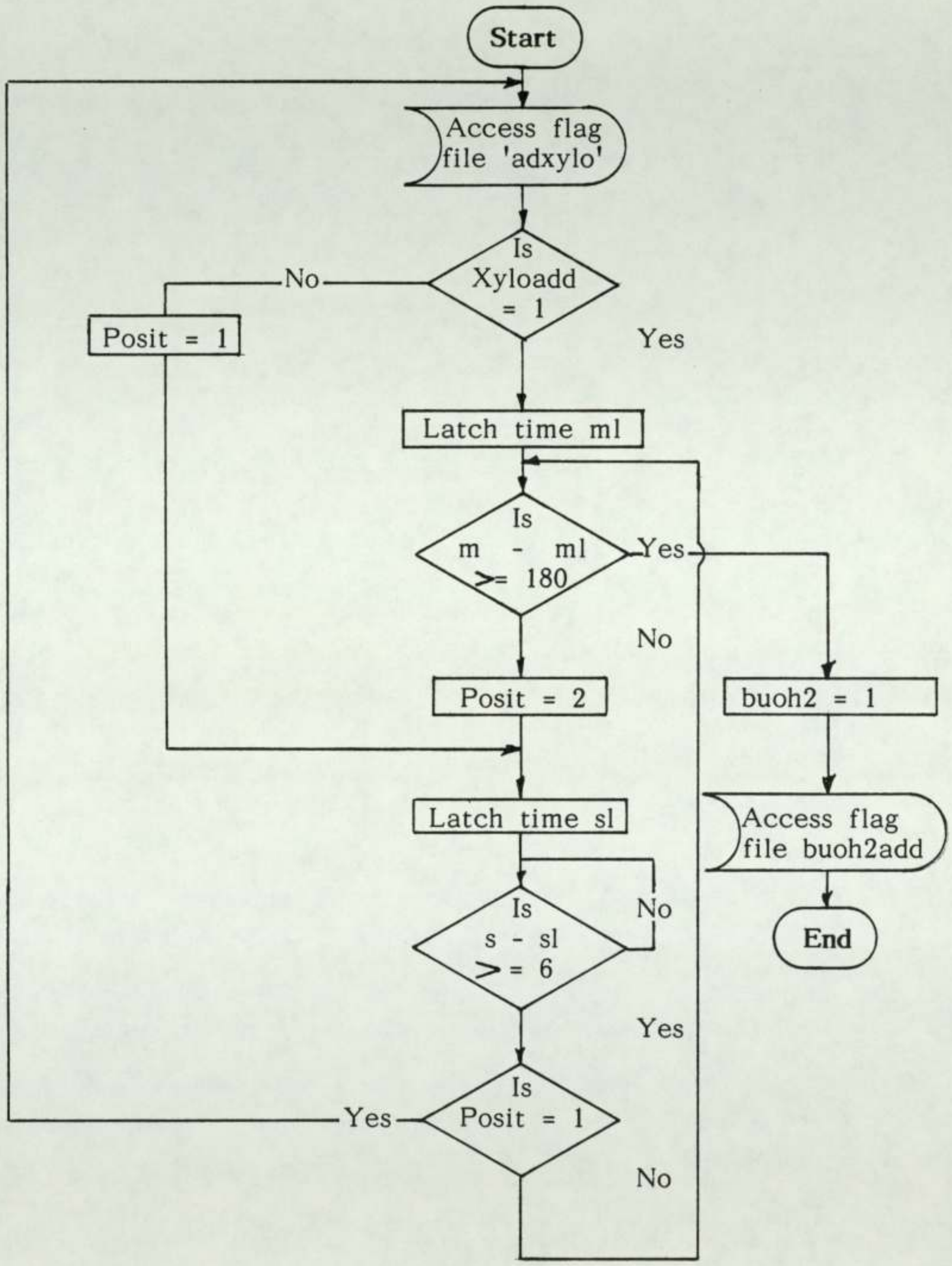
FIGURE A3.10 Procedure 'addxylol'



```
PROCEDURE addxylol
REM
REM Procedure for indicating the addition point for
REM
REM
REM the Xylol charge. This is a timing routine from
REM
REM the time boiling has occured
REM
REM
REM Definition of variables
DIM boil1,adxylol:BYTE
ON ERROR GOTO 20
mhb=0
REM Accessing of boil flag file to detect onset of boiling
REM and obtain boiling time in minutes
1 OPEN £boil1,"/d1/boil"
READ £boil1,tboil
CLOSE £boil1
REM Position indicator
posit=1
REM Check to see if boiling has occurred
IF tboil=0 THEN 7
REM Fixing of current time in minutes
5 m=VAL(MID$(DATE$,13,2))
mh=VAL(MID$(DATE$,10,2))*60
IF mh<mhb THEN
mha=1440
ELSE
mha=0
ENDIF
REM Position indicator
posit=2
tm=m+mh+mha
mhb=mh+mha
REM If time elapsed then goto step 10
IF tm-tboil>=15 THEN 10
REM One minute timing loop
7 s1=VAL(MID$(DATE$,13,2))
REPEAT
s=VAL(MID$(DATE$,13,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=1
REM Return operation to correct point in procedure
IF posit=1 THEN 1
GOTO 5
REM Accesing flag file adxylo to indicate Xylol should be added
10 xyload=1
OPEN £adxylol,"/d1/adxylo"
WRITE £adxylol,xyload
CLOSE £adxylol
GOTO 30
REM Error trap for accessing errors
20 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
```

```
s=s+60
ENDIF
UNTIL s-s1>=5
GOTO 1
30 END
```

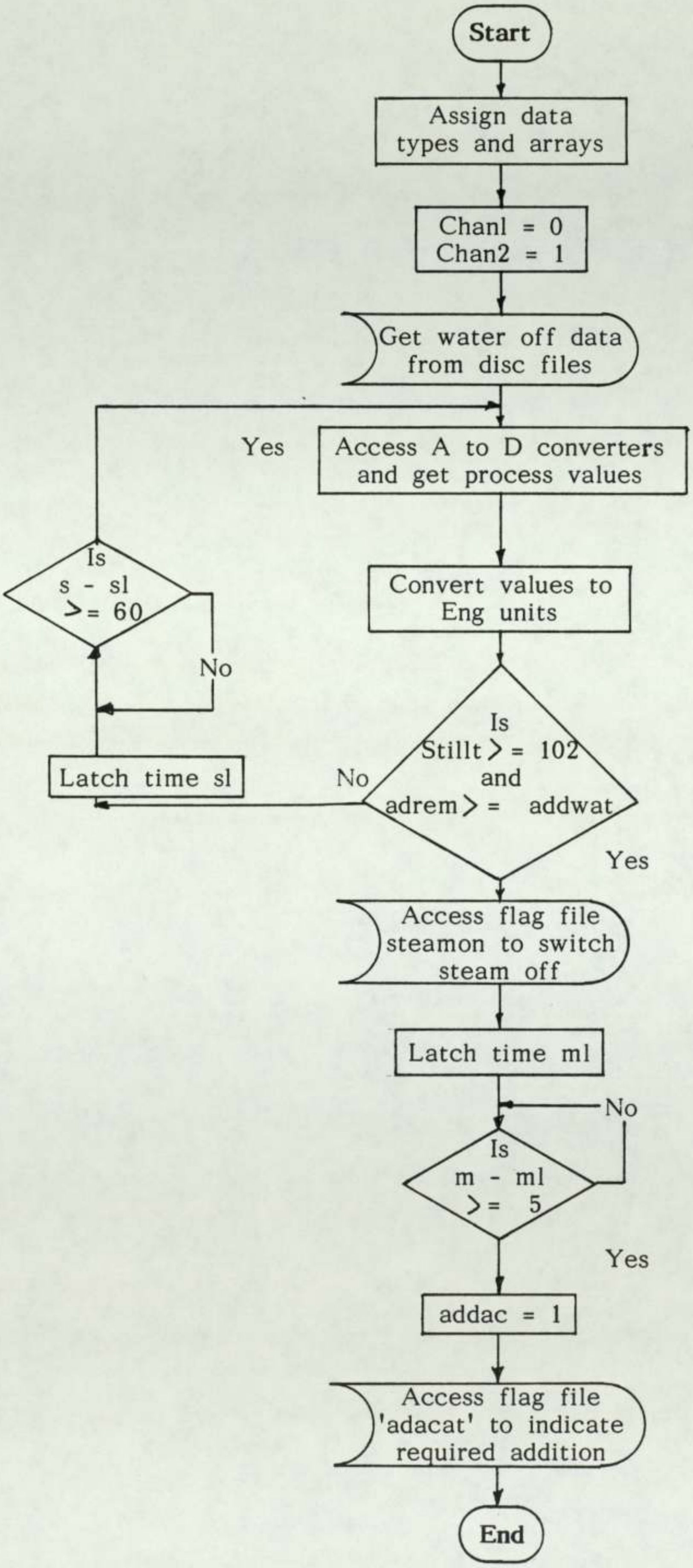
FIGURE A3.11 Procedure 'buoh2add'



```
PROCEDURE buoh2add
REM
REM Procedure to indicate the point when the second Butanol
REM
REM charge should be made. This is a timing routine.
REM
REM
REM Definition of variables
DIM adbuoh1,boil1:BYTE
REM Error trap subroutine
ON ERROR GOTO 8
REM Accessing of flag file boil to indicate onset of boiling
REM and obtain time of boiling in minutes
1 OPEN £boil1,"/d1/boil"
READ £boil1,tboil
CLOSE £boil1
REM Position indicator
posit=1
REM Test to see if boiling has occurred
IF tboil=0 THEN 7
REM Fixing current time in minutes
5 m=VAL(MID$(DATE$,13,2))
mh=VAL(MID$(DATE$,10,2))*60
IF mh<mhb THEN
mha=1440
ELSE
mha=0
ENDIF
tm=m+mh+mha
mhb=mh+mha
REM Position indicator
posit=2
REM Test to see if time has elapsed
IF tm-tboil>=195 THEN
buoh2a=1
REM If time has elapsed then value in flag file adbuoh is
REM changed to indicate that the Butanol(2) charge should
REM be made
OPEN £adbuoh1,"/d1/adbuoh"
WRITE £adbuoh1,buoh2a
CLOSE £adbuoh1
GOTO 10
ENDIF
REM One minute timing loop
7 sm1=VAL(MID$(DATE$,13,2))
REPEAT
sm=VAL(MID$(DATE$,13,2))
IF sm<sm1 THEN
sm=sm+60
ENDIF
UNTIL sm-sm1>=1
IF posit=1 THEN 1
GOTO 5
REM Five second timing loop to trap disc access errors
8 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+1
```

```
ENDIF
UNTIL s-s1>=5
GOTO 1
10 END
```

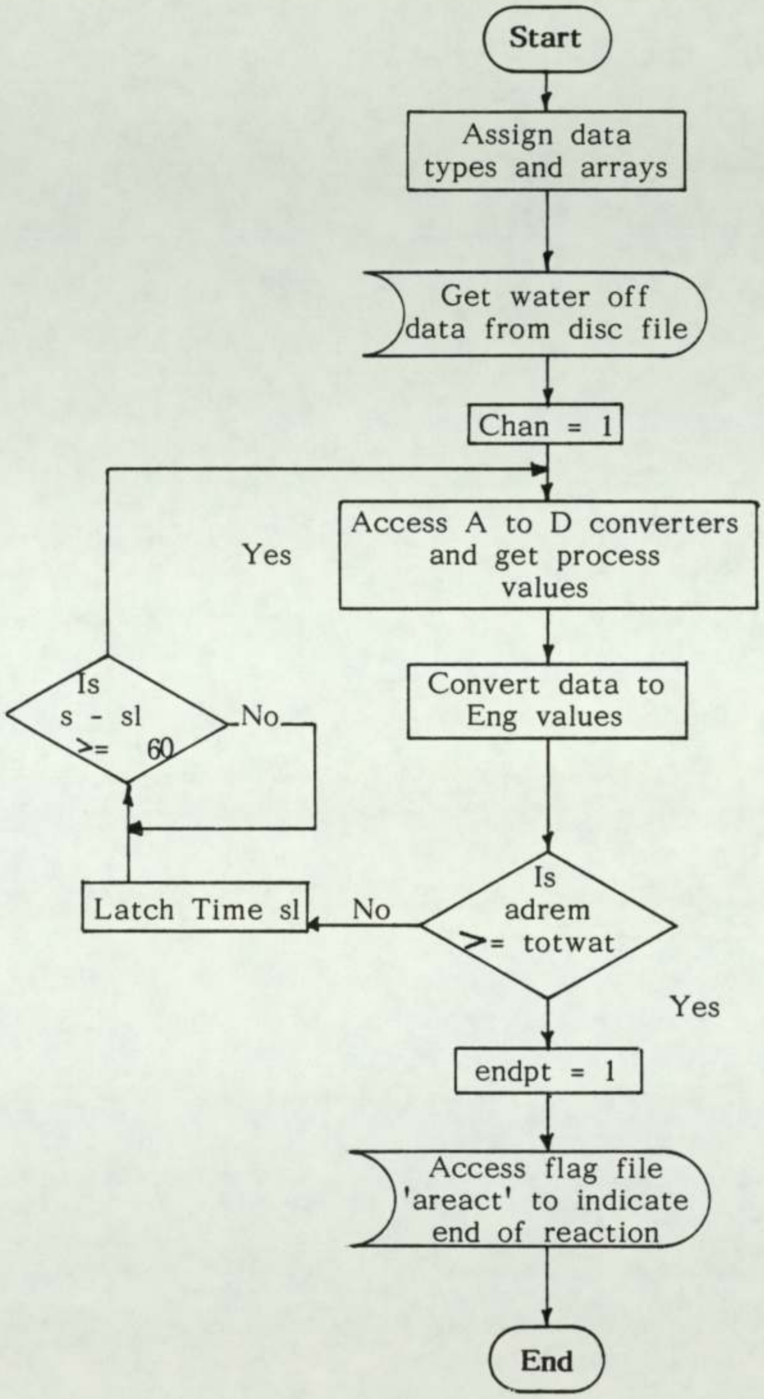
FIGURE A3.12 Procedure 'acatad'



```
PROCEDURE acatad
REM
REM Procedure to indicate the point for the Acid Catalyst addition
REM
REM Definition of variables
DIM ad1,adacat1,adjust1,steamon1:BYTE
DIM chan1,chan2,dat1,dat2,stval,dat3,dat4:INTEGER
DIM typr(3):STRING
DIM avad(10),b(10):REAL
REM Disc accessing error trap
ON ERROR GOTO 7
k=1
REM Definition of A-D channels
chan1=0
chan2=1
REM Accessing of data file adjust to obtain the quantities
REM of water to be removed during this stage
1 OPEN £adjust1,"/d1/adjust"
READ £adjust1,ax,bs,c,typr(3),addwat,totwat
CLOSE £adjust1
REM Accessing of A-D converter to obtain digital values
5 OPEN £ad1,"/anlg"
BASE 0
PUT £ad1,chan1
GET £ad1,dat1
PUT £ad1,chan2
GET £ad1,dat2
CLOSE £ad1
BASE 1
REM Conversion of digital values to engineering values
stillt=.0242*dat1+30.
REM Loop to average the previous ten values for the Aqueous
REM Distillate Tank Weight
IF k<=10 THEN
avad(k)=dat2
ELSE
FOR j=1 TO 9
b(j)=avad(j+1)
NEXT j
b(10)=dat2
ENDIF
IF k<10 THEN
ad=0
FOR j=1 TO k
ad=ad+avad(j)
NEXT j
dat=ad/k
ELSE
ad=0
FOR j=1 TO 10
ad=ad+b(j)
NEXT j
dat=ad/10
ENDIF
REM Conversion of the Aqueous Distillate digital value
adrem=2.004*dat
IF k=1 THEN
value1=adrem
ENDIF
k=k+1
```

```
REM Test to see if a spiked value has been received by the system
REM If one has, then previous AD value used
IF adrem>value1+value1/5 THEN
adrem=value1
ENDIF
value1=adrem
REM Test to see if required temperature and mass of water removed
REM has been achieved
IF stillt>=103. AND adrem>=addwat-addwat*.05 THEN
stval=5
REM Altering value in steam pressure flag file
OPEN £steamon1,"/d1/steamon"
WRITE £steamon1,stval
CLOSE £steamon1
REM Timing loop of five minutes
m1=VAL(MID$(DATE$,13,2))
REPEAT
m=VAL(MID$(DATE$,13,2))
IF m<m1 THEN
m=m+60
ENDIF
UNTIL m-m1>=5
addac=1
REM Accessing flag file adacat to alter the flag value to indicate
REM that the acid catalyst should now be added
OPEN £adacat1,"/d1/adacat"
WRITE £adacat1,addac
CLOSE £adacat1
REM End procedure
GOTO 10
ENDIF
REM One minute timing loop
s1=VAL(MID$(DATE$,13,2))
REPEAT
s=VAL(MID$(DATE$,13,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=1
GOTO 5
REM Disc accessing error trap
7 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=5
IF k=1 THEN 1
GOTO 5
10 END
```

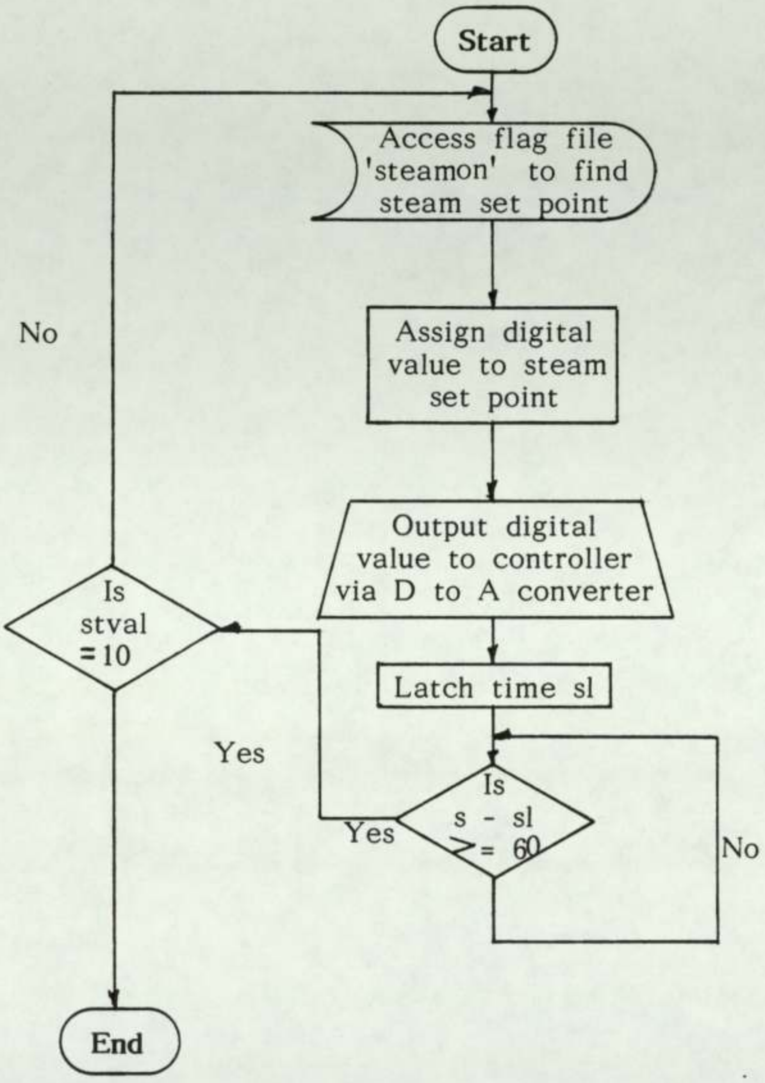
FIGURE A3.13 Procedure 'acidreact'



```
PROCEDURE acidreact
REM
REM Procedure to indicate the end of the Acid Reaction stage
REM
REM This procedure monitiors the Mass of Aqueous Distillate
REM
REM removed for the reaction
REM
REM
REM Definition of variables
DIM ad1,areact1,adjust1:BYTE
DIM dat,chan,dat1:INTEGER
DIM typr(3):STRING
DIM avad(10),b(10):REAL
REM Disc accessing error trap
ON ERROR GOTO 7
k=1
REM Accessing flag file adjust to obtain mass of water to be
REM removed
1 OPEN £adjust1,"/d1/adjust"
READ £adjust1,ax,bs,c,typr(3),d,totwat
CLOSE £adjust1
REM Definition of A-D channels
chan=1
REM Accessing of A-D converter
5 OPEN £ad1,"/anlg"
BASE 0
PUT £ad1,chan
GET £ad1,dat
CLOSE £ad1
BASE 1
dat1=dat
REM Averaging routine for Aqueous Distillate Mass
IF k<=10 THEN
avad(k)=dat
ELSE
FOR j=1 TO 9
b(j)=avad(j+1)
NEXT j
b(10)=dat
ENDIF
IF k<10 THEN
ad=0
FOR j=1 TO k
ad=ad+avad(j)
NEXT j
dat1=ad/k
ELSE
ad=0
FOR j=1 TO 10
ad=ad+b(j)
NEXT j
dat1=ad/10
ENDIF
6 adrem=2.004*dat1
IF k=1 THEN
value1=adrem
ENDIF
k=k+1
REM Test to check if spiked value received by A-D converter
```

```
IF adrem>value1+value1/5 THEN
adrem=value1
ENDIF
value1=adrem
REM Test to see if required mass of Aqueous Distillate has been
REM removed
IF adrem>=totwat THEN
endpt=1
REM Altering of value in flag file to indicate end of reaction
OPEN £areact1,"/d1/areact"
WRITE £areact1,endpt
CLOSE £areact1
REM End procedure
GOTO 10
ENDIF
REM One minute timing loop
s1=VAL(MID$(DATE$,13,2))
REPEAT
s=VAL(MID$(DATE$,13,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=1
GOTO 5
REM Disc accessing error timing loop
7 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=5
IF k=1 THEN 1
GOTO 5
10 END
```

