AN INVESTIGATION OF CONTROL STRATEGIES FOR BATCH AND CONTINUOUS DISTILLATION COLUMNS, USING A DIGITAL COMPUTER

BY

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SYNOPSIS

SYNOPSIS

The work undertaken has been principally concerned with the practical application of a number of control ideas to a pilot scale distillation unit.

The ideas under investigation covered two main areas - namely, the control of individual loops, and the choice of overall operation strategy.

For the control of the individual loops, a number of digital control algorithms were developed. Initially, programmes were written to represent simple three term controllers, but the work was extended to also include specialist controllers. These controllers included non-linear algorithms, cascade configurations, and routines to process the measured variable signal. In addition, a controller using a simple piece of digital logic was also investigated, and found to function extremely well.

The second area of investigation was into the development of overall operating strategies, and has been centred principally on batch distillation. Here the main concern has been the optimisation of the time dependent reflux ratio.

Initially, a theoretical investigation of the conventional operating techniques was undertaken. The batch distillation tasks were then optimised using Dynamic Programming.

The processes considered were more amenable to constant

reflux ratio operation than fixed top product composition, and when compared with constant reflux, the optimal strategy indicated savings of the order of 7% of total energy requirement.

A practical investigation was also undertaken, whereupon savings of about 5% were indicated when using the optimal reflux strategy.

The process did not follow the predicted path, and therefore, a routine was developed to up-date the optimal reflux strategy during the experimental runs.

Finally, as a result of experience gained from the experimental work, the snatch operating technique was devised. This consisted of constantly running the distillation unit at total reflux, except for the periodic snatching of distillate from the reflux drum.

The experimental investigation of this technique indicated a reduction in total heat requirement, and hence run time, of the order of 30%, when compared with the constant reflux ratio technique.

A description of the development and application of the digital controllers, together with an outline of the work on the optimal reflux strategy, was presented in Brighton at the International Symposium on Distillation (1).

1. KROPHOLLER H.W. and LEWINGTON S.

   Paper presented at the INTERNATIONAL SYMPOSIUM on DISTILLATION, Brighton, England, 1969.

INTRODUCTION

INTRODUCTION

There is a growing interest in improving the control of chemical processes.

It has now been realized that the original procedure of installing automatic control on a plant, and adjusting the control parameters by 'rule-of-thumb' technique is often not very successful. (2).

Computer control of chemical processes was first introduced about 10 years ago, but a high proportion of installations now in operation are simply the digital equivalent of two - or three - term analogue controllers.

Computer control may be implemented in two modes, namely direct digital control, and supervisory control. As the names suggest, with the former the computer acts as the controller, and with the latter, the computer merely adjusts the set-points of analogue controllers.

The advent of the digital computer in the control field, has resulted in a new sprectrum of control tasks with their own inherent problems. This is best illustrated by the growing desire to add the task of operating the controlled unit in an optimal manner, to the standard function of holding the controlled variables at their desired values. (3,4).

The desire to operate complete chemical units in an optimal manner has led to the development of new techniques in control, such as multi-variable control, steady-state optimisation, dynamic optimisation, sub-optimal control, adaptive control etc. For the implementation of these optimal control strategies, it is often necessary to ascertain a certain degree of information about the dynamics of the process and its control system.

The computer has also resulted in a new range of control algorithms, designed to handle sampled data, and with a flexibility in operation, governed almost solely by the speed of the digital machine in use. As a consequence, the advent of the digital computer as a controller has meant that for a given process a greater range of process variables are available as controlled variables.

Finally, from the use of the digital computer as a control instrument, small units of sophisticated logic for control purposes have evolved.

This may be illustrated in several ways. There are the units of electronic logic designed to accept the output from a chromatograph, and process the signal such that it may be employed as the measured variable in control loop. The controller for this loop may be an integrated part of the signal processing logic, or may be an independent unit. Small units of digital logic may be built to act as controllers, based either on a simple algorithm, or on one of considerable sophistication.

In addition, the major pneumatic controller manufacturers are now marketing pneumatic units of considerable sophistication, designed as 'Tailor Made' units to suit individual requirements.