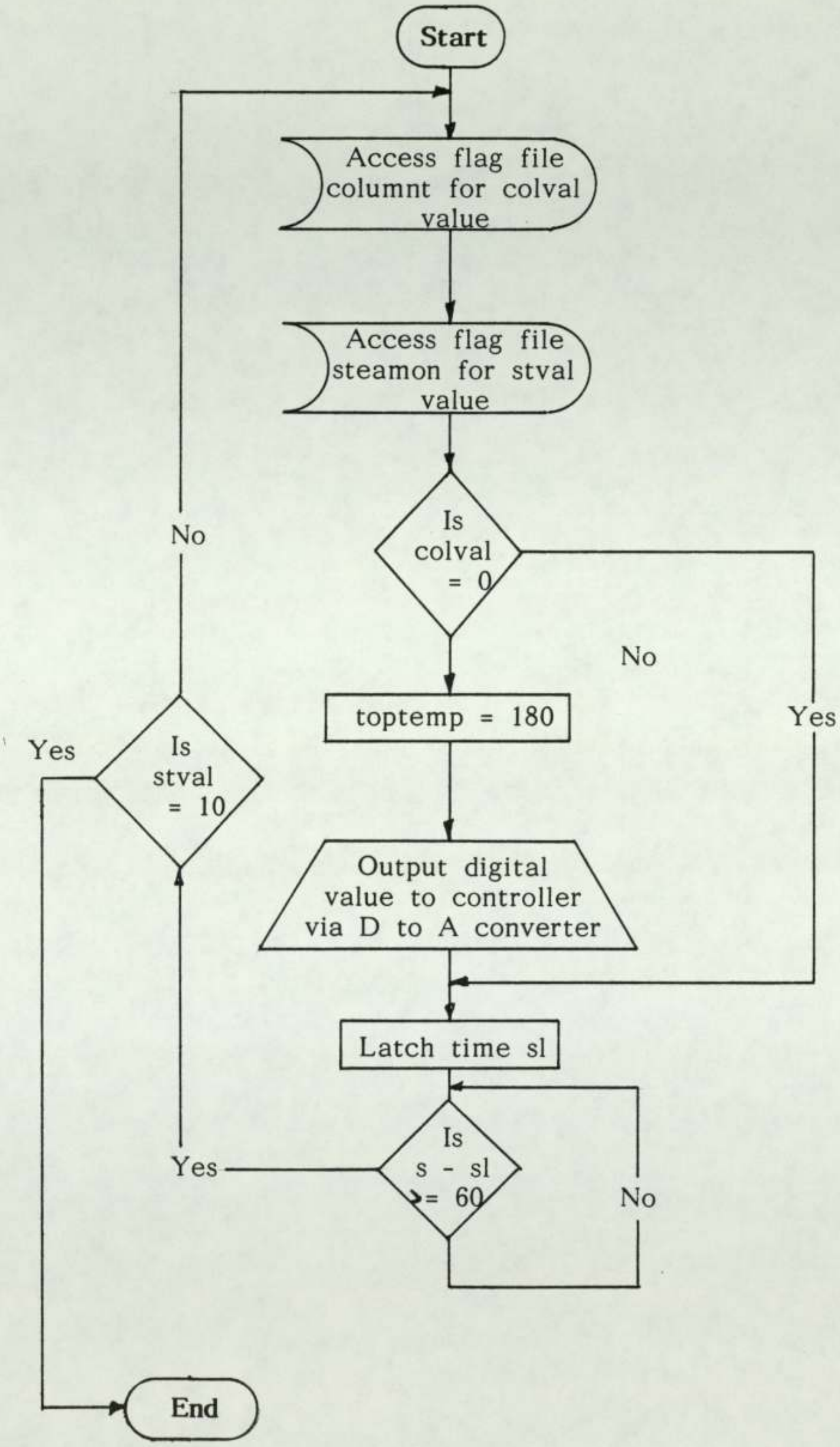
FIGURE A3.14 Procedure 'steamset'



```
PROCEDURE steamset
REM
REM Procedure which adjusts the steam pressure to the reactor
REM
REM based on flag values in files
REM
REM
REM Definition of variables
DIM steamon1,stset1,stpress:BYTE
DIM stval:INTEGER
REM Disc accessing error trap
ON ERROR GOTO 7
REM Accessing flag file steamon to obtain value for
REM steam pressure
5 OPEN £steamon1,"/d1/steamon"
READ £steamon1,stval
CLOSE £steamon1
REM Flag values and their corresponding digital values
IF stval=0 THEN
stpress=0
ENDIF
IF stval=1 THEN
stpress=190
ENDIF
IF stval=2 THEN
stpress=126
ENDIF
IF stval=3 THEN
stpress=0
ENDIF
IF stval=5 THEN
stpress=32
ENDIF
IF stval=10 THEN
stpress=0
ENDIF
REM Accessing D-A converter and sending appropriate values to
REM set point controller
OPEN £stset1,"/da1"
FOR i=1 TO 1000
PUT £stset1,stpress
NEXT i
CLOSE £stset1
REM One minute timing loop
s1=VAL(MID$(DATE$,13,2))
REPEAT
s=VAL(MID$(DATE$,13,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=1
GOTO 5
REM Disc accessing error trap timing loop
7 errnum=ERR
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
```

```
UNTIL s-s1>=5
GOTO 5
10 END
```

FIGURE A3.15 Procedure 'coltop'



```
PROCEDURE coltop
REM
REM Procedure to adjust the Column Top Temperature Controller
REM
REM
REM Definition of variables
DIM column_on1,colset1,vacuum1,toptemp,vacu:BYTE
DIM colval,colval1:INTEGER
REM Disc accessing error trap
ON ERROR GOTO 8
colval1=0
REM Setting value on Column Top Temp Controller to zero
OPEN £colset1,"/da3"
FOR i=1 TO 1000
PUT £colset1,colval1
NEXT i
CLOSE £colset1
REM Accessing flag file columnt to obtain setting for controller
5 OPEN £column_on1,"/d1/columnt"
READ £column_on1,colval
CLOSE £column_on1
REM Test to see if controller requires to be set
IF colval=colval1 THEN 7
IF colval=1 THEN
REM Setting for top temp. controller
toptemp=172
ELSE
toptemp=0
ENDIF
REM Sending set point value to the D-A converter to send out
REM to controller
OPEN £colset1,"/da3"
FOR i=1 TO 1000
PUT £colset1,toptemp
NEXT i
CLOSE £colset1
colval1=colval
REM One minute timing loop
7 s1=VAL(MID$(DATE$,13,2))
REPEAT
s=VAL(MID$(DATE$,13,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=1
GOTO 5
8 errnum=ERR
REM Disc accessing error trap timing loop
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=5
GOTO 5
10 END
```

FIGURE A3.16 Procedure 'vacstrip'

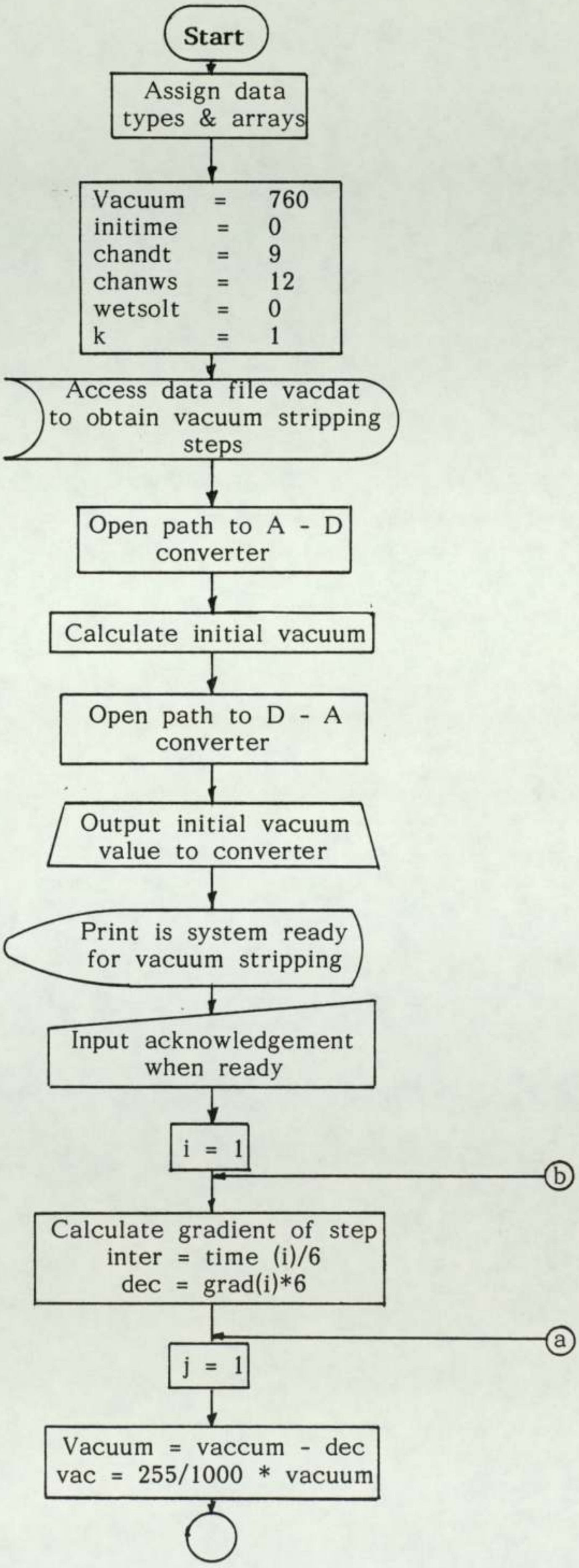
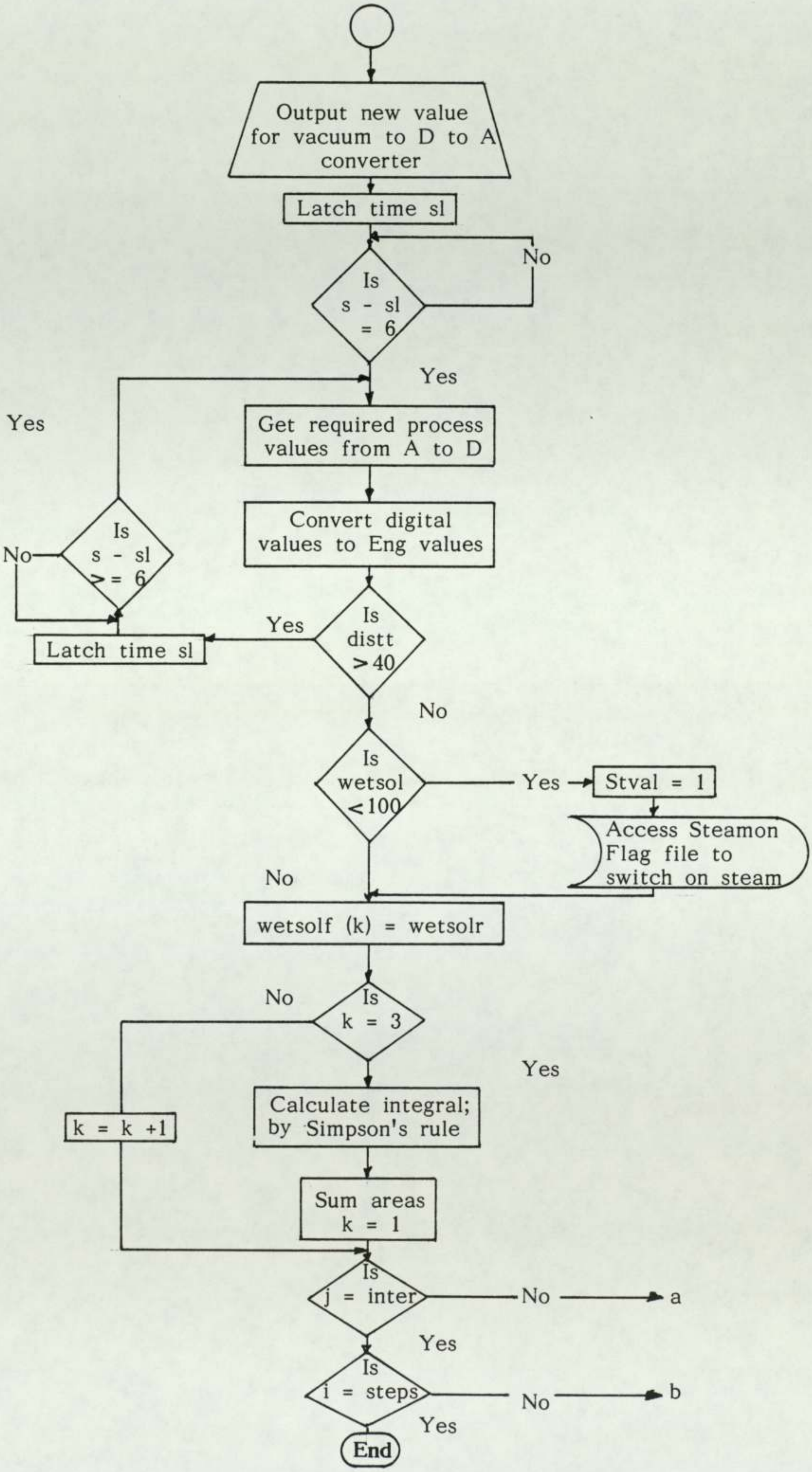


FIGURE A3.16 Procedure 'vacstrip' (C'td)



```
PROCEDURE vacstrip
REM
REM Procedure to Vacuum Strip solvent at reaction temperature
REM
REM without cooling the reactor contents
REM
REM
REM Definition of variables
DIM vac,vac1,vacdat1,ad1,steamon1,stval:BYTE
DIM steps,chandt,chanws:INTEGER
DIM datdt,datws:REAL
DIM time(10),vacu(10),wetsolf(3),grad(10):REAL
DIM an(1):STRING
REM Setting of atmospheric pressure
vacuum=760.
initime=.0
REM Setting of channels on A-D converter
chandt=9
chanws=12
REM Initialising wet solvent flow
wetsolt=.0
k=1
REM Accessing file containing steps for reduction in pressure
OPEN £vacdat1,"/d1/vacdat"
GET £vacdat1,steps
GET £vacdat1,time
GET £vacdat1,vacu
CLOSE £vacdat1
REM Accessing A-D converter
OPEN £ad1,"/anlg"
REM Accessing D-A converter
OPEN £vac1,"/da4"
REM Setting initial digital value for vacuum
vac=255/1000*vacuum
PUT £vac1,vac
5 PRINT "To indicate that system is ready for Vacuum Stripping enter, v"
PRINT
INPUT "Answer is  -  ",an(1)
IF an(1)="v" THEN 10
GOTO 5
REM Calculation of stepwise reductions in pressure with time
10 FOR i=1 TO steps
grad(i)=(vacuum-vacu(i))/(time(i)-initime)
inter=time(i)/6
dec=grad(i)*6
FOR j=1 TO inter
vacuum=vacuum-dec
vac=255/1000*vacuum
REM Output of vacuum to controller
PUT £vac1,vac
REM Six second timing loop
s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=6
REM Obtain Distillate temp and wet solvent flow from A-D converter
PUT £ad1,chandt
```

```
GET £ad1,datdt
PUT £ad1,chanws
GET £ad1,datws
REM Conversion of digital values to engineering values
distt=.01226*datdt
wetsolr=.7424*datws
REM Test to check if distillate temp. to high
IF distt>40. THEN
REM If distillate temp to high then pause in timing loop
REM until value is low enough to proceed
20 s1=VAL(MID$(DATE$,16,2))
REPEAT
s=VAL(MID$(DATE$,16,2))
IF s<s1 THEN
s=s+60
ENDIF
UNTIL s-s1>=6
PUT £ad1,chandt
GET £ad1,datdt
distt=.01226*datdt
IF distt>40 THEN 20
ENDIF
REM Test to see if the wet solvent rate is to low
IF wetsolr<100. THEN
stval=65
REM If solvent rate low then steam turned on at low pressure
OPEN £steamon1,"/da1"
PUT £steamon1,stval
CLOSE £steamon1
ENDIF
REM Calculation of quantity of wet solvent removed
wetsolf(k)=wetsolr
IF k=3 THEN
area=h/3*(wetsolf(1)+4*wetsolf(2)+wetsolf(3))
wetsolt=wetsolt+area
k=0
ENDIF
k=k+1
NEXT j
NEXT i
REM Closing paths to A-D and D-A converters
CLOSE £vac1
CLOSE £ad1
END
```

FIGURE A3.17 BBC Graphics Program (main Program)

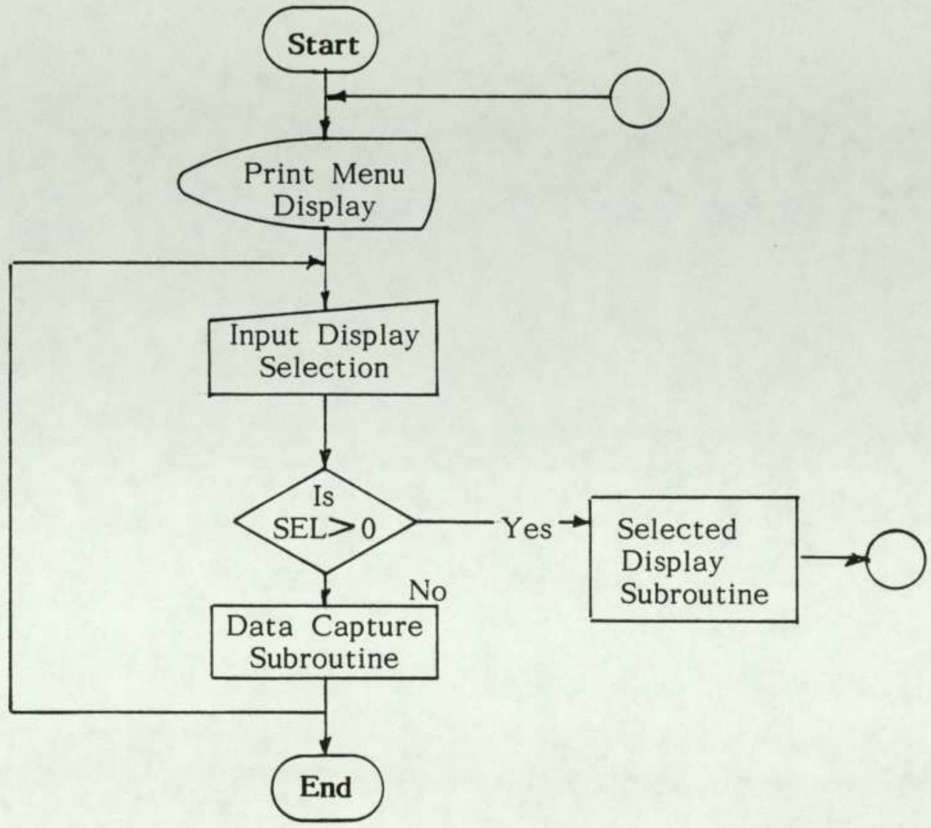


FIGURE A3.18 BBC Graphics Program (Subroutine for Mimic Diagram Display)

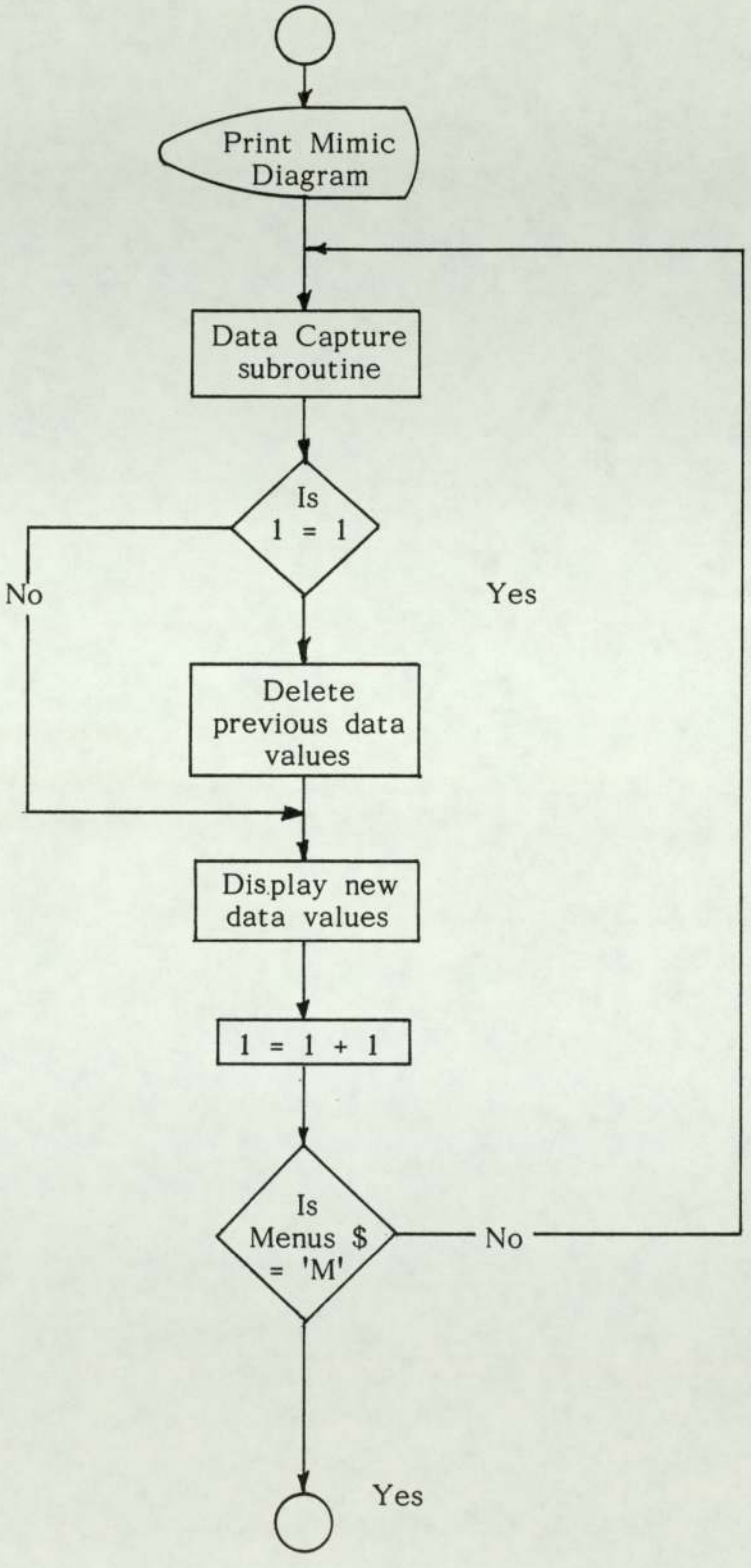


FIGURE A3.19 BBC Graphics Program (Subroutine for Bar Chart Display)

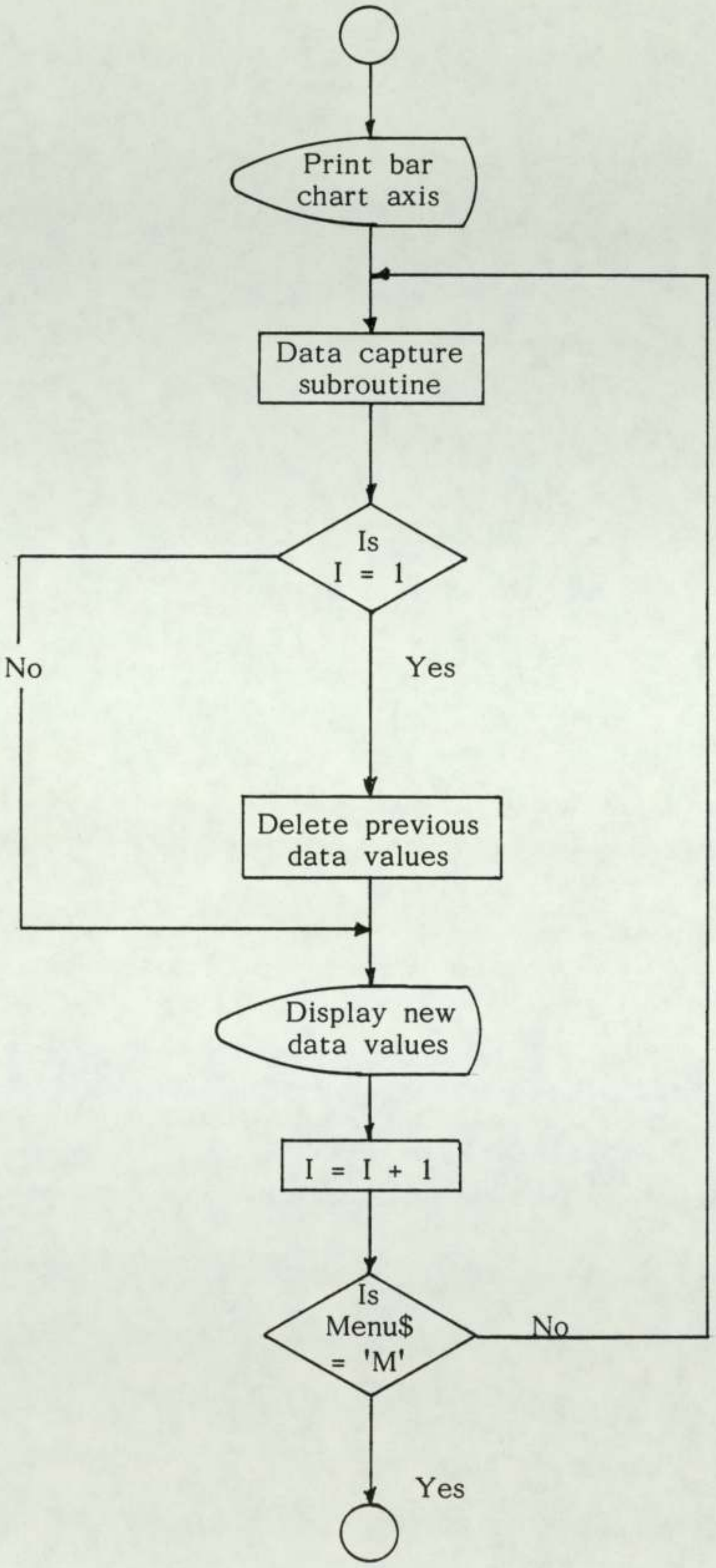


FIGURE A3.20 BBC Graphics Program (Subroutine for Trend Diagram Displays

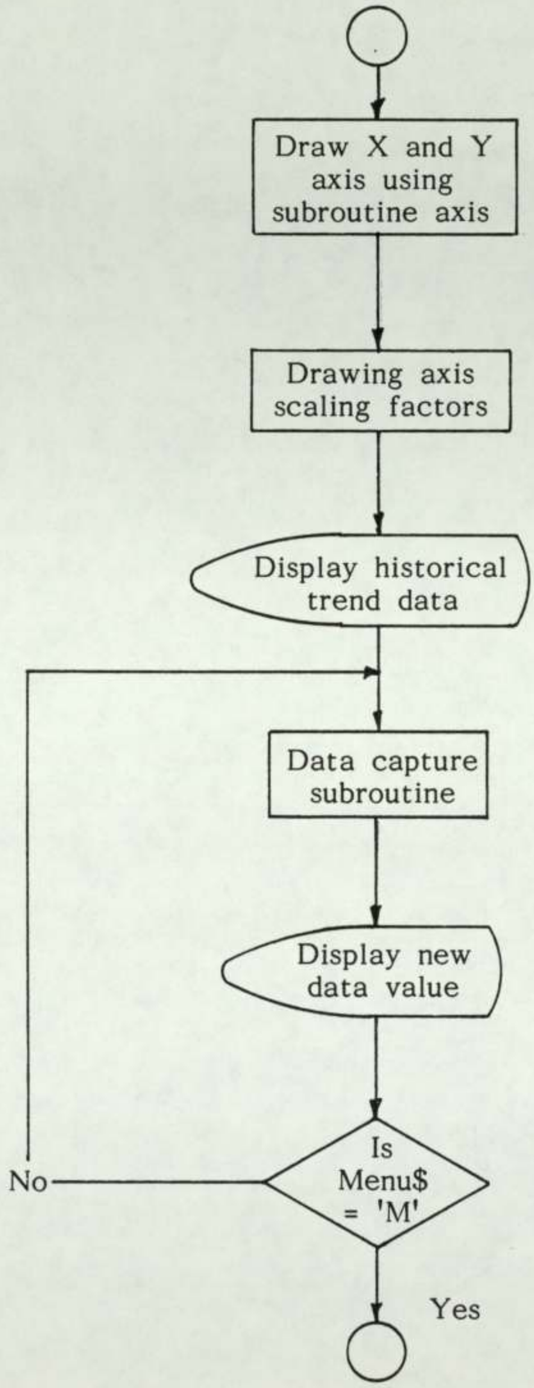
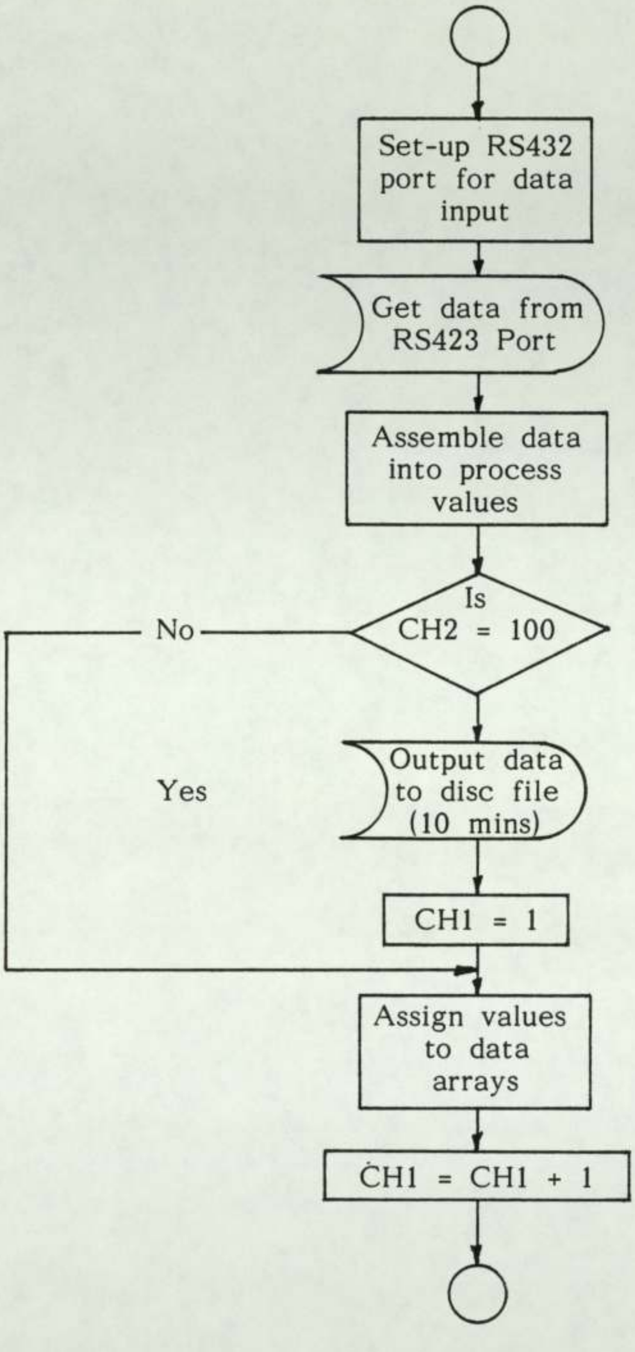