When considering the economic justification for the installation of a digital control computer, it must be realized the straight-forward replacement of analogue instrumentation by the digital equivalent will make little difference.

However, the real economic benefits which accrue from computer control are gained by using its full power and flexibility. This may be illustrated by the implementation of various modes of control (including cascade control, and both feed forward and feed back control, and by use of non-linear controllers where advantageous). (5).

In addition, the computer may be used for a variety of other tasks, including, optimization routines, alarm scanning, and data logging.

The application of the controller to the batch distillation column where the column had been partially designed for ease of control, illustrates the important point that a slight redesign of the process plant to suit the control scheme is often simpler than to endeavour to design complex control systems to suit the existing plant (6).

LITERATURE SURVEY

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LITERATURE SURVEY.

3. INTRODUCTION.

Distillation operations may be carried out in one of two ways, either according to the continuous distillation technique, or according to the batch distillation technique.

Although, it is fashionable to consider that continuous units are better than batch units, this is not always the case. One must consider the distillation task concerned, and the relative merits of the two techniques. A typical case where it would be advantageous to employ a batch distillation procedure, is when the separation of a multi-component mixture is considered. In this example, a simple batch column only need be employed, instead of a train of continuous columns.

When one considers the control of distillation columns in general, the problem may be sub-divided into various sections. The first task which must be considered is the control of the process variables at their desired values, whether these be constant or variable. The second problem is to define optimal operation, and to control the column such that it is operated in an optimal manner.

Finally, with batch distillation there is an additional variable. It is the time dependent reflux ratio, and the fact that the conditions within the batch column are constantly altering with time, whereas within the continuous column ideally steady state conditions exist.

The control of distillation units by conventional analogue controllers is comprehensively covered in a number of books (7,8,9,10).

The use of analogue controllers for feed forward control of chemical plant has been limited to ratio control, and other simple applications (11).

The earlier application of digital computers consisted of superimposing the computer on an analogue feed-back system (12, 13). This was in the form of supervisory control, or feed-forward control by adjusting the set-points of the analogue controllers.

This earlier work has led to the replacement of the conventional analogue controller by the digital computer (14, 15, 16).

The control of a distillation unit by a computer may be considered as consisting of two objectives.

The first objective is the development of control algorithms designed to both cope with the sampled data nature in which the readings are taken from the plant, and to make use also of the increased power and flexibility of the controller.

The second objective is the development of techniques which may be employed to operate the column in a more efficient manner (here the object is usually to operate the column in an optimal or a sub-optimal manner).

An optimising control system may be defined as a system which attempts to get the best performance from a plant or process according to a criterion such as maximum production or minimum cost (17).

This task of the computer may be further sub-divided into off-line and on-line analysis of the problem.

The off-line analysis is usually based on either a steady-state model or a dynamic model. In addition, an insight into the dynamic behaviour of the system is usually imperative to control the system satisfactorily. The on-line analysis requires a model of the process which may be solved faster than the process response time.

Finally, on-line programmes may be employed, which include, as well as an optimum trajectory for the controlled variable(s), a range of information about the plant under different conditions. The data for this on-line programme is usually obtained from an off-line programme which includes a process model. It could also include experimental information.

The purpose of the on-line programme would be to control the plant along the predicted optimum trajectory, check its progress, and if necessary, re-calculate a new optimum trajectory from the stored data.

This may be illustrated for a batch distillation unit by storing the predicted profile of the still composition with time, for the calculated optimum reflux ratio trajectory. If the plant differs from this predicted profile, the optimum reflux ratio profile would be re-calculated from the stored data.

3.1 CONTROL ASPECTS PECULIAR TO BATCH DISTILLATION.

As has been mentioned previously, the concept of batch distillation as out-moded is quite popular. However, although many operations which were originally batch are now carried out in continuous columns, this trend has slowed down considerably.

The flexibility of the batch plant is extremely useful, especially for the smaller chemical manufacturer. Not only may a number of products be obtained from a given multi-component mixture, but in addition, a wide range of feed-stocks may be fed to the batch distillation unit, and under a variety of operating conditions, produce innumerable products. The versatility of modern columns is such that distillation units are easily built to cope with a large variety of liquid and vapour rates through the columns.