FIGURE A3.21 BBC Graphics Program (Data Capture Subroutine)



```
100 REM **********************************************************
110 REM *                                                        *
120 REM * Procedure to accept packages of data from the SEED-19  *
130 REM *                                                        *
140 REM * Computer and display these in Graphical Form.  The     *
150 REM *                                                        *
160 REM * displays taking the form of mimic diagrams, bar charts *
170 REM *                                                        *
180 REM * or trend diagrams.                                     *
190 REM *                                                        *
200 REM **********************************************************
210 REM
220 REM
230 REM MENU SELECTION ROUTINE
240 REM
250 REM Defining variables and arrays
260 DIM X(5),PROVAL(12),Y(5),OV(5)
270 DIM OFAD(500),VISCRE(500),TEMPS(500)
280 CH1=1
290 SFIL=0
300 REM Setting display parameters for numeric values
310 @%=&0002010A
320 GOSUB 510
330 RSYNC=0
340 SEL$=INKEY$(50)
350 REM Selection of display based on choice made from menu
360 IF SEL$="1" THEN GOSUB 670
370 IF SEL$="2" THEN GOSUB 1320
380 IF SEL$="3" THEN GOSUB 3610
390 IF SEL$="4" THEN GOSUB 3270
400 IF SEL$="5" THEN GOSUB 2800
410 IF RSYNC=1 THEN 320
420 REM Return to data capture routine when selection not made
430 GOSUB 2130
440 GOTO 340
450 END
460 REM
470 REM
480 REM
490 REM Main Menu display subroutine giving optional displays
500 REM
510 MODE 1:VDU 19,0,1;0;:COLOUR 2
520 PRINT TAB(4,3)"MENU OF VARIOUS PROCESS DISPLAYS"
530 PRINT TAB(3,4)"___________________________________ "
540 PRINT TAB(0,7)"To Select Display enter number and WAIT":COLOUR 3
550 PRINT TAB(1,12)"1 Still12 Flowsheet and Temperatures"
560 PRINT TAB(1,14)"2 Key Process Varibles"
570 PRINT TAB(1,16)"3 Temperature Variation"
580 PRINT TAB(1,18)"4 Viscosity Variation"
590 PRINT TAB(1,20)"5 A.D. Variation"
600 REM End of Main Menu subroutine
610 RETURN
620 REM
630 REM
640 REM
650 REM Subroutine creating a Mimic Diagram Display for the Reactor System
660 REM
670 REM Construction of display
680 MODE1:GCOL 0,2:ADREM1=0.:SHAD1=0.
```

```
  690 MOVE 200,200:DRAW 200,400:DRAW 400,400:DRAW 400,200:DRAW 300,100:DRAW 200,
200
  700 DRAW 200,200:GCOL 0,2:MOVE 300,380:DRAW 300,200:MOVE 300,100:DRAW 300,0
  710 MOVE 220,400:GCOL 0,2:DRAW 220,800:DRAW 300,800:DRAW 300,400:MOVE 220,440
  720 GCOL 0,1:DRAW 300,440:DRAW 220,760:DRAW 300,760:DRAW 220,440
  730 MOVE 260,800:DRAW 260,900:DRAW 500,900
  740 GCOL 0,2:DRAW 500,940:DRAW 700,940:DRAW 700,860:DRAW 500,860:DRAW 500,900
  750 MOVE 540,940:GCOL 0,1:DRAW 540,860:MOVE 660,940:DRAW 660,860:MOVE 540,924
  760 DRAW 660,924:MOVE 540,908:DRAW 660,908:MOVE 540,892:DRAW 660,892:MOVE 540,
376
  770 DRAW 660,876:MOVE 520,940:DRAW 520,1000:MOVE 680,860:DRAW 680,800
  780 MOVE 700,900:GCOL 0,1:DRAW 1000,900:DRAW 1000,800
  790 GCOL 0,2:DRAW 950,800:DRAW 950,620:DRAW 1050,620:DRAW 1050,800:DRAW 1000,8
00
  800 GCOL 0,3:MOVE 1050,710:DRAW 950,710:MOVE 1050,780:DRAW 950,780
  810 GCOL 0,1:MOVE 1050,665:DRAW 1100,665:DRAW 1100,500:GCOL 0,2:DRAW 1040,500
  820 DRAW 1040,380:DRAW 1160,380:DRAW 1160,500:DRAW 1100,500:GCOL 0,1
  830 MOVE 1160,400:DRAW 1280,400:MOVE 950,755
  840 DRAW 680,755:DRAW 680,700:GCOL 0,2:DRAW 640,700:DRAW 640,600
  850 DRAW 720,600:DRAW 720,700:DRAW 660,700
  860 GCOL 0,1:MOVE 680,600:DRAW 680,520:DRAW 370,520:DRAW 370,400:MOVE 0,350
  870 DRAW 360,350:DRAW 240,300:DRAW 360,240:DRAW 0,240:VDU5:GCOL 0,3
  880 MOVE 600,400:PRINT "STILL 12":MOVE 1000,850:PRINT "SEPARATOR":MOVE 1130,60
0
  890 PRINT "A.D.":MOVE 1130,550:PRINT"TANK":MOVE 1150,360:PRINT "KG."
  900 MOVE 500,100:VDU5:PRINT"To Return to MENU":MOVE 500,60:PRINT"enter 'M' and
 WAIT":VDU4
  910 MOVE 580,360:DRAW 875,360:K=1
  920 REM
  930 REM
  940 REM Calling Data Capture routine for next package of data
  950 GOSUB 2130
  960 REM Assigning new data variable names
  970 STILLT=PROVAL(1):AD1=INT(PROVAL(2)*20.04):ADREM=AD1/10:STEAMF=PROVAL(3)
  980 COILP=PROVAL(4):CWI=PROVAL(6):CWO=PROVAL(5):VACU=PROVAL(7):COLTT=PROVAL(8)
  990 DISTT=PROVAL(9):ADFLOW=PROVAL(10):REFLUXR=PROVAL(11):WETSOLR=PROVAL(12)
 1000 GOTO 1080
 1010 REM Deletion of old data values from mimic display
 1020 GCOL 0,0:VDU 5
 1030 MOVE 200,150:PRINT STILLT:MOVE 112,800:PRINT COLTT:MOVE 190,1000:PRINT CWO
 1040 MOVE 500,830:PRINT CWI:MOVE 700,950:PRINT DISTT :MOVE 800,360:PRINT ADREM
 1050 REM Deletion of filled in tank level display
 1060 VDU 4
 1070 GOTO 970
 1080 B=ADREM*120/3500:SHAD=385+B
 1090 IF K=1 THEN 1150
 1100 GCOLO,0:VDU 4
 1110 IF ADREM<ADREM1 THEN
 1120 MOVE 1045,SHAD:MOVE 1155,SHAD:PLOT 85,1155,SHAD1:MOVE1045,SHAD1:PLOT 85,10
45,SHAD
 1130 ELSE
 1140 REM Displaying of new data values on mimic diagram
 1150 VDU5:GCOL 0,3
 1160 MOVE 200,150:PRINT STILLT:MOVE 112,800:PRINT COLTT:MOVE 190,1000
 1170 REM Shading in of new tank level on diagram
 1180 MOVE 800,360:PRINT ADREM:VDU 4:GCOLO,1:IF SHAD>495 THEN SHAD=495
 1190 MOVE 1045,385:MOVE 1155,385:PLOT 85,1155,SHAD:MOVE 1045,SHAD:PLOT 85,1045,
385
 1200 ADREM1=ADREM:SHAD1=SHAD
 1210 REM Check to see if change of display is required
```

```
1220 MENU$=INKEY$(50):IF MENU$="M" THEN 1280
1230 K=2
1240 REM Access Data Capture routine for next package of data
1250 GOSUB 2130
1260 REM Return to start of routine to refresh data
1270 GOTO 1020
1280 RSYNC=1:RETURN
1290 REM
1300 REM
1310 REM
1320 REM Display of main process variables in bar chart form
1330 REM
1340 REM Construction of Bar Chart Axis and Titles
1350 MODE 0:VDU 19,0,6,0,0,0:VDU 19,7,1,0,0,0:VDU 5:GCOL 0,7
1360 MOVE 120,900:PRINT"STILL":MOVE 120,852:PRINT"TEMP":MOVE 120,804:PRINT"    D
EGC"
1370 MOVE 420,900:PRINT"A.D.":MOVE 380,852:PRINT"REMOVED":MOVE 380,804 :PRINT"
   KG"
1380 MOVE 640,900:PRINT"REACTION":MOVE 640,852:PRINT"TIME":MOVE 640,804:PRINT"
 HRS"
1390 MOVE 900,900:PRINT"WET SOLV":MOVE 900,852:PRINT"  OFF":MOVE 900,804:PRINT"
    L"
1400 MOVE 1120,852:PRINT"VISCOSITY":MOVE 1160,804:PRINT"   CP"
1410 MOVE 68,200:PRINT"30":MOVE 68,300:PRINT"50":MOVE 68,400:PRINT"70"
1420 MOVE 68,500:PRINT"90":MOVE 56,600:PRINT"110":MOVE 56,700:PRINT"130"
1430 MOVE 344,200:PRINT"0":MOVE 312,300:PRINT"750":MOVE 296,400:PRINT"1500"
1440 MOVE 296,500:PRINT"2250":MOVE 296,600:PRINT"3000":MOVE 296,700:PRINT"3750"
1450 MOVE 594,200:PRINT"0":MOVE 594,300:PRINT"5":MOVE 572,400:PRINT"10"
1460 MOVE 572,500:PRINT"15":MOVE 572,600:PRINT"20":MOVE 572,700:PRINT"25"
1470 MOVE 864,200:PRINT"0":MOVE 832,300:PRINT"200":MOVE 832,400:PRINT"400"
1480 MOVE 832,500:PRINT"600":MOVE 832,600:PRINT"800":MOVE 816,700:PRINT"1000"
1490 MOVE 1124,200:PRINT"0":MOVE 1124,300:PRINT"2":MOVE 1124,400:PRINT"4"
1500 MOVE 1124,500:PRINT"6":MOVE 1124,600:PRINT"8":MOVE 1108,700:PRINT"10"
1510 REM Construction of Scale Markers on axis
1520 XI=120
1530 FOR I=1 TO 5
1540 IF I=1 GOTO 1560
1550 XI=XI+260
1560 YI=200:MOVE XI,YI:DRAW XI,YI+500:YINC=YI
1570 FOR K=1 TO 6
1580 XSC=XI-10
1590 IF K=1 GOTO 1610
1600 YINC=YINC+100
1610 MOVE XI,YINC:DRAW XSC,YINC
1620 NEXT K
1630 NEXT I
1640 PASS=1:MOVE 100,100
1650 VDU5:PRINT "To Return to MENU,enter 'M' and then WAIT":VDU4
1660 REM Accessing Data Capture routine for next package of data
1670 GOSUB 2130
1680 IF PASS=1 THEN 1730
1690 REM Deletion of previous data from display
1700 GCOL 0,0:MOVE 0,804:PRINT STILLT:MOVE 258,804:PRINT ADREM:MOVE 518,804
1710 PRINT RTIME:MOVE 778,804:PRINT WETSOL:MOVE 1020,804:PRINT VISC:XB=130:YB=2
00
1720 REM Assigning new values to the variables
1730 STILLT=PROVAL(1)
1740 ADREM=(INT(PROVAL(2)*20.04))/10
1750 WETSOL=0.0:VISC=5.5:RTIME=15.7
1760 IF STILLT>=30 THEN T=(STILLT-30) ELSE T=0
```

```
 1770 A=500
 1780 REM Calculation of heights of bar charts
 1790 Y(1)=(T/100)*A:Y(2)=(ADREM/3750)*A:Y(3)=(RTIME/25)*A:Y(4)=(WETSOL/1000)*A:
Y(5)=(VISC/10)*A
 1800 REM Deletion of previous bars from display
 1810 XB=130:YB=200:GCOL 0,0
 1820 FOR J=1 TO 5
 1830 IF J=1 THEN 1850
 1840 XB=XB+260
 1850 IF Y(J)<OV(J) THEN
 1860 MOVE XB,Y(J):MOVE XB+40,Y(J):PLOT 85,XB+40,OV(J):MOVE XB,OV(J):PLOT 85,XB,
Y(J)
 1870 ELSE
 1880 NEXT J
 1890 REM Shading in of new bars on display
 1900 GCOL 0,1
 1910 XB=130:YB=200
 1920 FOR J=1 TO 5
 1930 IF J=1 GOTO 1950
 1940 XB=XB+260
 1950 MOVE XB,YB:MOVE XB+40,YB:PLOT 85,XB+40,YB+Y(J)
 1960 MOVE XB,YB+Y(J):PLOT 85,XB,YB:DRAW XB,YB
 1970 OV(J)=Y(J)
 1980 NEXT J
 1990 REM Printing of new data values on display
 2000 GCOL 0,1:VDU 5:MOVE 0,804:PRINT STILLT
 2010 MOVE 258,804:PRINT ADREM:MOVE 518,804:PRINT RTIME:MOVE 778,804:PRINT WETSO
-
 2020 REM Check to see if change in menu is required
 2030 MOVE 1020,804:PRINT VISC:MENU$=INKEY$(50):IF MENU$="M" THEN 2060
 2040 REM Return to wait for next data package
 2050 GOTO 1670
 2060 RSYNC=1:RETURN
 2070 REM
 2080 REM
 2090 REM
 2100 REM Subroutine for Data Capture and assembly of process values
 2110 REM
 2120 REM System setting to receive data through RS423 port
 2130 *FX2,2
 2140 *FX7,4
 2150 *FX2,1
 2160 *FX3,6
 2170 *FX21,1
 2180 REM Collection of ASCII coded characters for assembly into process
 2190 REM values
 2200 FOR J=1 TO 12
 2210 INPUT CLEN
 2220 FOR I=1 TO CLEN
 2230 INPUT X(I)
 2240 NEXT I
 2250 IF CLEN=1 THEN X(2)=0 ELSE GOTO 2270
 2260 GOTO 2310
 2270 IF CLEN=2 THEN 2310
 2280 IF CLEN=3 THEN 2330
 2290 IF CLEN=4 THEN 2350
 2300 IF CLEN=5 THEN 2370
 2310 PROVAL(J)=X(1)+X(2)/10
 2320 REM Assembly of characters into single process values
 2330 P1=10*X(1):PROVAL(J)=P1+X(2)+X(3)/10
```

```
 2340 GOTO 2380
 2350 P1=100*X(1):P2=10*X(2):PROVAL(J)=P1+P2+X(3)+X(4)/10
 2360 GOTO 2380
 2370 P1=1000*X(1):P2=100*X(2):P3=10*X(3):PROVAL(J)=P1+P2+P3+X(4)+X(5)/10
 2380 NEXT J
 2390 *FX2,2
 2400 *FX3,4
 2410 REM Dummy value used for viscosity while problems occur with
 2420 REM Viscometer
 2430 REVISC=2.0
 2440 REM Test to see if 10 mins. passed since data last stored on disc
 2450 IF SFIL=0 THEN GOTO 2480
 2460 IF CH1=100 THEN GOTO 2600 ELSE CH1=CH1+1
 2470 GOTO 2760
 2480 REM MAKE NEW DATA FILE
 2490 NGR=1
 2500 SFIL=1
 2510 D=OPENUP "DATAREC"
 2520 PTR£D=0
 2530 PRINT£D,NGR
 2540 PRINT£D,PROVAL(2),PROVAL(1),REVISC
 2550 FP=PTR£D
 2560 CLOSE£D
 2570 V=1
 2580 OFAD(1)=PROVAL(2):VISCRE(1)=2.:TEMPS(1)=PROVAL(2)
 2590 GOTO 2760
 2600 REM UPDATE DATA FILE
 2610 D=OPENIN "DATAREC"
 2620 PTR£D=0
 2630 INPUT£D,NGR
 2640 NGR=NGR+1
 2650 CLOSE£D
 2660 D=OPENUP "DATAREC"
 2670 PRINT£D,NGR
 2680 PTR£D=FP
 2690 PRINT£D,PROVAL(2),PROVAL(1),REVISC
 2700 FP=PTR£D
 2710 CLOSE£D
 2720 V=V+1
 2730 OFAD(V)=PROVAL(2):TEMPS(V)=PROVAL(1):VISCRE(V)=2.
 2740 CH1=1
 2750 REM End of Bar Chart display subroutine
 2760 RETURN
 2770 REM
 2780 REM
 2790 REM
 2800 REM Trend Display to indicate the rate of A.D. Removal
 2810 REM
 2820 REM Goto subroutine to construct X and Y axis
 2830 GOSUB 3150
 2840 REM Printing of scale values on axis
 2850 VDU 5:MOVE 71,115:PRINT "0":MOVE 39,295:PRINT"700"
 2860 MOVE 23,475:PRINT"1400":MOVE 23,655:PRINT"2100":MOVE 23,835:PRINT"2800"
 2870 MOVE 23,1015:PRINT "3500":MOVE 10,590:PRINT"ADOFF":MOVE 26,550:PRINT"KG":M
OVE 325,950
 2880 PRINT"VARIATION IN THE A.D. REMOVED"
 2890 MOVE 320,920
 2900 PRINT"-----------------------------"
 2910 PO1=100
 2920 REM Plotting of historical data
```

```
2930 FOR I=1 TO V
2940 Y=(OFAD(I)*900/3500)*2.004+100
2950 PLOT 69,PO1,Y
2960 PO1=PO1+12+2/9
2970 NEXT I
2980 REM Access Data Capture routine for latest values
2990 GOSUB 2130
3000 REM Plotting of latest data values
3010 Y=PROVAL(2)*900/3500*2.004+100
3020 POSIT=PO1+(10*1100)/(25*60*60)
3030 PLOT 69,POSIT,Y
3040 PO1=POSIT
3050 MENU$=INKEY$(0)
3060 REM Test to see if a change of display is required
3070 IF MENU$="M" THEN 3090
3080 GOTO 2990
3090 RSYNC=1:RETURN
3100 REM
3110 REM
3120 REM
3130 REM Subroutine to construct X and Y axis
3140 REM
3150 MODE0
3160 VDU 19,1,2;0;
3170 MOVE 100,1000:DRAW 100,100:DRAW 1200,100:XI=100:YI=1000:MOVE XI,YI
3180 FOR I=1 TO 11:DRAW XI-10,YI:YI=YI-90:MOVE XI,YI:NEXT I
3190 MOVE 100,100:XI=100:YI=100
3200 FOR I=1 TO 26:DRAW XI,YI-10:XI=XI+44:MOVE XI,YI:NEXT I
3210 VDU5:MOVE 95,75:PRINT"0":MOVE 315,75:PRINT "5":MOVE 525,75:PRINT"10"
3220 MOVE 745,75:PRINT "15":MOVE 965,75:PRINT "20":MOVE 1185,75:PRINT "25"
3230 RETURN
3240 REM
3250 REM
3260 REM
3270 REM Subroutine to display a trend graph of viscosity rise
3280 REM
3290 REM Goto subroutine to construct axis
3300 GOSUB 3150
3310 REM Addition of scale values to axis
3320 VDU 5:MOVE 71,115:PRINT"0":MOVE 71,295:PRINT"4":MOVE 71,475:PRINT "8"
3330 MOVE 55,655:PRINT"12":MOVE 55,835:PRINT"16":MOVE 55,1015:PRINT "20"
3340 MOVE 10,590:PRINT"VISC.":MOVE 10,550:PRINT"POISE":MOVE 325,950
3350 PRINT"VARIATION IN THE RESIN VISCOSITY"
3360 MOVE 320,920
3370 PRINT"-------------------------------"
3380 REM Displaying of historical data values
3390 PO1=100
3400 FOR I=1 TO V
3410 Y=VISCRE(I)*900/20+100
3420 PLOT 69,PO1,Y
3430 PO1=PO1+12+2/9
3440 NEXT I
3450 REM Accessing data capture routine for latest values
3460 GOSUB 2130
3470 REM Displaying of latest values
3480 Y=REVISC*900/20+100
3490 POSIT=PO1+(10*1100)/(25*60*60)
3500 PLOT 69,POSIT,Y
3510 PO1=POSIT
3520 MENU$=INKEY$(0)
```

```
 3530 REM Test to see if a change of display is required
 3540 IF MENU$="M" THEN 3570
 3550 REM Rrturn to display latest data
 3560 GOTO 3460
 3570 RSYNC=1:RETURN
 3580 REM
 3590 REM
 3600 REM
 3610 REM Subroutine to display the variation of Reactor Temp. with time
 3620 REM Calling subroutine to construct X and Y axis
 3630 GOSUB 3150
 3640 REM Addition of scale values to display
 3650 VDU 5:MOVE 55,115:PRINT"30":MOVE 55,295:PRINT"50":MOVE 55,475
 3660 PRINT "70":MOVE 55,655:PRINT"90":MOVE 39,835:PRINT"110":MOVE 39,1015:PRINT
 "130"
 3670 MOVE 620,50:PRINT"HOURS":MOVE 10,590:PRINT"TEMP.":MOVE 10,550:PRINT"DEGC":
MOVE 325,950
 3680 PRINT"VARIATION IN THE STILL TEMPERATURE"
 3690 MOVE 320,920
 3700 PRINT"-------------------------------------"
 3710 REM Display of historical data
 3720 PO1=100
 3730 FOR I=1 TO V
 3740 Y=(TEMPS(I)-30)*900/100+100
 3750 PLOT 69,PO1,Y
 3760 PO1=PO1+12+2/9
 3770 NEXT I
 3780 REM Accessing Data Capture package for latest values
 3790 GOSUB 2130
 3800 REM Display of latest data values
 3810 Y=(PROVAL(1)-30)*900/100+100
 3820 POSIT=PO1+(10*1100)/(25*60*60)
 3830 PLOT 69,POSIT,Y
 3840 PO1=POSIT
 3850 MENU$=INKEY$(0)
 3860 REM Test to see if a change of display has been requested
 3870 IF MENU$="M" THEN 3900
 3880 REM Return to get next set of data
 3890 GOTO 3790
 3900 RSYNC=1:RETURN
>
```

FIGURE A3.22 Typical Device Driver

```
          use /d0/defsfile
          mod prtend,prtnam,devic+objct,reent+1,prtmgr,prtdrv
          fcb write.
          fcb $ff
          fdb $f50C
          fcb prtnam-*-1
          fcb dt.scf
*default path options
          fcb 0
          fcb 0
          fcb 1
          fcb 0
          fcb 1
          fcb 0
          fcb 0
          fcb 0
          fcb 0
          fcb 0
          fcb c$cr
          fcb c$eof
          fcb 0
          fcb 0
          fcb 0
          fcb 0
          fcb 0
          fcb '-
          fcb 0
          fcb $15
          fcb 0
          fdb 0
prtmgr    fcs "scf"
prtnam    fcs "dtr"
prtdrv    fcs "acia"
          emod
prtend    equ *
```

APPENDIX 4

Ultra-Violet Analyser - T.P.A. Okometer Description
- Analyser Calibration Sheet
- Calibration Curves
- Interpolation Formulae
- Computer Program
- Table of Solvent Composition

Density Meter - Calibration Settings
- Calibration Curves

TPA OKOMETER

Main Features

Separate optical and electronic sealed modules

Simple adjustment of measuring ranges

Results not influenced by flow velocity

Not influenced by stray light

Wide range of applications

Immediate response

Optional BASEEFA approved purge system

Optional automatic zero adjustment

Principle of Operation

Radiation from the source (UV, Visible or near IR) passes through a collimating lens and the two beams, which are produced by an aperture mask, are filtered to provide a suitable wavelength. The beams are chopped and passed through the sample and reference cells before being concentrated onto the detector.

The chopping is so arranged that sample, reference and dark levels are available for the signal processing circuits, where they are converted to a signal equivalent to the absorbance of the sample. Output signals in either analogue or digital format are available for both local and remote display.

Description

The OKOMETER Photometer is used for the continuous measurement of the concentration of liquids and vapours with absorbtion bands in the visible, ultraviolet and near IR range.

Five different types of OKOMETER Photometers are available; all use two parallel light beams. The types are OKOMETER type 111, 3, 4 for the visible range, OKOMETER type 112 for measuring in the ultraviolet range and OKOMETER type 115 for the near IR range. For measurement in the blue, red and near IR range, the light source of the OKOMETER is a tungsten lamp, and for measuring in the ultra-violet range, the light source is a low pressure mercury discharge lamp.

Lenses, filters and cell windows for OKOMETER type 111, 4, 5 are made of glass; types 112, 3 use quartz.

The OKOMETER is housed in two 19" industrial modules which have been specially designed for use in Zone 1 or in the presence of corrosive atmospheres. Optional purge control unit and wall mounting cabinet are available along with a full range of data processing modules.