Lord Rayleigh was the first worker to produce a mathematical treatment of batch distillation (18). Since then, a variety of work has been carried out, including the work of Kropholler (19), who used an analogue computer to solve equations for batch distillation which included the effects of column hold-up. The number of operational elements required is however large, and only a small number of theoretical stages may be treated in this way.

Solutions of the equations using digital computers can be made by one of two methods. The differential equations may be transformed into difference equations, or they may be integrated numerically by one of the many integration techniques available.

The two methods were compared by O'Brien (20), who found that they both gave similar results. However, he also noted that the computation for both were quite lengthy.

The optimisation of a batch distillation unit may satisfy a number of criteria. However, because of the varying conditions within the unit, it is apparent that the computational load of an optimisational analysis would be rather large.

Solutions to this time-optimal problem have been restricted to binary systems and have been based on simple mathematical models of the process. The problem may be solved in one of three ways. These are by the method of dynamic programming, the calculus of variations or by Pontryagins maximum principle.

Mitten and Prabhakar (21) defined the optimum reflux policy as that which supplied a given amount of distillate in the minimum time. They found that savings of the order of 5% were possible, when compared with the more standard techniques of constant reflux ratio or constant top product composition.

Both Coward (22) and Price (23) presented solutions to the optimisation problem based on the maximum principle of Pontryagin, and defined optimal operation as that which gave the required distillate in the shortest possible time. Coward also presented the calculus of variations solution and showed that the two methods gave identical results. In all the cases considered, the saving by operating in an optimal manner was a few per cent.

These previously mentioned optimisation analyses used models based on the McCabe-Thiele assumptions for a no-hold-up situation. A constant boil-up rate was also assumed.

Unfortunately, it has been found that for real columns, where the hold-up must be considered, the problem is somewhat more complex (24, 25). Robinson, infact, investigated optimal operation of an industrial batch distillation unit, and concluded that generally it was imperative that the effect of column hold-up be included in the model of the process (24). His work took the form of both experimental and a theoretical analysis of the process, and as a result, he suggested a sub-optimal operation strategy.

Although considerable work has been carried out on continuous columns (particularly useful is the review by Rosenbrook (26), there is little information about batch columns. However, this is to be expected, since the transient response of continuous columns involves disturbances on a steady-state system, whereas that of a batch unit introduces disturbances on an unsteady-state system.

If one considers a batch distillation unit, conventionally there are two modes of operation. They are to operate the column either at a fixed reflux ratio with a variable distillate composition, or with a fixed distillate composition with a variable reflux ratio. The optimal operation of batch columns has been investigated, and typical of this work is that of Mitten and Prabhakar (21), who sub-divided the distillation task into a number of stages, all at a different fixed reflux ratio.

These authors compared the results of carrying out the batch distillation task according to this technique, and the two conventional techniques. They were able to demonstrate that certain small savings could be made by operating according to the optimal analysis, but their results are naturally somewhat dependent on the process which they analysed.

There are further possibilities available, pulsed columns (27), which are deliberately subjected to pulsating pressures across the distillation column, cyclic operation (28, 29), where the boil-up rate is cycled between the desired rate and zero boil-up, and snatch operation, where the column is cycled between total reflux and the periodic removal of snatches of distillate.

These techniques have all been developed to take advantage of the column dynamics. Although, they all result in improved column performance, difficulty and cost of implementing the column operation according to these techniques must be compared with any saving achieved through increased column throughput or separation.

3.2 CONTROL ASPECTS PECULIAR TO CONTINUOUS DISTILLATION.

The simple first level control of the process variables is similar with both techniques of distillation. However, the object with ideal operation of a continuous unit is to maintain the appropriate steady-state conditions within the column, whereas for a batch column, one must endeavour to maintain the optimum trajectory of the transient conditions within the column.

Since for continuous distillation, the reflux ratio is not time dependent, feed-forward control is based on taking appropriate action to cope with disturbances to the plant as they occur. Control in this manner must be based on a satisfactory process model. The requirements of the model are that it represents the process quite well, and that its response time is several times faster than that of the distillation unit.