TECHNICAL DATA

Input

Spans

0-0.01A to 0-2A between 0 and 3A light absorbtion between 254....2000 nm.

Sample Flow

30 to 60 1/h.

Sample Temperature

10....50^{o}C subject to moisture content.

Output

Output Signal

0....20mA or 4....20mA into 750 Ω max.

Characteristic

Output linear 0-2 Absorbance units

Settling Time

Preset at 10 s for amplifier, 1 s for cell (excluding sample tubing).

Error

2% of span max.

Zero Drift

2% of span per week, non-cumulative, max.

Power Supply

Supply Voltage

110/220/240V + 10% -15%

Supply Frequency

50/60 Hz +4%

Power Consumption

150VA.

Environmental Conditions

Temperature Limits

10°C - 50°C (functional over the range 0°C to 50°C).

Shock Vibration

Low sensitivity to shock and vibration in operation.

Housing

Two 19" modules, protected to IP54 to DIIN 40050 (IEC 144). An optional wall mounting case protected by IP55).

General

Thermostat

Built-in for optical compartment at 50°C + 2°C.

Mounting

One 19" rack x 4U and one 19" rack x 5U to DIN 41494 (IEC

297). Optional wall mounting case available TPA 950-00161.

Weight

Electronic module	12 Kg with purge housing.
Optical module	10 Kg with purge housing.

CALIBRATION SHEET FOR TPA 110 SERIES

Model TPA	112	Serial No: 018502
W/O No:	80288	
Mains Supply:	240 Volts 50 Hz	
To Measure:	0-20% Xylol	
In the presence of:	n-Butanol	
This instrument supplied with:		
Measuring Cell	Material	Stainless Steel
	Length	1 mm
Filter	Type	-
Source	Type	Pen Ray 285 mm - wavelength
Detector	Type	Ultra Violet
Sample Condition	Temp.	20°C
	Pressure	1007 mb
	Flow	-
	Concentration	See Below
Calibration	Wire Size	-
	Wire No	-
	Output	-
Amplifier Setting	Gain	High 132
	Zero	495

Xylol in n-butanol	mA ouput
0	4.0
1	6.0
5	8.0
10	11.00
20	20.00

Figure A4·1 Calibration Curve U-V Analyser

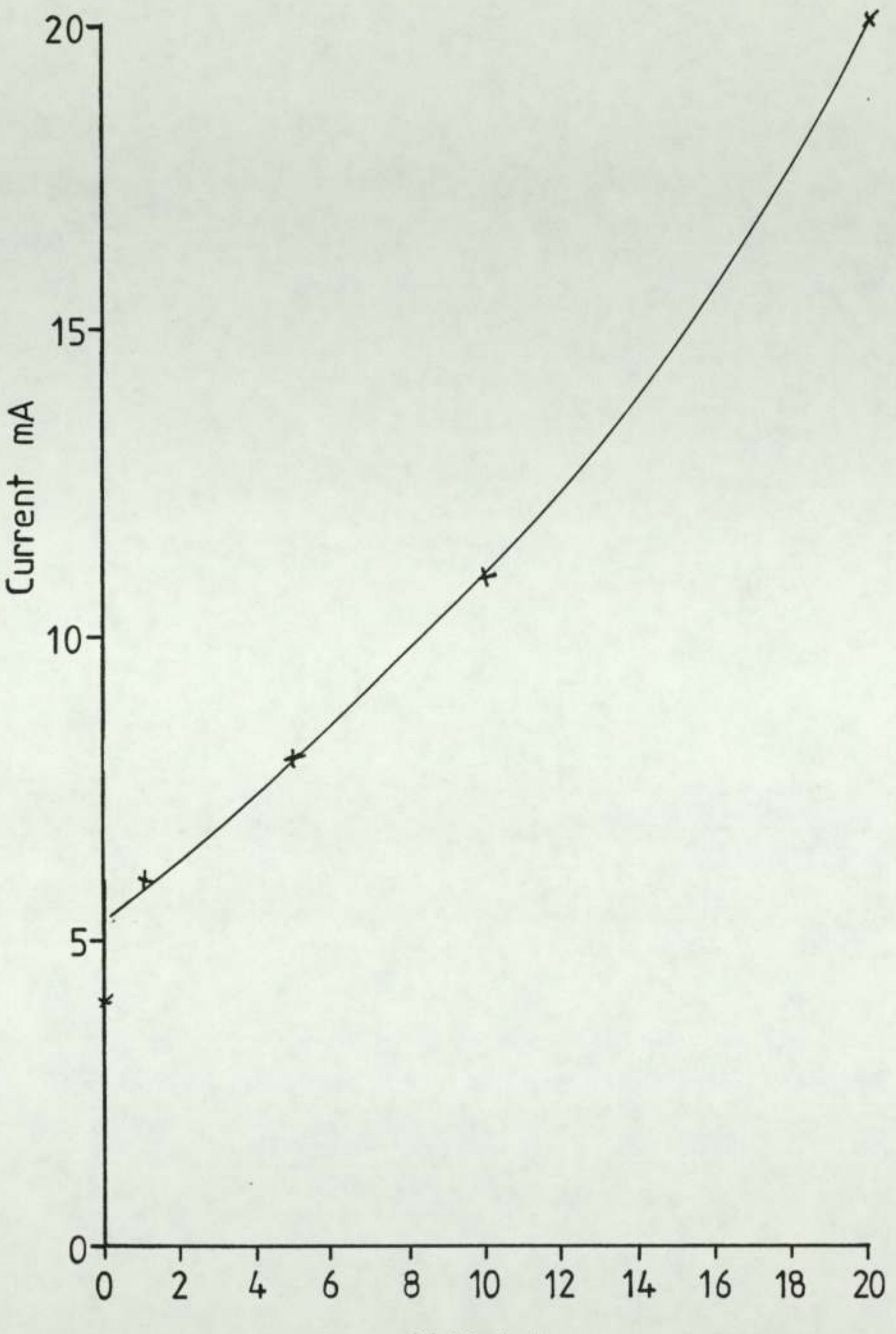
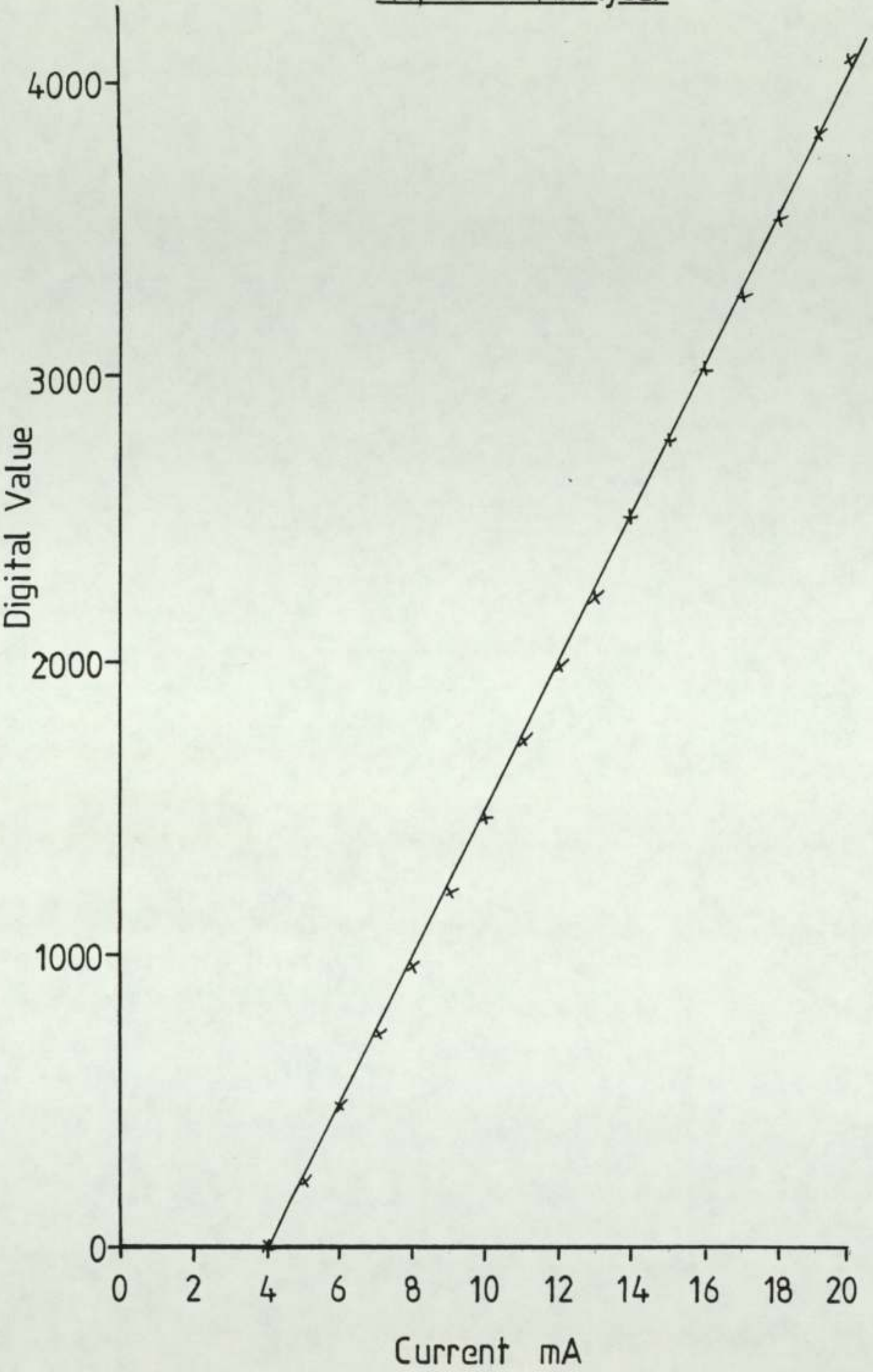


Figure A4·2 Analogue - Digital Conversion Chart for U-V Analyser



LAGRANGE INTERPOLATION FORMULAE

The formula for a polynomial of degree n is expressed as :-

$$y(x) \quad = \quad \underset{j=o}{\overset{n}{}} \quad \frac{\Pi\,(x)}{(x - x_j)\,\Pi\,(x_j)} \qquad f\,(x_j)$$

where $\Pi = (x - x_o)\,(x - x_1) \,\ldots\ldots\ldots\ldots\ldots\, (x - xn)$

$$\Pi\,(x_j) = (x_j - x_o)\,(x_j - x_1) \,\ldots\ldots\ldots\, (x_j - xn)$$

where $(x_j - x_j)$ is the omitted factor

Thus: $f(x) = y(x) + En(x)$

$$En = \frac{1}{(n + 1)\,!} \qquad ll\,(x) f^{(n + 1)}(E)$$

e.g. For an interpolation polynomial of degree 3

$$y(x) = \frac{(x - x_1)\,(x - x_2)\,(x - 3_3)}{(x_o - x_1)\,(x_o - x_2)\,(x_o - x_3)} \qquad f(x_o)$$

$$+ \frac{(x - x_o)\,(x - x_2)\,(x - x_3)}{(x_1 - x_o)\,(x_1 - x_2)\,(x_1 - x_3)} \qquad f(x_1)$$

$$+ \frac{(x - x_o)\,(x - x_1)\,(x - x_3)}{(x_2 - x_o)\,(x_2 - x_1)\,(x_2 - x_3)} \qquad f(x_2)$$

$$+ \frac{(x - x_o)\,(x - x_1)\,(x - x_2)}{(x_3 - x_o)\,(x_3 - x_1)\,(x_3 - x_2)} \quad f(x_3)$$

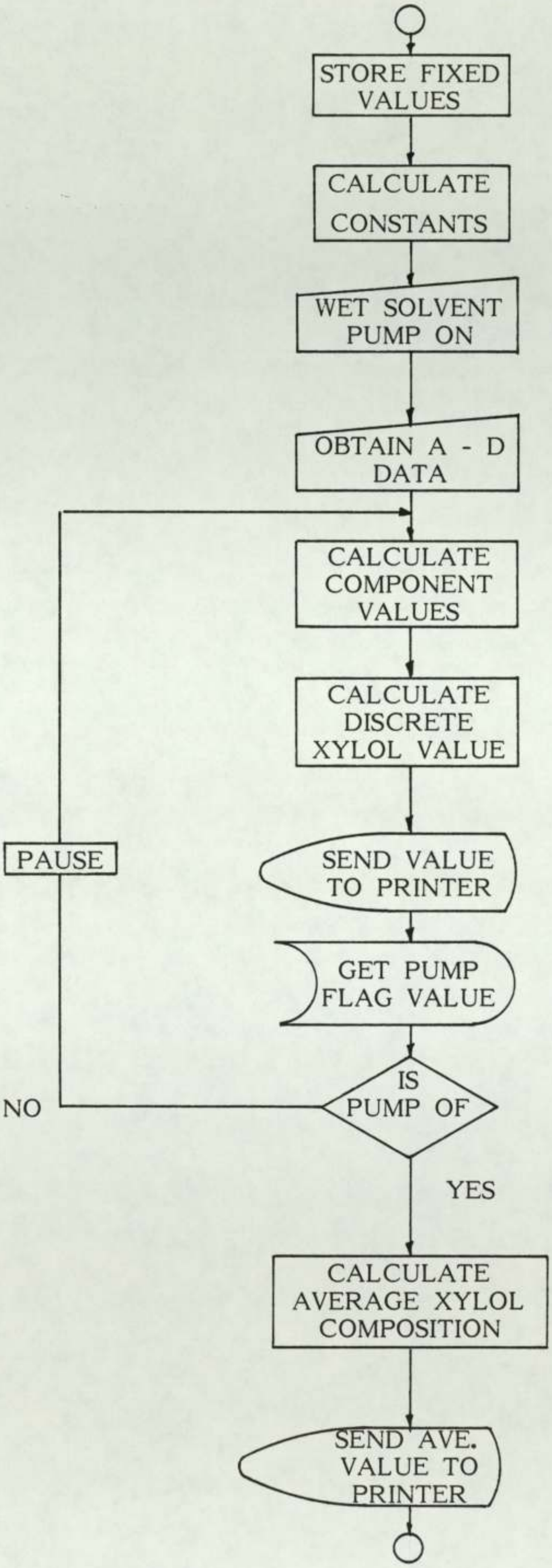
OUTLINE OF COMPUTER PROGRAM

This program was written so that the signal output from the U-V analyser could be monitored every six seconds during the charging operation. The analyser outputs were then converted to actual xylol contents using an interpolation method outlined in this Appendix. On completion of the charging operation the average xylol composition of the charge was calculated from the discrete values obtained.

The program is initiated by entering the program name 'UVanalysis', this automatically loads and runs the procedure. The operation of the procedure is suspended until it has been informed via a keyboard command that the Wet n-Butanol pump has been switched on. It will then proceed to calculate the xylol composition every six seconds, printing these values onto a logsheet. To terminate this operation another program name must be entered. 'Pumpoff', which sets-up a flag indicating that the charging operation has been completed. The procedure then calculates the average xylol composition of the charge and prints this value on the logsheet, before terminating itself.

The average value for the xylol composition can then be used as a basis for adjusting pure solvent charges either manually or by feeding this value to an appropriate computer program.

FIGURE A4.3 PROCEDURE U-V ANALYSIS



```
PROCEDURE uvanalysis
REM
REM Procedure to calculate the Weight% Xylol in the Wet Solvent
REM
REM using the output from an Ultra-Violet analyser
REM
REM
REM Definition of the variables and arrays
DIM ad1,p1:BYTE
DIM chan:INTEGER
DIM xylo(500):REAL
DIM off1:BYTE
DIM i:INTEGER
DIM a:REAL
DIM dat:INTEGER
DIM rtime(6):STRING
i=1
REM Definition of A-D channel
chan=13
REM Known values for the Lagrange Interpolation routine
x0=490.2
x1=968.4
x2=1748.
x3=4084.4
y0=1.
y1=5.
y2=10.
y3=20.
REM Calculation of constants for interpolation routine
v1=(x0-x1)*(x0-x2)*(x0-x3)
v2=(x1-x0)*(x1-x2)*(x1-x3)
v3=(x2-x0)*(x2-x1)*(x2-x3)
v4=(x3-x0)*(x3-x1)*(x3-x2)
REM Acknowledgement that solvent is being charged to reactor
1 PRINT "If wet solvent pump switched on enter 1"
INPUT "Solvent pump state ",onoff
IF onoff=1 THEN 5
GOTO 1
REM Send analysis data to printer
5 OPEN £p1,"/pS":WRITE
REM Access A-D converter to obtain digital value
6 OPEN £ad1,"/anlg"
PUT £ad1,chan
GET £ad1,dat
a=dat+968.4
CLOSE £ad1
BASE 1
REM Calculation of weight % Xylol in wet solvent
f1=y0*(a-x1)*(a-x2)*(a-x3)/v1
f2=y1*(a-x0)*(a-x2)*(a-x3)/v2
f3=y2*(a-x0)*(a-x1)*(a-x3)/v3
f4=y3*(a-x0)*(a-x1)*(a-x2)/v4
xylo1=f1+f2+f3+f4
rtime(6)=MID$(DATE$,10,5)
REM Logging of data on printer
PRINT £p1,rtime(6),i,xylo1,a
REM Accessing flag file to check if pump has been switched off
OPEN £off1,"flagoff":READ
READ £off1,onoff
CLOSE £off1
```

```
xylo(i)=xylol
REM Test to see if pump is off
IF onoff=0 THEN 50
REM Pause for 600 ticks
SHELL "sleep 600"
i=i+1
REM Return to step 6 to obtain next value for xylol content
GOTO 6
50 xylol=0
REM Average Xylol composition for no. of samples
FOR k=1 TO i
xylol=xylol+xylo(k)
NEXT k
perxylol=xylol/i
PRINT £p1
PRINT £p1,"The percentage xylol in the Wet Solvent stream is  - "

; perxylol
CLOSE £p1
END
```

TABLE A4.1 WET SOLVENT COMPOSITIONS

Wt%

Sample No.	Date	Resin Type	n-Butanol	Water	Xylol	Formaldehyde	Total
1	19.3.85 (1)	615	56.8	7.9	14.8	6.5	86
2	19.3.85 (2)	615	57.5	8.3	14.6	5.5	86
3	16.4.85	615	77.8	5.3	15.4	2.4	101
4	19.4.85	6022	74.8	5.2	16.5	2.5	99
5	21.4.85	675	74.6	7.4	17.2	3.5	103
6	30.4.85	645	72.3	5.5	19.9	2.3	100
7	1.5.85	645	68.9	8.7	22.0	1	100

The compositions of the above solvent samples were determined using Gas-Liquid Chromatography (GLC).

DENSITY METER CALIBRATION

Calibration Settings

Us = 40

N = 12

To = 254 400 Span = 800

Ov = 254 400 10V = 255 200

Temperature Compensation

Temp ^oC	Density g/cc
14.9	0.836
15.8	0.835
16.1	0.835
16.6	0.834
17.0	0.834
17.2	0.833
17.5	0.833
17.9	0.833
18.2	0.831
18.9	0.831
19.9	0.830
20.8	0.829
22.0	0.828
22.8	0.827
24.3	0.826
24.8	0.826
25.5	0.825

Density Change = 0.011 g/cc

For Temperature Change = $10{\cdot}6^oC$

$\therefore \beta = 0.011/10.6 = 10.38^{x10^{-4}}$ g/cc/^oC

Mechanical Compensation value

$= 10.38 \times 0.83 \times 10^{-4} = 8.6 \times 10^{-4}$

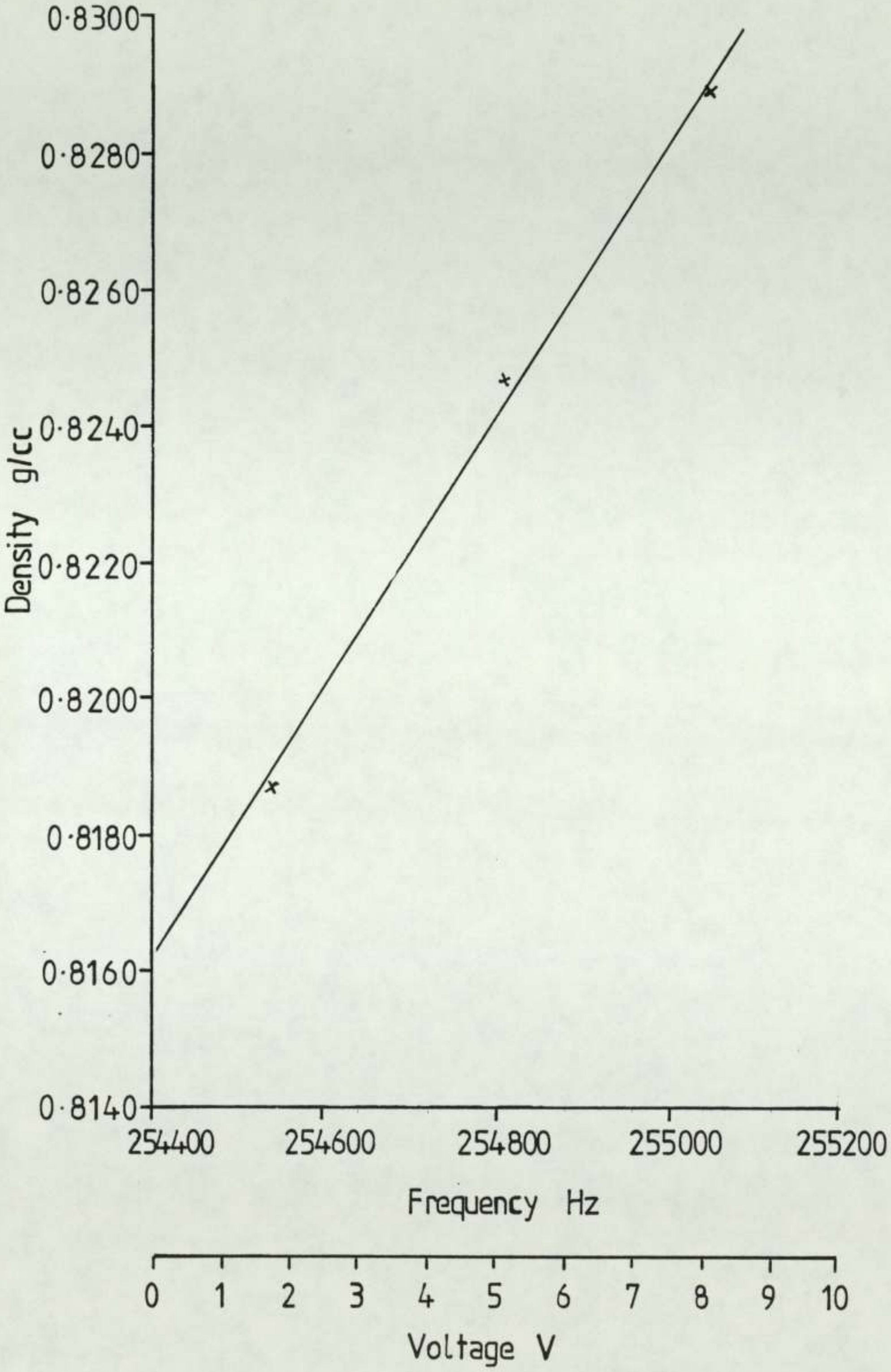
from the graph of Frequency (T) vs Density the following calibration curve was obtained.

$$Y = 2.00 \text{ x } 10^{-5} \text{ x } -4.2642$$

where Y is the fluid density in g/cc

X is the resonant frequency, in Hertz, of the U-tube and its contents

Figure A4·4 Calibration Curve for Density Meter



APPENDIX 5

Logsheets - Computer Aided Control Trial Logsheets
- Computer Control Trial Logsheets

TABLE A5.1 Computer Aided Control Trial Logsheet

Resin Type: C

Date: 21 April 1985

Time	Observations
14.30	Charging commenced Formalin and normal-butanol added to reactor. pH adjusted to 6.3 by addition of a caustic solution.
16.00	Wet normal-butanol charged to reactor. Xylol content of wet solvent determined as 17.55% by in-line Ultra-Violet analysis. Density of wet solvent charged to reactor = 0·8377 g/cc by off-line density measurement. Mass of water added to reactor in wet solvent = 124 kg by manual calculation. Water to be removed during dehydration stage is 2101 kg and during acid reaction stage is 2631 kg. Tank Tare Value = 153 kg.
16.15	Steam switched on to reactor jacket and coils.
16.45	Boiling of vessel contents commenced.
17.00	Prompt generated by computer for Xylol addition at correct time. Xylol charge made to reactor.
20.00	Prompt generated by computer for addition of second Butanol charge. This occured correctly.
21.40	Acid catalyst added to reactor. Prompt generated late for catalyst addition. Observed that oscillation occured on graphics display for aqueous distillate tank value, probably reason for late generation of prompt. Require to smooth tank weight value.
02.10	Reaction stage completed with 2765 kg of water removed. Computer generated prompt at 2630 kg. Again probably due to oscillation of tank weight value.

TABLE A5·1 Computer Generated Logsheet

TIME	stean FLOW KG/HR	COIL PRESS BAR	STILL TEMP DEGC	DIST degC DEGC	COLUMN TOP TEM DEGC	A.D. OFF L/HR	ALCO TANKWT Kg	cf.TEMP DEGC	CR.TEMP DEGC
16:37	328.1	1.4	96.9	50.2	91.1	.0	156.3	14.6	32.6
17:07	589.8	2.1	96.2	50.2	90.4	.0	144.3	15.1	39.4
17:17	589.4	2.1	96.2	50.2	90.5	524.5	242.5	15.6	41.2
17:27	561.1	2.1	96.1	50.2	90.5	510.8	312.6	16.3	42.5
17:37	574.4	2.1	96.1	50.2	90.5	538.6	398.8	17.0	42.8
17:47	561.1	2.1	96.1	50.2	90.5	526.0	462.9	17.5	42.8
17:57	559.3	2.1	96.1	50.2	90.5	499.3	549.1	17.9	43.2
18:07	554.5	2.1	96.1	50.2	90.6	499.6	655.3	18.4	43.3
18:17	779.1	2.6	96.2	50.2	90.7	594.3	741.5	18.1	47.1
18:27	745.7	2.6	96.3	50.2	90.7	615.1	851.7	16.6	45.1
18:37	746.8	2.6	96.3	50.2	90.8	604.7	931.9	15.2	43.9
18:47	735.8	2.6	96.4	50.2	90.8	556.8	1026.0	14.1	42.1
18:57	716.8	2.6	96.4	50.2	90.8	561.6	1124.2	14.6	42.3
19:07	708.3	2.6	96.6	50.2	90.9	568.0	1200.4	15.4	43.1
19:17	697.3	2.6	96.6	50.2	91.0	530.5	1288.6	16.0	43.3
19:27	685.2	2.6	96.8	50.2	91.0	540.1	1418.8	15.9	42.7
19:37	658.4	2.6	97.0	50.2	91.1	530.5	1448.9	14.7	41.1
19:47	640.8	2.6	97.2	50.2	91.2	524.1	1551.1	13.9	38.8
19:57	616.2	2.6	97.4	50.2	91.3	125.1	1675.3	13.4	33.8
20:07	802.6	3.1	98.2	50.2	92.2	496.3	1731.5	12.7	39.5
20:17	763.7	3.1	98.8	50.2	92.5	458.8	1781.6	12.6	39.5
20:27	730.3	3.1	99.3	50.2	92.8	443.2	1867.7	14.0	40.2
20:37	692.9	3.1	99.8	50.2	93.2	420.6	1931.9	15.6	40.4
20:47	657.3	3.1	100.4	50.2	93.6	395.0	1996.0	17.5	41.8
20:57	637.8	3.1	100.6	50.2	90.0	441.4	2014.0	17.1	41.1
21:07	604.1	3.1	101.3	50.2	88.9	456.6	2152.3	16.0	39.3
21:17	577.3	3.1	102.3	50.2	88.9	453.2	2244.5	15.2	37.7
21:27	527.0	3.1	103.2	50.2	89.2	408.3	2252.5	14.5	35.6

TABLE A5·1 Cont.

21:37	488.1	3.1	104.6	50.2	89.8	446.2	2288.6	14.0	34.6
21:47	455.8	3.1	105.3	50.2	90.3	368.6	2384.8	13.6	34.0
21:57	424.3	3.1	106.1	50.2	90.7	328.1	2426.8	13.4	32.7
22:07	383.5	3.1	107.2	50.2	91.3	294.0	2491.0	13.2	31.4
22:17	350.1	3.1	108.4	50.2	92.3	255.8	2601.2	13.0	29.6
22:27	322.6	2.7	109.8	50.2	93.0	186.7	2629.2	12.8	28.2
22:37	301.7	3.1	111.3	50.2	93.6	193.4	2693.4	12.6	27.2
22:47	269.7	3.1	112.8	50.2	94.5	154.8	2665.3	12.5	26.1
22:57	236.0	3.1	114.2	50.2	95.2	148.5	2643.3	12.5	25.1
23:07	220.2	3.1	115.5	50.2	96.3	128.1	2701.4	14.0	26.0
23:17	207.4	3.1	116.8	50.2	97.4	63.5	2675.3	15.7	27.3
23:27	191.6	3.1	118.0	50.2	98.5	65.3	2717.4	16.9	28.0
23:37	174.3	3.1	119.1	50.2	99.3	34.5	2765.5	17.2	28.0
23:47	163.3	3.1	120.0	50.2	100.2	.0	2765.5	15.9	26.3
23:57	154.9	3.1	120.9	50.2	101.1	.7	2765.5	14.7	24.6
00:07	144.6	3.1	121.6	50.2	101.6	.0	2755.5	13.8	23.0
00:17	133.6	3.1	122.3	50.2	102.1	.0	2765.5	13.2	22.4
00:27	128.5	3.1	122.9	50.2	102.6	.0	2783.6	12.6	22.1
00:37	152.3	3.2	123.3	50.2	103.0	.0	2781.6	12.2	21.2
00:47	118.5	3.1	123.7	50.2	103.2	.0	2765.5	12.9	21.5
00:57	115.6	3.1	124.1	50.2	103.3	.0	2839.7	14.4	22.9
01:07	115.2	3.1	124.4	50.2	103.7	.0	2797.6	15.7	24.3
01:17	112.7	3.1	124.6	50.2	104.1	.0	2755.5	16.9	25.4
01:27	105.0	3.1	124.9	50.2	104.4	.0	2857.7	16.5	25.1
01:37	104.2	3.1	125.1	50.2	104.5	.0	2785.6	15.1	23.5
01:47	102.4	3.1	125.3	50.2	104.8	.0	2771.5	14.0	22.1
01:57	99.5	3.1	125.5	50.2	105.1	.0	2813.6	13.3	21.2
02:07	9.5	3.9	125.6	50.2	105.2	.0	2815.6	12.6	20.2
02:17	8.4	3.9	114.9	50.2	93.5	.0	2859.7	12.8	12.7
02:27	8.8	3.9	103.4	50.2	74.0	.0	2865.7	14.0	14.0

TABLE A5.2 Computer Aided Control Trial Logsheet

Resin Type: B

Date: 30 April 1985

Time	Observations
15.40	Charging commenced Formalin and normal-butanol added to reactor. pH adjusted to 6·1 by addition of a caustic solution
16.30	Wet butanol charged to reactor. Xylol content of wet solvent determined as 25·2% by in-line Ultra-Violet analysis. Probable fault in analyser. Density of wet solvent charged to reactor = 0.8360 g/cc by off-line density measurement. Mass of water added to reactor in wet solvent = 88 kg by manual calculation. Gives mass of water to be removed during dehydration stage as 2050 kg and during acid reaction stage as 2595 kg.
18.50	Steam switched on to reactor jacket coils.
19.20	Boiling of vessel contents commenced.
19.35	Prompt generated by computer for Xylol addition at correct time. Charge made to reactor.
22.35	Computer generated prompt at correct time for addition of second butanol charge.
01.20	Acid catalyst added to reactor. Prompt generated at correct point for end of dehydration. Oscillations still occuring on graphics display of aqueous distillate tank weight.
07.15	Reaction stages completed with 2383 kg water removed. Predicted value 2596·11 kg. Difference probably due to assumed value being used for water to be removed during acid reaction stage for B type resins.

TABLE A5·2 Computer Generated Logsheet

TIME	stean FLOW KG/HR	COIL PRESS BAR	STILL TEMP DEGC	DIST degC DEGC	COLUMN TOP TEM DEGC	A.D. OFF L/HR	ALCO TANKWT Kg	cf.TEMP DEGC	CR.TEMP DEGC
19:03	1395.0	3.1	73.1	50.2	55.0	.0	84.2	16.8	17.1
19:13	390.9	1.5	95.1	50.2	88.6	.0	112.2	17.1	25.2
19:23	387.9	1.5	96.3	50.2	90.7	.0	130.3	17.0	26.2
19:33	382.0	1.5	96.0	50.2	90.4	.0	96.2	17.4	20.7
19:43	1296.2	3.1	95.7	50.2	90.1	290.3	130.3	17.5	33.5
19:53	1271.7	3.1	95.6	50.2	90.1	380.9	192.4	17.9	33.7
20:03	1243.0	3.1	95.5	50.2	90.2	53.5	236.5	18.2	33.8
20:13	1229.1	3.1	95.4	50.2	90.2	100.6	266.5	18.2	33.4
20:23	1202.7	3.1	95.4	50.2	90.3	189.3	282.6	18.1	33.3
20:33	1193.9	3.1	95.3	50.2	90.4	124.0	308.6	18.0	33.1
20:43	1210.7	3.1	95.3	50.2	90.5	88.3	328.7	18.0	33.2
20:53	1246.7	3.1	95.4	50.2	90.6	83.9	362.7	17.9	33.3
21:03	1236.4	3.1	95.4	50.2	90.6	131.8	388.8	17.8	33.2
21:13	1240.1	3.1	95.5	50.2	90.6	64.2	416.8	17.8	33.0
21:23	1200.8	3.1	95.6	50.2	90.7	.0	464.9	17.8	32.9
21:33	1185.4	3.1	95.6	50.2	90.7	.0	485.0	17.8	32.7
21:43	1168.5	3.1	95.6	50.2	90.8	88.3	559.1	17.6	32.6
21:53	1152.0	3.1	95.6	50.2	90.8	463.6	637.3	17.6	32.7
22:03	1142.5	3.1	95.7	50.2	90.9	461.4	709.4	17.5	32.6
22:13	1119.0	3.1	95.7	50.2	90.9	624.7	791.6	17.5	32.4
22:23	1099.9	3.1	95.8	50.2	91.0	675.2	847.7	17.4	32.3
22:33	1089.6	3.1	95.7	50.2	90.6	664.5	969.9	17.3	26.4
22:43	1054.4	3.1	96.0	50.2	91.0	672.3	1046.1	17.0	31.6
22:53	980.3	3.1	96.1	50.2	91.1	511.9	1084.2	16.9	30.9
23:03	944.3	3.1	96.2	50.2	91.1	640.0	1218.4	16.8	30.8
23:13	937.0	3.1	96.4	50.2	91.1	815.9	1292.6	16.4	30.1
23:23	884.1	3.1	96.7	50.2	91.1	599.5	1418.8	16.1	29.4
23:33	835.7	3.1	96.9	50.2	91.1	556.1	1472.9	15.9	28.6

TABLE A5·2 Cont.

23:43	756.0	3.1	97.3	50.2	91.4	518.2	1593.2	15.6	27.7
23:53	682.3	3.1	97.8	50.2	91.8	499.6	1649.3	15.8	27.1
00:03	631.6	3.1	98.3	50.2	92.2	459.6	1757.5	15.7	26.6
00:13	593.8	3.1	98.9	50.2	92.7	406.5	1895.8	15.5	25.9
00:23	564.1	3.1	99.7	50.2	93.3	379.0	1895.8	15.4	25.3
00:33	526.3	3.1	100.5	50.2	94.0	336.7	1992.0	15.3	24.8
00:43	486.6	3.1	101.4	50.2	94.7	298.1	1977.9	15.2	24.3
00:53	458.4	3.1	102.5	50.2	95.6	267.3	2046.1	15.1	23.7
01:03	422.4	3.1	103.6	50.2	96.5	218.6	2152.3	15.0	23.1
01:13	401.9	3.1	104.8	50.2	97.5	200.1	2198.4	14.8	22.6
01:23	66.4	1.3	105.7	50.2	97.6	.0	2170.3	14.6	16.4
01:33	163.7	2.1	104.9	50.2	91.0	157.8	2252.5	14.4	20.2
01:43	157.1	2.1	105.1	50.2	89.5	175.6	2252.5	14.1	19.1
01:53	149.0	2.1	105.8	50.2	89.2	177.8	2280.6	13.8	18.5
02:03	138.0	2.1	106.7	50.2	89.4	165.2	2300.6	13.3	17.7
02:13	128.5	2.1	107.6	50.2	90.0	146.6	2344.7	13.1	17.3
02:23	119.3	2.1	108.6	50.2	91.3	131.8	2404.8	13.4	17.3
02:33	106.8	2.1	109.6	50.2	92.1	98.4	2352.7	13.7	17.3
02:43	101.3	2.1	110.5	50.2	93.0	108.8	2444.9	13.7	17.2
02:53	94.0	2.1	111.5	50.2	94.4	77.2	2408.8	13.8	17.1
03:03	89.2	2.1	112.3	50.2	94.8	61.2	2454.9	13.8	16.9
03:13	79.6	2.1	113.3	50.2	95.3	.0	2509.0	13.7	16.7
03:23	76.3	2.1	114.2	50.2	96.1	.0	2416.8	13.7	16.6
03:33	67.2	2.1	114.9	50.2	96.8	.0	2509.0	13.6	16.3
03:43	65.0	2.1	115.7	50.2	97.4	.0	2533.1	13.4	16.1
03:53	59.8	2.1	116.3	50.2	97.9	.0	2573.1	13.4	15.9
04:03	57.3	2.1	116.9	50.2	98.7	.0	2472.9	13.4	15.8
04:13	54.7	2.1	117.4	50.2	98.1	.0	2491.0	13.2	14.0
04:23	55.4	2.1	117.5	50.2	99.0	.0	2537.1	13.0	15.3
04:33	51.0	2.1	118.1	50.2	98.3	.0	2487.0	12.7	15.0

TABLE A5·2 Cont.