The models developed to demonstrate the behaviour of continuous columns are concerned with the change from one steady-state position to another. Because of the absence of initial or final non-steady state behaviour (only transient non-steady state being considered), models of quite considerable sophistication have been developed to represent continuous columns (30).

An extremely useful review of the work on continuous distillation has been carried out by Rosenbrock (26).

Finally, there is a further extension to which models of continuous columns have been applied. That is the determination of the best transition path from one set of operating conditions to another. This may be as a result of a change in feed material to the columns, or because of a change in the desired product proportions from the distillate unit.

3.3 ANALYSIS OF BASIC DIRECT DIGITAL CONTROL.

A comprehensive review of the literature on the use of digital computer in the process control field has been carried out by T.J.

Williams (31). In particular he draws attention to the importance of satisfactory interface equipment (transducers etc). A review of the problem of automatic control of chemical processes was also carried out by Rosenbrock (32), and specifically in the distillation field by Gould (33).

When analysing digital control loops, it is often useful to consider them as consisting of three distinct sections. The measuring unit, which uses a detector to measure the output from the process. The controlling unit, which accepts the signal from the process variable, and then generates an appropriate output according to the control algorithm with which it has been programmed.

Finally, the regulating unit accepts this output, and generates the power to drive the correcting unit. A typical arrangement of the computer and its interface equipment is illustrated in figure (3.1).

When considering a direct digital control installation, two distinct aspects of the scheme must be considered. They are the hardware side, which refers to the equipment, and the software side, which refers to the programmes. Here we are primarily concerned with the software aspect.

Several symposia concerned actually with direct digital control, or with automation in the process industries generally, have been held (34, 35). At these symposia a large proportion of pertinent papers have been presented.