04:43	48.8	2.1	118.5	50.2	98.4	.0	2481.0	12.5	14.6
04:53	48.1	2.1	118.9	50.2	99.0	.0	2541.1	12.2	14.2
05:03	49.9	2.1	119.3	50.2	99.6	.0	2573.1	12.4	14.4
05:13	44.4	2.1	119.6	50.2	100.2	.0	2629.2	12.6	14.5
05:23	41.1	2.1	120.0	50.2	100.8	.0	2499.0	12.4	14.4
05:33	43.3	2.1	120.2	50.2	101.1	.0	2523.0	11.9	13.8
05:43	40.4	2.1	120.5	50.2	101.4	.0	2491.0	11.8	13.6
05:53	37.4	2.1	120.7	50.2	101.8	.0	2499.0	13.5	15.2
06:03	35.6	2.1	120.9	50.2	102.0	.0	2573.1	15.1	17.0
06:13	34.5	2.1	121.1	50.2	101.5	.0	2567.1	16.3	18.3
06:23	38.5	2.1	121.3	50.2	101.2	.0	2491.0	17.4	19.4
06:33	35.2	2.1	121.5	50.2	101.3	.0	2583.2	15.6	17.5
06:43	39.6	2.1	120.7	50.2	96.2	.0	2545.1	14.3	14.6
06:53	38.9	2.1	121.1	50.2	102.9	.0	2499.0	13.5	15.2
07:03	36.7	2.1	121.4	50.2	99.5	.0	2637.3	13.1	14.8
07:13	9.5	3.9	121.5	50.2	99.7	.0	2601.2	12.6	13.6
07:23	7.3	3.8	112.1	50.2	87.6	.0	2547.1	12.4	12.3
07:33	19.8	3.8	101.6	50.2	68.7	50.5	2539.1	12.3	12.3
07:43	8.1	3.8	92.7	50.2	55.0	.0	2637.3	12.2	12.1
07:53	.7	3.7	84.4	50.2	56.6	5.6	2483.0	11.8	11.7
08:03	1.5	3.8	77.6	50.2	56.6	.0	2509.0	11.7	11.6
08:13	.0	3.8	71.8	50.2	55.2	95.0	2499.0	13.2	13.1
08:23	.7	3.8	67.8	50.2	56.4	.0	2507.0	14.6	14.6
08:33	.4	3.8	67.5	50.2	56.6	.0	2499.0	15.0	15.3
08:43	1.8	3.8	67.8	50.2	55.2	324.4	2509.0	13.7	13.8
08:53	360.8	1.9	68.1	50.2	56.2	.0	2489.0	12.8	13.0
09:03	204.8	2.0	78.7	50.2	55.6	1520.1	2497.0	12.2	12.3
09:13	141.7	1.7	88.2	50.2	77.0	98.0	2499.0	12.0	18.6
09:23	146.1	1.7	87.2	50.2	77.4	36.4	2583.2	11.9	16.5

TABLE A5.3 Computer Aided Control Trial Logsheets

Resin Type: C

Date: 1 May 1987

Time	Observations
18.30	Charging commenced with Formalin and normal-butanol added to reactor. pH adjusted using caustic soda solution.
19.40	Wet normal-butanol charged to reactor. Xylol content of wet solvent assumed to be 15·5% due to apparent failure of U-V analyser. Density of wet solvent charged to reactor = 0.8400 g/cc by off-line density measurement. Mass of water added to reactor in wet solvent = 123 kg by manual calculation. Mass of water to be removed during dehydration stage is 2100 kg and total mass off at end of acid reaction stage is 2630 kg. Tank Tare Value = 11 kg.
19.55	Steam switched on.
20.25	Boiling of vessel contents commenced.
20.40	Prompt for Xylol addition made at correct time. Xylol charge made to reactor.
23.40	Second normal butanol charged to reactor. Prompt not generated by computer.
00.05	Prompt generated by computer for second normal butanol charge. 25 minutes late. Possible software fault or difficulty in accessing flag files.
00.45	Computer prompt generated for addition of acid catalyst.
01.10	Acid catalyst added to reactor.

TABLE A5.3 Continued

Time	Observations
06.00	Reaction stage completed with 2397 kg of water removed. Predicted value 2630 kg. Difference due to assumed value being used for water produced during acid reaction stage for B type resins.

TABLE A5·3 Computer Generated Logsheet

TIME	stean FLOW KG/HR	COIL PRESS BAR	STILL TEMP DEGC	DIST degC DEGC	COLUMN TOP TEM DEGC	A.D. OFF L/HR	ALCO TANKWT Kg	cf.TEMP DEGC	CR.TEMP DEGC
20:04	1343.2	3.2	66.5	50.2	55.0	.0	92.2	12.2	12.2
20:14	279.7	1.3	92.6	50.2	60.9	.0	94.2	12.0	13.0
20:24	200.7	1.0	96.1	50.2	90.3	.0	106.2	12.9	19.9
20:34	198.9	1.0	95.9	50.2	90.3	.0	120.2	13.7	20.3
20:44	978.4	3.1	95.4	50.2	89.9	568.7	170.3	13.2	27.5
20:54	962.6	3.0	95.3	50.2	90.0	588.7	256.5	13.1	27.1
21:04	1097.7	3.4	95.3	50.2	90.1	606.9	348.7	13.2	26.7
21:14	881.5	3.1	95.3	50.2	90.2	588.0	430.9	13.3	26.8
21:24	816.9	3.1	95.3	50.2	90.4	591.0	535.1	13.7	26.9
21:34	807.4	3.1	95.3	50.2	90.5	566.8	637.3	14.1	26.8
21:44	866.9	3.1	95.4	50.2	90.6	587.3	739.5	13.9	27.2
21:54	885.9	3.1	95.5	50.2	90.6	597.6	817.6	13.7	27.0
22:04	880.4	3.1	95.6	50.2	90.7	1021.2	889.8	13.6	26.8
22:14	835.7	3.1	95.6	50.2	90.7	782.5	1002.0	13.5	26.6
22:24	805.9	3.1	95.7	50.2	90.8	537.9	1108.2	13.4	26.3
22:34	739.5	3.1	95.7	50.2	90.8	558.7	1178.4	13.4	25.6
22:44	721.9	3.1	95.7	50.2	90.8	547.2	1264.5	13.6	25.6
22:54	708.7	3.1	95.9	50.2	90.8	527.1	1374.7	13.4	25.2
23:04	656.2	3.1	96.1	50.2	90.8	518.6	1464.9	13.3	24.7
23:14	635.3	3.1	96.3	50.2	90.8	513.8	1603.2	13.2	24.2
23:24	596.7	3.1	96.6	50.2	90.8	483.3	1629.3	13.0	23.6
23:34	568.5	3.1	97.0	50.2	90.8	471.4	1693.4	13.0	23.3
23:44	557.1	3.1	97.5	50.2	91.8	200.8	1721.4	12.8	18.9
23:54	567.0	3.1	98.6	50.2	92.4	.0	1789.6	12.5	22.1
00:04	532.9	3.1	99.3	50.2	92.8	.0	1883.8	12.4	21.8
00:14	513.1	3.1	100.0	50.2	93.5	.0	1903.8	12.4	21.6
00:24	472.3	3.1	100.9	50.2	94.1	.0	2052.1	12.4	21.1
00:34	433.8	3.1	101.8	50.2	94.9	18.2	2032.1	12.4	20.8

TABLE A5·3 Cont.

00:44	408.8	3.1	102.8	50.2	95.7	.0	2088.2	12.9	20.8
00:54	387.9	3.1	103.9	50.2	96.6	.0	2096.2	13.1	20.6
01:04	357.5	3.1	105.0	50.2	97.5	.0	2160.3	13.2	20.4
01:14	164.8	2.1	104.5	50.2	91.9	.0	2198.4	13.2	19.1
01:24	160.7	2.1	104.0	50.2	89.4	.0	2230.5	13.1	18.3
01:34	152.7	2.1	104.5	50.2	89.0	.0	2234.5	13.0	17.8
01:44	141.3	2.1	105.3	50.2	88.9	.0	2284.6	13.0	17.6
01:54	129.9	2.1	106.1	50.2	89.1	.0	2312.6	12.9	17.2
02:04	124.0	2.1	107.0	50.2	89.4	.0	2360.7	12.8	16.9
02:14	114.9	2.1	107.9	50.2	90.1	.0	2358.7	12.7	16.6
02:24	106.1	2.1	108.9	50.2	91.1	.0	2358.7	12.7	16.3
02:34	97.6	2.1	109.9	50.2	92.2	.0	2438.9	12.5	15.9
02:44	93.2	2.1	110.8	50.2	93.2	.0	2398.8	12.0	15.2
02:54	84.8	2.1	111.7	50.2	93.8	.0	2418.8	11.9	14.9
03:04	80.0	2.1	112.6	50.2	94.5	.0	2454.9	12.3	15.1
03:14	74.1	2.1	113.5	50.2	95.6	.0	2402.8	12.5	15.2
03:24	67.5	2.1	114.3	50.2	96.2	.0	2573.1	12.7	15.3
03:34	69.7	2.1	114.5	50.2	99.3	.0	2537.1	12.7	15.4
03:44	65.3	2.1	115.3	50.2	95.8	.0	2481.0	12.8	15.3
03:54	62.4	2.1	115.9	50.2	97.7	.0	2499.0	12.9	15.4
04:04	56.9	2.1	116.6	50.2	98.2	.0	2541.1	13.1	15.5
04:14	61.3	2.1	116.5	50.2	90.4	.0	2509.0	13.6	13.7
04:24	58.7	2.1	117.1	50.2	98.0	.0	2491.0	13.6	16.1
04:34	56.9	2.1	117.6	50.2	96.7	.0	2545.1	13.2	15.5
04:44	52.1	2.1	118.0	50.2	97.9	.0	2487.0	12.9	15.1
04:54	51.4	2.1	118.5	50.2	99.1	.0	2573.1	13.3	15.6
05:04	48.1	2.1	118.8	50.2	99.4	.0	2545.1	13.5	15.7
05:14	45.9	2.1	119.2	50.2	99.7	.0	2499.0	13.6	15.8
05:24	45.1	2.1	119.5	50.2	99.9	.0	2537.1	13.7	15.8
05:34	42.2	2.1	119.8	50.2	100.3	.0	2573.1	13.8	15.8

TABLE A5·3 Cont.

05:44	43.7	2.1	120.1	50.2	100.7	.0	2587.2	13.8	15.8
05:54	41.5	2.1	120.3	50.2	101.2	.0	2583.2	13.8	15.8
06:04	41.8	2.1	120.5	50.2	101.4	.0	2583.2	13.8	15.9
06:14	41.1	2.1	120.7	50.2	101.5	.0	2583.2	13.7	15.7
06:24	38.9	2.1	120.9	50.2	101.4	.0	2637.3	13.4	15.4
06:34	37.8	2.1	121.0	50.2	101.8	.0	2537.1	13.4	15.3
06:44	38.9	2.1	121.2	50.2	101.9	.0	2637.3	13.6	15.5
06:54	8.1	3.8	119.8	50.2	100.2	.0	2499.0	13.7	13.8
07:04	9.5	3.8	109.0	50.2	83.8	.0	2629.2	14.0	14.1
07:14	6.6	3.8	99.4	50.2	66.4	.0	2637.3	14.2	14.3
07:24	5.9	3.8	90.9	50.2	55.0	.0	2537.1	14.2	14.3
07:34	.0	3.7	83.3	50.2	55.4	11.5	2529.0	14.1	14.2
07:44	.0	3.7	76.9	50.2	56.2	130.7	2543.1	14.3	14.3
07:54	.4	3.7	71.6	50.2	55.1	95.0	2529.0	14.7	14.8
08:04	1.8	3.8	70.6	50.2	56.2	.0	2527.0	14.5	14.7
08:14	390.1	2.1	71.4	50.2	55.0	.0	2711.4	14.6	15.0
08:24	234.5	2.3	82.0	50.2	60.5	.0	2609.2	14.5	14.9
08:34	207.0	2.3	87.8	50.2	78.1	50.5	2637.3	14.3	21.0
08:44	185.0	2.3	89.3	50.2	79.4	12.6	2693.4	14.6	20.4
08:54	7.3	3.8	90.8	50.2	78.0	49.0	2603.2	14.7	16.4
09:04	9.2	3.8	91.2	50.2	68/9	.0	2609.2	14.5	14.6
09:14	8.4	3.8	91.2	50.2	61.2	.0	2655.3	14.2	14.4
09:24	9.2	3.8	91.1	50.2	55.0	.0	2601.2	14.1	14.2
09:34	1.5	3.8	90.5	50.2	55.0	.0	2581.2	13.7	13.8
09:44	2.6	3.8	90.5	50.2	55.0	70.9	2571.1	13.7	13.8
09:54	8.1	3.8	90.9	50.2	66.3	.0	2587.2	14.1	14.5
10:04	5.5	3.8	93.4	50.2	77.4	.0	2693.4	14.1	14.4
10:14	7.3	3.8	89.5	50.2	67.4	36.4	2615.2	14.1	14.2
10:24	7.0	3.8	81.9	50.2	58.5	.0	2601.2	14.2	14.3
10:34	.0	3.7	74.6	50.2	58.3	.0	2563.1	14.0	14.1

TABLE A5.4 Computer Control Trial Logsheet

Resin Type: C

Date 13 February 1986

Time	Observations
21.15	Procedure begin loaded and operation started Procedures steamset and coltop loaded automatically and operating. Prompt generated to request resin selection, Type C selected, raw material requirements displayed. Dry charge selected and all flag files created and initialised.
22.45	Procedure dry loaded and operation begun. AD Tare wt calculated to be 146 kg. Raw material charge quantities requested and entered. Water to be removed during dehydration stage including tare value calculated as 2256 kg and total water off to end of acid reaction stage calculated as 2786 kg. Flag files for steam on and charging have had values altered. Procedures opfiles and data logs loaded and operating. procedure prodat, still-log and transfer loaded automatically by procedure datalogs.
22.55	Steam Pressure set to 3 bar
23.05	Set point controllers set to remote operation.
23.30	Procedure inout loaded and operation started. Procedure addxylol loaded and operated automatically from inout.
23.32	Prompt generated to request acknowledgement when boiling occurs. Reactor steam pressure set at 0.5 bar.
23.40	Boiling of reactor contents commenced. On set of boiling acknowledged.
23.55	Xylol addition prompt generated. Xylol charge made to reactor.

TABLE A5.4 Continued

23.59	Procedure buoh2add loaded automatically and operating after acknowledgement for Xylol charging complete.
00.00	Steam pressure raised to 3 bar.
02.55	Prompt generated for butanol(2) addition.
03.00	Procedure acatad loaded automatically and operating after acknowlegement for butanol(2) charge completion made.
05.51	Steam pressure dropped to 0.5 bar at end of dehydration stage. Acid catalst added.
06.09	Procedure acidreact loaded automatically and operating after addition of acid catalyst. Steam pressure set at 2 bar and column top temperature set to 92°C.
06.15	Control process ended ready for vac stripping. Probably due to electrical spike affecting value for aqueous distillate tank weight.

TABLE A5·4 Computer Generated Logsheet

TIME	STEAM FLOW KG/HR	COIL PRESS BAR	STILL TEMP DEGC	DIST degC DEGC	COLUMN TOP TEM DEGC	A.D.' OFF L/HR	ALCO TANKWT Kg	CF.TEMP DEGC	CR.TEMP DEGC
22:42	.0	3.6	30.2	10.6	55.0	.0	154.3	9.8	9.6
Steam Pressure set at 3 bar at 22:43									
22:52	.0	3.6	30.0	10.5	55.0	.0	156.3	8.7	8.4
23:02	1496.6	2.9	40.7	10.4	55.0	.0	134.3	7.7	7.3
23:12	1334.8	2.9	64.8	10.3	55.0	.0	126.3	8.0	7.6
Steam pressure set at 0.5 bar at 23:20									
23:22	161.8	.9	91.7	10.8	85.7	.0	182.4	8.8	15.9
23:32	76.0	.5	94.0	11.0	92.4	.0	176.4	9.4	14.2
23:42	79.3	.5	93.8	11.6	92.4	.0	190.4	10.3	14.7
Steam Pressure set at 3 bar at 23:48									
23:52	1237.5	3.0	91.9	17.7	90.5	522.7	214.4	11.5	27.2
00:02	1242.7	3.0	91.9	18.2	90.6	81.7	268.5	11.7	27.4
00:12	1225.8	3.0	91.9	17.6	90.7	166.3	300.6	11.2	26.7
00:22	1210.0	3.0	91.9	16.9	90.8	766.5	366.7	10.8	26.1
00:32	1194.2	3.0	91.8	16.4	90.8	500.8	400.8	10.4	25.4
00:42	1179.2	3.0	91.8	16.2	90.9	.0	468.9	10.0	25.2
00:52	1184.7	3.1	91.8	15.7	91.0	.0	511.0	9.7	24.8
01:02	1159.4	3.0	91.8	15.4	91.0	11.1	545.1	9.3	24.4
01:12	1144.7	3.1	91.8	15.1	91.1	95.0	611.2	9.0	23.9
01:22	1134.4	3.1	91.8	14.6	91.1	691.9	677.4	8.9	23.4
01:32	1121.6	3.1	91.8	14.4	91.2	36.4	701.4	8.7	23.3
01:42	1120.8	3.1	91.8	14.2	91.2	397.9	763.5	8.5	23.0
01:52	966.3	2.7	91.8	14.0	91.3	71.3	849.7	8.2	22.8
02:02	1035.3	2.8	91.8	13.0	91.3	687.1	889.8	8.0	21.1
02:12	1081.5	3.1	91.9	13.3	91.3	.0	943.9	7.8	21.8
02:22	1048.9	3.1	91.9	13.2	91.3	549.0	1000.0	7.7	21.7
02:32	1060.6	3.1	92.0	13.5	91.4	583.2	1052.1	8.2	22.1
02:42	1058.8	3.1	91.8	11.5	83.4	.0	1134.3	9.9	11.1
02:52	1022.1	3.1	92.8	16.7	91.3	235.7	1190.4	11.1	25.3
03:02	1008.9	3.1	92.9	17.2	91.4	231.6	1274.5	11.5	26.0

TABLE A5·4 Cont.

```
---------------------------------------------------------------------------------
03:12     960.8   2.9     93.1     16.3     91.5     369.4    1308.6  10.6     24.5
---------------------------------------------------------------------------------
03:22     1001.5  3.2     93.3     15.7     91.6     .0       1372.7  10.0     24.1
---------------------------------------------------------------------------------
03:32     1010.4  3.2     93.6     15.2     91.8     143.7    1460.9  9.6      23.2
---------------------------------------------------------------------------------
03:42     893.6   3.1     93.9     14.9     92.0     .0       1589.2  9.0      22.7
---------------------------------------------------------------------------------
03:52     870.5   3.1     94.2     14.4     92.4     531.6    1667.3  8.5      21.9
---------------------------------------------------------------------------------
04:02     814.4   3.1     94.8     13.7     92.7     477.4    1649.3  8.1      20.8
---------------------------------------------------------------------------------
04:12     770.3   3.1     95.4     13.4     93.4     434.3    1721.4  8.2      20.3
---------------------------------------------------------------------------------
04:22     724.5   3.1     96.2     14.2     94.0     374.2    1883.8  9.4      20.8
---------------------------------------------------------------------------------
04:32     701.3   3.1     97.0     14.9     94.8     351.9    1973.9  10.2     21.2
---------------------------------------------------------------------------------
04:42     640.4   3.1     98.0     15.3     95.6     321.1    2006.0  10.9     21.4
---------------------------------------------------------------------------------
04:52     611.8   3.1     98.9     15.6     96.5     262.8    2010.0  11.4     21.4
---------------------------------------------------------------------------------
05:02     564.4   3.1     100.0    15.8     97.4     200.1    2088.2  11.7     21.3
---------------------------------------------------------------------------------
05:12     529.9   3.1     101.1    15.9     98.4     197.1    2142.3  11.9     21.2
---------------------------------------------------------------------------------
05:22     496.9   3.1     102.1    15.1     99.3     153.7    2126.2  11.1     19.9
---------------------------------------------------------------------------------
05:32     461.0   3.1     103.1    13.7     100.2    124.7    2198.4  9.7      18.2
---------------------------------------------------------------------------------
05:42     449.2   3.1     103.6    11.8     100.4    144.8    2174.3  8.9      16.0
---------------------------------------------------------------------------------
Steam pressure set at 0.5 bar at 05:51
---------------------------------------------------------------------------------
05:52     425.7   3.1     104.5    12.2     101.5    67.2     2198.4  8.6      16.3
---------------------------------------------------------------------------------
Column Top Temperature set at 05:56 Set value =90 degC
---------------------------------------------------------------------------------
Steam Pressure set at 2 bar at 05:56
---------------------------------------------------------------------------------
Steam switched off to await Solvent Stripping at 05:59
---------------------------------------------------------------------------------

Formalin(litres)        2738.0

Butanol(1) (litres)        3050.0

Butanol(2) (litres)        726.0

Iso-Butanol(1) (litres)        .0

Iso-Butanol(2) (litres)        .0

Wet Butanol(1) (litres)        .0

Wet Iso-Butanol(1) (litres)        .0




Xylol (litres)        415.0

Acid Catalyst (litres)        3500.0

Antifoam Soln (ml)        1075.0

Melamine (Kg)        .0
```

TABLE A5.5 Computer Control Trial Logsheet

Resin Type: B

Date 25 February 1986

Time	Observation
09.03	Procedure begin loaded and operation started. Resin Type B selected as dry charge. Procedures steamset and coltop loaded automatically and operation started. All flag files created and initialised.
09.08	Procedure dry loaded and operating correctly. Charging commenced. AD tank tare weight determined as 109 kg. Water to be removed by end of dehydration stage calculated as 2257 kg including tare weight of tank. Water to be removed by end of acid reaction stage calculated as 2807 kg including tare weight of tank.
10.07	Charge completion acknowledg ed. Steam pressure automatically adjusted to 3 bar. Procedure prodat, still-log and transfer loaded and operating correctly.
10.15	Procedure inout loaded and operating. Procedure addxylol loaded automatically.
10.48	Prompt operated to check for boiling. Pressure dropped to 0·5 bar.
11.02	Acknowlegment made on commencement of boiling of reactor contents.
11.17	Prompt generated for addition of Xylol to reactor,
11.20	Charging of Xylol completed.
11.21	Procedure buoh2add loaded and operating. Steam pressure set at 3 bar.
14.27	Prompt generated for addition of second butanol charge to reactor. Charge made and acknowledged.