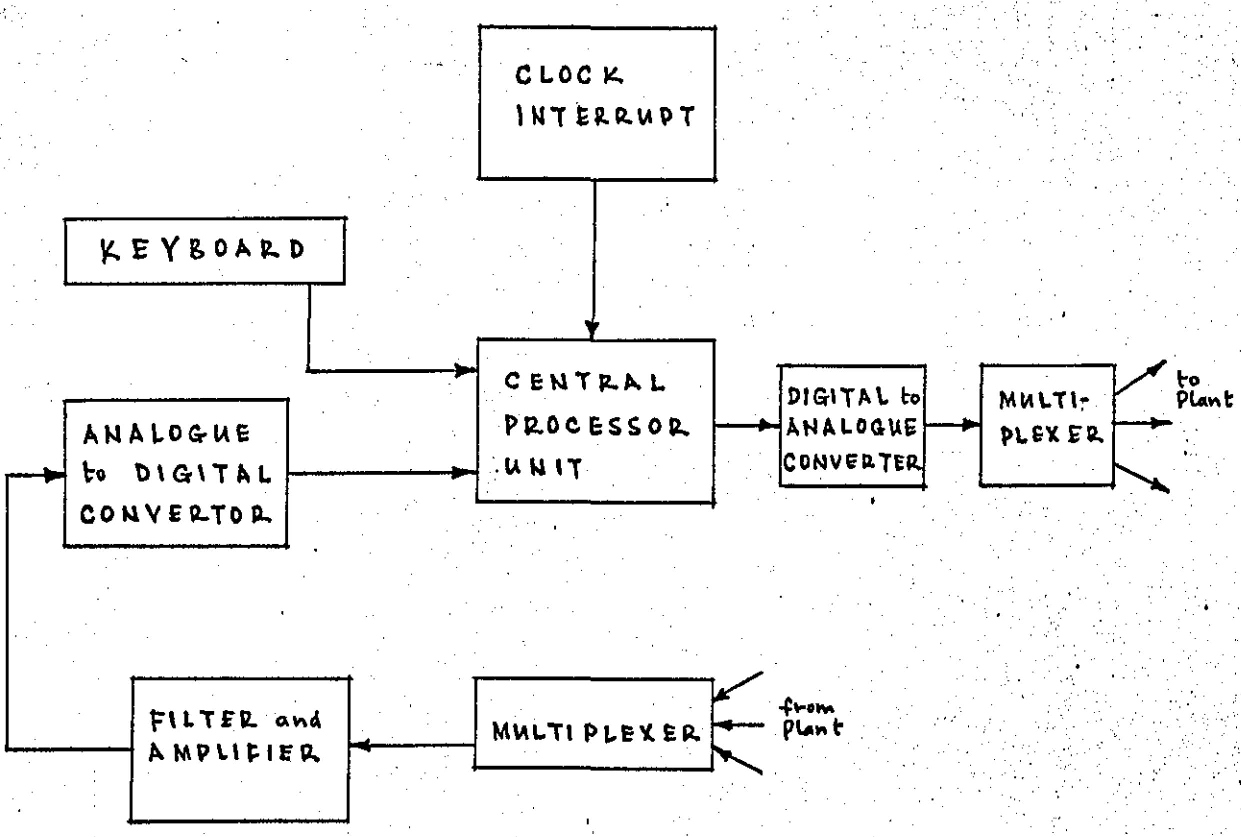

FIGURE 3.1

COMPUTER AND INTERFACE EQUIPMENT FOR DIRECT DIGITAL CONTROL USE

There are two facets of the digital control programmes. The first is the development of the actual control algorithm, and the second is the handling of the sampled data imput.

3.3.1 THE CONTROL ALGORITHM.

Control computer manufacturers have developed a number of control programmes themselves (36, 37), but, in addition, various authors have suggested a variety of algorithms (38, 39).

Digital control algorithms may take the form of the standard conventional 3-term controller, but without the usual inter-action between the various modes. However, the real asset of digital control is that algorithms of considerable sophistication may be developed. These algorithms are usually based on non-linear action of the controller, governed by the magnitude and trajectory of the error signal.

A typical example of this is the algorithm which chooses one of two possible sets of controller constants, depending on the magnitude of the error signal. Other algorithms exist which include previous outputs and error signals, in an endeavour to cope with spurious signals from the plant (40).

A number of authors have also suggested the use of a small section of digital logic as a controller (41).

There has been considerable development of digital control algorithms in cascaded loops, since here the flexibility of the digital controller may be used to its full potential (42).

This new range of controllers have introduced inherent problems in loop tuning, which have been studied by a number of authors (43, 44, 45).

In addition, a number of self-adjusting controllers have been developed (46, 47).

3.3.2 THE DEVELOPMENT OF NEW MEASURED VARIABLES.

A further result from the development of digital controllers is the greater range of possible process variables. Since the digital computer is able to process the process variable signal before it is used in the control algorithm, a number of on-line analysers are used to provide this signal. A survey of on-line analysis has been carried out by British Chemical Engineering (48). The on-line chromatograph is perhaps the most significant of these analysers (49).

A typical illustration of the use of on-line chromatographs in the control of distillation columns is detailed by Scott (50). He points out both the importance, and the difficulties, of choosing a suitable and representative sample.

The output from the chromatograph may be converted into a variety of readings (e.g. ratio of peak heights, or composition), and it may be used for a variety of purposes (e.g. the process variable of a D.D.C. algorithm, or as a possible alarm signal).

A typical arrangement is illustrated in Figure 3.2.