TABLE A5.5 Continued

14.18	Procedure adacat loaded automatically and operating
15.58	Pressure dropped to 0.5 bar. Mass of Aqueous Distillate removed = 2254 kg. Temperature of reactor contents = 103.1°C.
16.03	Prompt generated for acid catalyst addition and acknowledgement made when addition made.
16.09	Procedure acidreact loaded automatically and operating. Steam pressure set at 2 bar. Column top temperature controller set to 92°C.
21.45	Required amount of aqueous distillate removed, steam switched off to await vacuum stripping operation. Still appears to be small oscillation on aqueous distillate tank weight value.

TABLE A5·5 Computer Generated Logsheet

```
Resin Type - BE645   Batch no. - 410   Shift - A


Charging commenced at  - 09:08

Formalin  - 2738. litres
Formalin water content  - .5261
Density of Formalin  - 1.1228 Kg/M3

A.D. Tank Tare Value  - 109.4184 Kg
n-Butanol(1) charged  - 3050. litres

Melamine charged  - 1075. Kg

Water content of charge  - 1617.35051 Kg

Water off during dehydration  - 2256.76891 Kg
Water off during Acid Reaction  - 2806.76891 Kg
```

TABLE A5·5 Cont.

TIME	STEAM FLOW KG/HR	COIL PRESS BAR	STILL TEMP DEGC	DIST degC DEGC	COLUMN TOP TEM DEGC	A.D. OFF L/HR	ALCO TANKWT Kg	CF.TEMP DEGC	CR.TEMP DEGC
10:11	.0	3.5	34.1	12.1	55.0	.0	122.2	11.1	11.0
Steam Pressure set at 3 bar at 10:12									
10:21	.0	3.5	33.3	12.1	55.0	.0	108.2	11.4	11.2
10:31	1419.9	2.8	36.6	12.2	55.0	.0	122.2	11.2	11.0
10:41	1408.2	2.9	59.6	12.3	55.0	.0	82.2	11.0	10.9
Steam Pressure set at 0.5 bar at 10:48									
10:51	75.6	.5	88.1	12.3	55.0	.0	102.2	10.9	10.7
11:01	73.0	.5	94.1	12.9	92.4	.0	162.3	11.2	16.5
11:11	75.2	.5	94.0	13.0	92.8	.0	148.3	11.5	16.1
11:21	87.3	.5	92.8	12.8	91.4	.0	166.3	11.9	13.8
Steam Pressure set at 3 bar at 11:22									
11:31	1050.4	3.1	94.2	16.9	91.0	.0	228.5	12.9	24.4
11:41	1078.2	3.0	95.1	25.9	92.1	1520.1	400.8	14.2	27.3
11:51	1015.5	2.9	94.8	23.0	92.3	209.0	607.2	15.4	31.7
12:01	1088.2	3.1	93.7	21.4	92.1	561.3	683.4	14.1	30.2
12:11	1071.3	3.1	93.2	19.2	92.0	525.6	797.6	12.8	28.1
12:21	1062.1	3.0	93.2	17.8	92.0	526.0	859.7	11.8	26.8
12:31	1059.9	3.1	93.2	17.0	92.0	522.3	945.9	11.2	25.9
12:41	979.5	3.0	93.2	16.4	92.0	533.1	1056.1	10.7	25.2
12:51	990.2	3.1	93.3	18.0	92.0	604.0	1152.3	12.4	26.9
13:01	998.6	3.1	93.4	19.4	92.0	610.3	1266.5	13.8	28.2
13:11	974.8	3.1	93.5	20.6	92.1	589.5	1358.7	15.0	29.4
13:21	934.7	3.1	93.7	20.1	92.2	605.4	1460.9	14.6	28.7
13:31	895.8	3.1	93.9	18.5	92.3	577.2	1547.1	13.1	26.9
13:41	873.8	3.1	94.2	17.0	92.5	569.1	1667.3	11.9	25.2
13:51	831.6	3.1	94.6	16.2	92.8	535.7	1693.4	11.3	24.1
14:01	780.6	3.1	95.0	15.9	93.1	464.8	1813.6	11.4	23.7
14:11	754.6	3.1	95.5	17.5	93.5	429.1	1871.7	13.1	25.0
14:21	792.0	3.1	95.9	16.9	92.9	401.3	1913.8	14.6	23.5
14:31	743.2	3.1	97.5	19.9	95.4	337.4	1941.9	15.3	26.9
14:41	712.7	3.1	98.4	18.8	96.2	311.4	2014.0	14.0	25.4

TABLE A5·5 Cont.

14:51	661.7	3.1	99.1	17.6	96.9	270.6	2090.2	12.8	23.8
15:01	599.3	2.9	100.0	16.3	97.7	210.5	2096.2	12.0	21.9
15:11	288.1	2.0	100.7	14.7	98.3	114.0	2152.3	11.3	18.9
15:21	247.0	2.0	101.1	15.1	98.6	99.1	2198.4	12.3	18.9
15:31	227.2	1.9	101.6	15.8	99.0	95.8	2178.3	13.3	19.5
15:41	220.6	1.9	102.2	17.4	99.5	77.6	2230.5	14.9	21.1
15:51	205.5	1.9	102.7	17.2	100.0	71.3	2252.5	14.5	20.6
Steam Pressure set at 0.5 bar at 15:57									
16:01	.4	.5	102.9	14.7	100.3	.0	2308.6	13.3	15.9
Column Top Temperature set at 16:09 Set value =90 degC									
Steam Pressure set at 2 bar at 16:09									
16:11	260.2	2.0	101.1	14.5	91.8	132.9	2242.5	12.5	18.3
16:21	250.7	2.1	57.4	14.5	89.6	236.5	2270.5	11.8	18.9
16:31	225.7	2.1	101.1	13.9	89.4	215.7	2326.6	11.4	18.0
16:41	220.9	2.1	59.1	14.7	89.4	183.0	2362.7	12.6	18.8
16:51	204.4	2.1	59.2	16.5	89.4	161.8	2398.8	14.6	20.5
17:01	185.7	2.0	103.0	17.5	89.4	153.7	2509.0	15.2	21.2
17:11	174.0	2.1	60.3	16.1	89.4	134.4	2481.0	13.9	19.5
17:21	159.3	2.1	104.8	14.9	89.5	115.8	2487.0	12.9	18.0
17:31	144.2	2.1	60.1	13.7	89.6	102.5	2519.0	11.6	16.4
17:41	133.6	2.1	106.6	13.1	89.8	92.1	2491.0	11.4	15.8
17:51	121.5	2.0	61.2	14.2	90.0	80.9	2583.2	12.7	16.9
18:01	112.3	2.1	108.6	15.3	90.4	78.7	2637.3	14.0	18.0
18:11	100.2	2.1	109.6	16.4	91.2	65.3	2639.3	15.1	18.9
18:21	106.1	2.1	109.6	14.9	96.1	.0	2637.3	14.1	16.7
18:31	93.6	2.0	110.9	13.9	92.6	58.3	2615.2	12.3	15.8
18:41	83.7	2.0	62.2	12.4	94.2	39.7	2641.3	11.2	14.3
18:51	81.8	2.0	62.1	12.9	95.4	34.5	2711.4	11.9	14.9
19:01	73.8	2.0	113.4	13.6	95.9	.0	2715.4	12.7	15.6
19:11	68.3	2.1	62.5	14.2	97.1	235.7	2673.3	13.5	16.3
19:21	62.4	2.0	114.7	14.8	96.7	.0	2729.4	14.1	16.9
19:31	66.1	2.1	62.1	14.5	103.0	.0	2765.5	14.8	16.7
19:41	60.6	2.1	62.7	15.0	97.0	.0	2747.5	14.0	16.8

TABLE A5·5 Cont.

19:51	55.8	2.1	115.6	13.7	98.1	.0	2711.4	12.6	15.3
20:01	53.2	2.1	63.1	12.6	98.9	21.5	2715.4	11.5	14.1
20:11	50.3	2.0	116.4	11.7	99.4	.0	2765.5	10.7	13.1
20:21	47.0	2.1	116.8	12.0	99.7	.0	2821.6	11.4	13.7
20:31	44.8	2.0	117.1	13.3	100.0	20.4	2765.5	12.8	15.1
20:41	41.5	2.1	117.3	14.7	100.1	.0	2747.5	14.3	16.6
20:51	39.6	2.1	117.6	15.7	100.3	.0	2765.5	15.4	17.7
21:01	37.4	2.0	117.8	14.4	100.6	.0	2739.5	13.5	15.9
21:11	37.8	2.0	117.9	12.9	100.9	.0	2821.6	11.9	14.2
21:21	38.2	2.0	118.1	11.8	101.2	.0	2745.5	11.1	13.2
21:31	36.0	2.1	118.3	12.8	101.4	.0	2729.4	12.4	14.5
21:41	33.4	2.1	118.4	14.0	101.4	.0	2753.5	13.5	15.7

Steam switched off to await Solvent Stripping at 21:47

TABLE A5·5 Cont.

Formalin(litres)	2738.0
Butanol(1) (litres)	3050.0
Butanol(2) (litres)	726.0
Iso-Butanol(1) (litres)	.0
Iso-Butanol(2) (litres)	.0
Wet Butanol(1) (litres)	.0
Wet Iso-Butanol(1) (litres)	.0
Xylol (litres)	415.0
Acid Catalyst (litres)	4.5
Antifoam Soln (ml)	500.0
Melamine (Kg)	1075.0
Urea (Kg)	.0

APPENDIX 6

Computer and Software Operating Procedure

SOFTWARE OPERATING PROCEDURE

1. Load System Disc in Drive A.

2. Load Data Disc in Drive B.

3. Reset system by pressing reset button on front of computer.

4. Enter begin↵ via the keyboard.

   You will then be prompted to enter the resin type to be selected for manufacture, this should be entered in the form BEXXXX

   A menu table will then be displayed giving charge quantities for dry and wet type batches. The appropriate code should be entered for the batch type selected. Once this has been done the necessary data files are created on the disc in Drive B.

5. Enter either dry ↵ or wet ↵ depending on which batch type has been selected for the chosen resin. Prompts are then generated listing the charges to be made and acknowledgement of the charge completion is by entering the quantity charged at the prompt.

   When the charging has finished all the necessary flag files are created and initialised.

6. Load program disc in Drive O on BBC Computer.

7. Enter 'Load Graphics'↵ .

8. Enter Run ↵ the graphics program is now operating on the BBC computer.

9. Enter datalogs ↵ on SEED computer. This loads and begins the operation of the data logging procedures.

10. Enter inout ↵. This procedure carries out monitoring of flag files and initiates procedures automatically at the various stages of the process. The operator should follow the prompts generated by the computer and make the appropriate acknowledgements when requested. The program will terminate the process at the correct time.

APPENDIX 7

Table of Steps for Vacuum Stripping Resin

TABLE A7.1 Table of Steps for Vacuum Stripping Resin

Step No.	Time Secs.	Time Step Secs.	Vacuum mm/ Hg	Vacuum Step mm/Hg
0	0	0	760	0
1	240	240	560	200
2	600	360	500	60
3	1200	600	300	200
4	1800	600	200	100

APPENDIX 8

Digital - Analogue Converter Configuration

SEED DA-1 INSTRUCTIONS

The SEED 6K8008a interface board is a dual 8 BIT Digital to Analogue converter for use in the range of SEED System 1 and 19 micro computers.

The design is based around two 8 BIT monolithic D to A converters which use an advanced form of R-2R ladder network to ensure full range accuracy and linearity.

The board is designed to be installed on the SS30 bus of any SEED System 1 to 19 Development System.

SPECIFICATIONS

Settling Time	2u Secs Max
Linearity	0.5 L.S.B.

DESCRIPTION

The converters are interfaced to the bus via a Motorola type Paralell Interface Adapter (68b21 P.I.A.) using the two 8 bit ports directly coupled to the converter's inputs. Also available from the P.I.A.'s are four control lines not required by the circuit, which have been made available to the user at the connector. One of which has a general purpose transistor connected to provide for a higher current drive if required (up to 800 mA). The load resistor of which may be removed if an open collector configuration is more desirable, the load voltage may be up to 40V. However, the total power dissipation of the transistor should not exceed 500 mW.

CALIBRATION

This is a very simple operation and is performed as follows :-

1. Set all the bits to an 'O' state.

2. Adjust R6(R3) until the output is at 0.00V.

3. Set all the bits to a '1' state.

4. Adjust R7(R2) until the maximum range is obtained.

If a bipolar span is desired the offset (R6/R3) may be adjusted further to suit the requirements.

The variable resistors mentioned above in brackets refer to channel 'B'.

CONNECTION LIST

The external connections are via a 10 way, 0.1in pitch connector on the edge of the P.C.B.

			Port 4	Port 6
Pin	1	+5v DO NOT EXCEED 500mA		
	2	CA1		
	3	GND (OV)		
	4	CA2		
	5	'A' SIDE O/P	16	3
	6	GND	15	2
	7	'B' SIDE O/P	14	1
	8	CBI		
	9	GND		
	10	CB2 Thru' trl (May be open collector)		

OPERATION

The board may be installed in the target system in any of the SS30 pin bus slots which determines the physical address of the board, the addresses of the registers below are relative to that physical address.

0	P.I.A. 'A' side Data/Direction
1	P.I.A. 'A' side control register
2	P.I.A. 'B' side Data/Direction
3	P.I.A. 'B' side control register

A more detailed description of the operational parameters and functions of the P.I.A. can be found in the relevant Data sheets, however here is a simplified description of the initialisation and use of the device for this application.

The register at addresses 0 and 2 above are dual purpose, i.e., at times they are the peripheral data registers, at others they are the 'direction of data' registers. This is determined by the DDR bit in the relevant control register. When this bit is high, register 0 and 2 are the interfaces peripheral data registers.

i.e. the data written to regs 0 and 2 go directly to the peripheral pins.

When in input mode, any signals applied to the peripheral pins may be read by the processor from regs 0 and 2.

When the DDR bit is low, register 0 and 2 are the Data Direction registers, i.e., the pattern of 'O's and '1's written into the register determine which of the peripheral data lines will be inputs or outputs.

e.g. If the DDR bit is LOW and the pattern OF hex (00001111) is written to register 0 then PA4 - PA7 will be sent to inputs and PAO - PA3 will be outputs.

For this application all the peripheral data lines must be set as outputs (FF hex, 11111111 bin).

REFERENCES

1. BIP Chemicals Limited, Popes Lane, Oldbury, Warley, West Midlands, B69 4PD, Technical Literature.

2. Okano, M. and Ogata, Y., J. Am. Chem. Soc., **74**, (1952), 5728.

3. Ebdon, J.R. and Heaton, P.E., Polymer, **18** (1977), 971-974.

4. Freeman, P.A., Control Parameters in the Batchwise Production of Synthetic Resins, M.Phil. Thesis, University of Aston, Chem. Eng. Dept. (1985).

5. Fraude, D.J., Instrumentation in the Chemical and Petroleum Industries, Symposium Proceedings, **16**, (1980), 75-94.

6. Uhlig, R.J., Digital Computer Applications to Process Control, ed., Van-Nouta Lemke, Pub., North Holland Publishing Company, (1977), 165-171.

7. Hughes, W.R., Real-Time Computing with Applications to Data Acquisition and Control, ed. D.A. Mellichamps, Pub. Van Nostrad Reinhold, (1983), 235-278.

8. Wright, J.D. and White, J.M., Real-Time Computing with Applications to Data Acquisition and Control, ed. D.A. Mellichamp, Pub. Van Nostrad Reinhold, (1983), 345 -378.

9. Shneiderman, B., Computer Surveys, **16**, No.3, (1984), 265-285.

10. Mehta, N., Proceedings of the International Conference on Systems, Man and Cybernetics: Bombay and New Delhi, **2**, (1983), 803-806.

11. Reilly, S.S. and Roach, J.W., IEEE Computer Graphics and Application, **4**, (1984), 42-51.

12. Sapita, R.F., Process Automation, **2**, (1982), 75-81.

13. Gordon, M., Halliwell, A. and Wilson, T., J. Appl., Poly. Sci, **10**, (1966), 1153-1170.

14. Aldersley, J.W., Gordon, M., Halliwell, A. and Wilson, T., Polymer, **9**, (1968), 395.

15. Tamita, B., J. of Poly. Sci. <u>15</u>, (1977), 2347-2365.

16. Patel, V.H., BIP Chemicals Limited/Aston University, Teaching Company Scheme, Technical Report, (January 1985).

17. Microware Systems Corporation, 5835 Grand Avenue, Des Moines, Iowa 50304, OS-9 Operating System Users Manual, (1981).

18. Gandhi, B.B., Some Quantitative Analytical Methods for the Analysis of Wet Solvent Stream Composition, RSC Project, Warley College of Technology, (1984).

19. Bowden, A.D., Private Communications, BIP Chemicals Limited.

20. Freeman, P.F., BIP Chemicals Limited/Aston University, Teaching Company Scheme, Technical Report, (January 1985).

21. Patel, V.H., Chemical Kinetic Investigation of a Commercial Batch Reactor Process, Ph.D. Thesis, University of Aston, Chem. Eng. Dept. (1987).

22. Foxboro-Yoxall Limited, Redhill, Surrey, Instruction Manual.