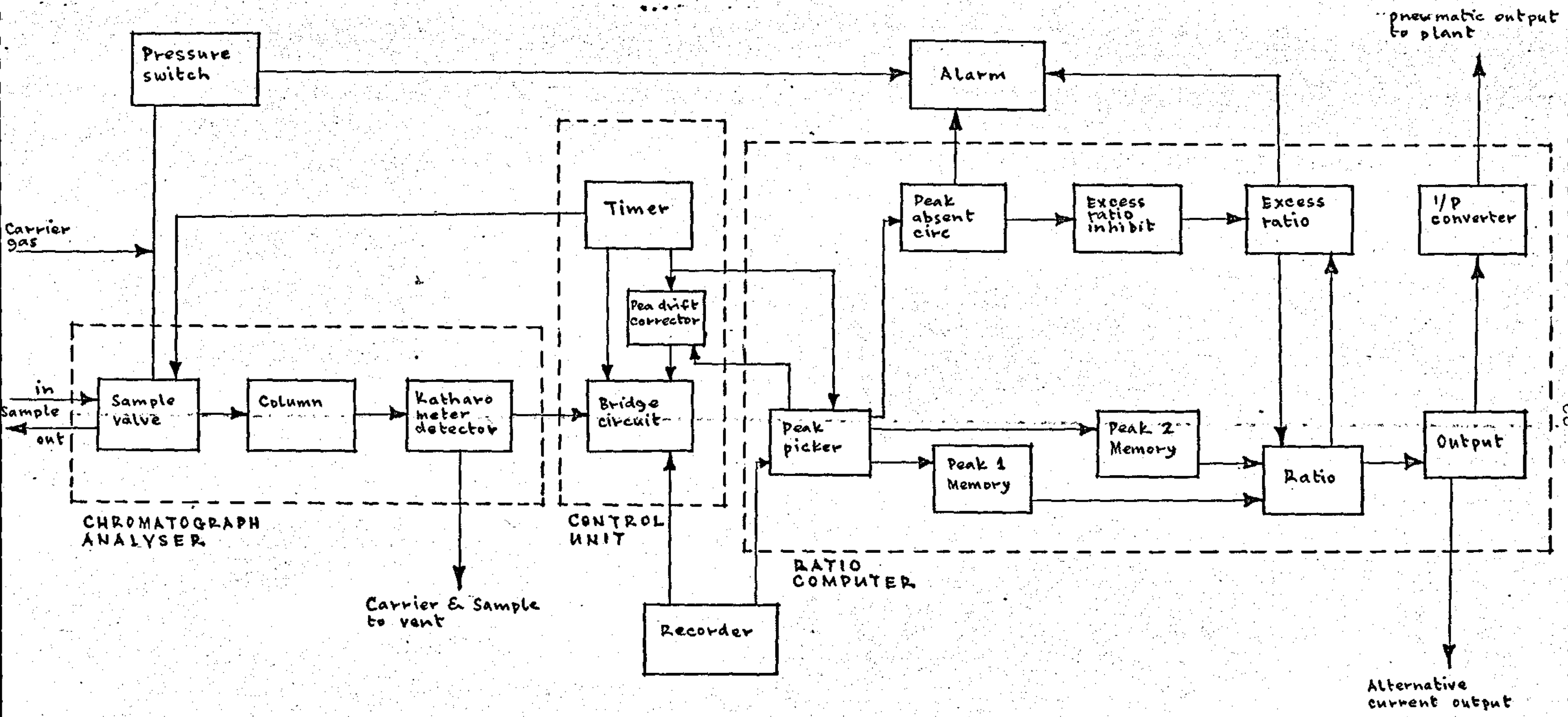

FIGURE 3.2.

OVERALL BLOCK DIAGRAM OF AUTOMATIC CHROMATOGRAPHIC ANALYSER.

3.3.3 CHOICE OF SAMPLING INTERVAL.

A parameter of major importance in a sampled-data control system is the magnitude of the sample interval and various recommendations have been made regarding this (51, 52).

It is obviously desirable to make this as large as possible for efficient use of computer capacity.

Yetter et al (51) examined the step response of loops using both analogue and sampled data control. Their criterion for choice of the sampling frequency was simply that the response of the sampled data loop must be indistinquishable from that of continuously measured loop. Using this criterion, they decided that the minimum adequate sampling rate was 8 samples per loop period, where the loop period was the period of the decay osillations following a set-point change.

They also carried out an examination of a number of controlled loops in order to determine typical recommended sampling rates.

Their results were as follows:-

< 1.25	seconds	40%
< 2.5	seconds	20%
< 5.0	seconds	15%
< 20	seconds	10%
< 80	seconds	10%
80+	seconds	5%

Eckmann et al (52), carried out a similar survey, and produced the following results for recommended sampling rates.

Flow - Loops	0.1 seconds
Pressure - Loops	1.0 seconds
Temperature - Loops	10 seconds
Composition - Loops	60 seconds

As has been mentioned previously, these authors chose a stringent arbitrary standard for the data measurement. If instead of their standard, maximum overshort, decay ratio or $\int E^2 dt$ etc. had been chosen, then larger sampling intervals would have been possible. Infact, a number of authors have used larger sampling intervals, when carrying out practical investigations with digital controllers and real items of chemical plant (46, 53). In addition, the signal/noise ratio is another important parameter in the choice of sampling interval. Finally, a further recommendation for the choice of sampling frequency was made by Lee (54) who suggested that the choice should lie within the range indicated by the equation,

$$1/8f \geqslant \Delta t \geqslant 1/16f \qquad (3.1)$$

where f is the natural frequency of the loop.

3.4 THE APPLICATION OF PROCESS MODELS TO CONTROL STRATEGIES.

The basis of the optimisation programme for distillation units, is the formulation of process models, and an understanding of the dynamic behaviour of the units.

The dynamic behaviour of distillation columns has been a field of particular interest for a number of years.

Comprehensive reviews of the papers on distillation dynamics have been carried out by Archer & Rothfus (55) in 1961 and by Rosenbrock (26) in 1962.

As has been mentioned previously, the interest in the theoretical analysis of unsteady-state distillation is a result of the desire to improve the control and performance of distillation columns. (Endeavours to improve the design of distillation units has also been a key factor).

Plate-type columns are usually described by a set of differential equations representing material balances on the plates, together with heat balances, and equations for the flow of liquid and vapour in the column.

Packed columns, on the other hand, are usually described by partial differential equations for the liquid and vapour streams.

From a theoretical stand point, these mathematical models have several awkward features. The main feature which is an inherent function of the columns, is that local disturbances are rapidly dispersed, whilst disturbances involving the whole of the column necessarily take a long time to decay.

In batch distillation, the target is normally to either predict the reflux strategy required to give a constant overhead product of the appropriate composition, or to ascertain the fixed reflux ratio which will result in the appropriate separation.

The earlier treatments on this subject were generally based on solutions which used simple linear expressions for the vapour-liquid equilibrium data. They generally endeavoured in particular to predict the effect of such parameters as say 'column hold-up' on the separation achieved in the unit. Typical of this work are papers by Rose (56), Bowman (57, 58) and Edgeworth-Johnstone (59).

The use of digital and analogue simulations has meant that the assumption of linear equilibrium relationships may be eliminated.

Rose et al (60) used an analogue computer to complete batch distillation curves assuming constant relative volatility for the vapour-liquid equilibrium data. Jackson & Pigford (61) carried out similar work on a digital computer.

In the latter part of the 1950's, a lot of work on the determination of the dynamic response of continuous columns to disturbances such as load change was carried out. Particular reference was made to the use of these investigations in the design of automatic feedback control system.

Details of digital and analogue simulations of the behaviour and control of both batch and continuous distillation columns are outlined in references (62, 63, 64, 65).

The conclusions to be drawn from the analogue and digital simulations are that with analogue studies either the column must have few plates, or an extremely simplified model must be employed. Alternatively, the digital machines may be used to solve more complex systems, however, these require a large amount of computing time.

Interest in the field of on-line digital control and optimisation, has increased the need for models which are quickly soluble.

Feed-forward control depends on a model which can be solved appreciably quicker than the time taken for the actual process to respond. Models which may be solved quickly can be obtained by approximating the process to a simple transfer function. Work on these lines has been reported by several authors. An example of this is the paper by Jofri et al (66), who used second-order approximation to experimental step response results on a ten-plate column in an analogue simulation to find the best controller settings for two feed-back control schemes.

3.5 CONSIDERATION OF CERTAIN PRACTICAL CONTROL PROBLEMS.

If we turn now to the practical consideration of the control of distillation columns, distillation units are fairly amenable to control, and for this reason, detailed theoretical studies are not essential for their successful operation. Naturally, this reduces the incentive for these detailed studies. However, the running costs of the columns, and the value of the materials they process, are often significant. Hence, there is often room for marginal improvement which result in substantial savings. This is particularly true, where the column has difficulty in reaching its designed performance, or is operated some way away from this level (67).

The control strategies based on steady-state behaviour are much simpler to handle than those based on dynamic behaviour, but they include the implied assumption that the dynamic effects do not lead to control difficulties. For this reason care must be taken to ensure that the columns are operated in a manner such that this is true, and that any difficulties are avoided.

The problem of the control of a distillation unit may be divided into two parts. The first task is to attain the desired separation within the column, and the second is to maintain the desired material balance over the column.

If we consider the first task, Anderson and McMillan (67) illustrated the use of manipulating the reflux and boil-up flow rates to govern top and bottom compositions of a continuous column. This illustrates a rather novel approach, since the inter-action between the loops is quite severe. However, this interaction can be overcome by multivariable control. Infact, the use of multivariable control theory illustrates possibilities for the use of a number of non-conventional control strategies.

Anderson and McMillan also carried out an analysis of the material balance loop. They indicated the usefulness of feed-forward control where feed disturbances may be detected, and the appropriate corrective action taken immediately. They included in their results a successful case study. However, a similar analysis was undertaken by Wardle and Wood (68) and their results were less optimistic. Further steady-states based optimisation analysies were carried out by Van der Grinten and Buys (69) and Rijnsdorp and Maarleveld (70).

The optimisation of an overall distillation task is considered by King (71).

The basis of the optimisation routine is an endeavour to minimise a cost function as the control parameter. This cost function depends on the state of the column, the disturbances applied to the column, and the control variable. Included in the routine is a model of the column, which is linearized about a steady working condition.

The difficulties encountered with this study are firstly the problem of whether the cost function is an accurate representation of cost, whilst the other difficulties amount to the physical problem of finding the optimal path, and then holding it.

3.6 THE PROBLEM OF CHOOSING THE APPROPRIATE PROCESS VARIABLE.

A final problem in the overall control of a distillation unit is the problem of the interaction of the process variables, and the choice of suitable measured variables (72).

For each variable to be controlled, one would like to be able to select one manipulated variable which affected that controlled variable and no other. However, often this cannot be done, and several measured variables are affected by several manipulated variables simultaneously. For example reflux changes and boil-up changes both affect compositions at both ends of the column, and also the pressure, and temperatures in the column are inter-dependent.

Such effects mean that interacting multivariable control systems have to be designed. The practical remedy has usually been to leave some of the variables under manual control.

A further difficulty with theoretical control studies, is that effects which are most important to the behaviour of the column under control, are often effects which are secondary to the main purpose of the column. An example of this is demonstrated by Luyben (73), who illustrated that the way in which the hold-up on a plate depends on vapour flow-rate can have a decisive effect on the control of composition by the manipulation of the boil-up rate.

APPARATUS EMPLOYED IN EXPERIMENTAL WORK AND THE BASIC COMMISSIONING PROCEDURE.

SECTION 4:- APPARATUS EMPLOYED IN EXPERIMENTAL WORK AND THE BASIC COMMISSIONING PROCEDURE.

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APPARATUS EMPLOYED IN EXPERIMENTAL WORK AND THE BASIC COMMISSIONING PROCEDURE

4. INTRODUCTION

The distillation work was carried out in a packed column, whose packing was equivalent to twenty-four theoretical plates. The column was four inches in diameter, and was operated at several boil-up rates all of the order of half a litre per minute.

The boil-up rate and reflux ratio were either governed using Taylor pneumatic controllers, or by using the departmental Ferranti Argus 108 digital computer in conjunction with an appropriate control routine. (Appendices 3, 4 and 5).

In addition, the composition at the top of the column, and in the still, could be monitered continuously by the use of an on-line chromatograph, which analysed samples every two minutes.

4.1 GENERAL DESCRIPTION OF THE BASIC DISTILLATION UNIT

A schematic diagram of the distillation unit is shown in Figure 4.1.

The still was a 50 litre spherical vessel with a steam heated thermosyphon reboiler. The packed section of the column consisted of four 2 ft. long x 4 inch I.D.Q.V.F. glass pipe sections, each of which contained 20 inches of stainless steel 'hyperfil' knit mesh packing. This is high efficiency packing, manufactured by Hydronyl Ltd., and specially designed to minimize gross liquid channelling. It is supplied in lengths equal to its diameter. Hence it was in 20 sections, each 4" in length.

A short tee-piece, of the same diameter as the column,

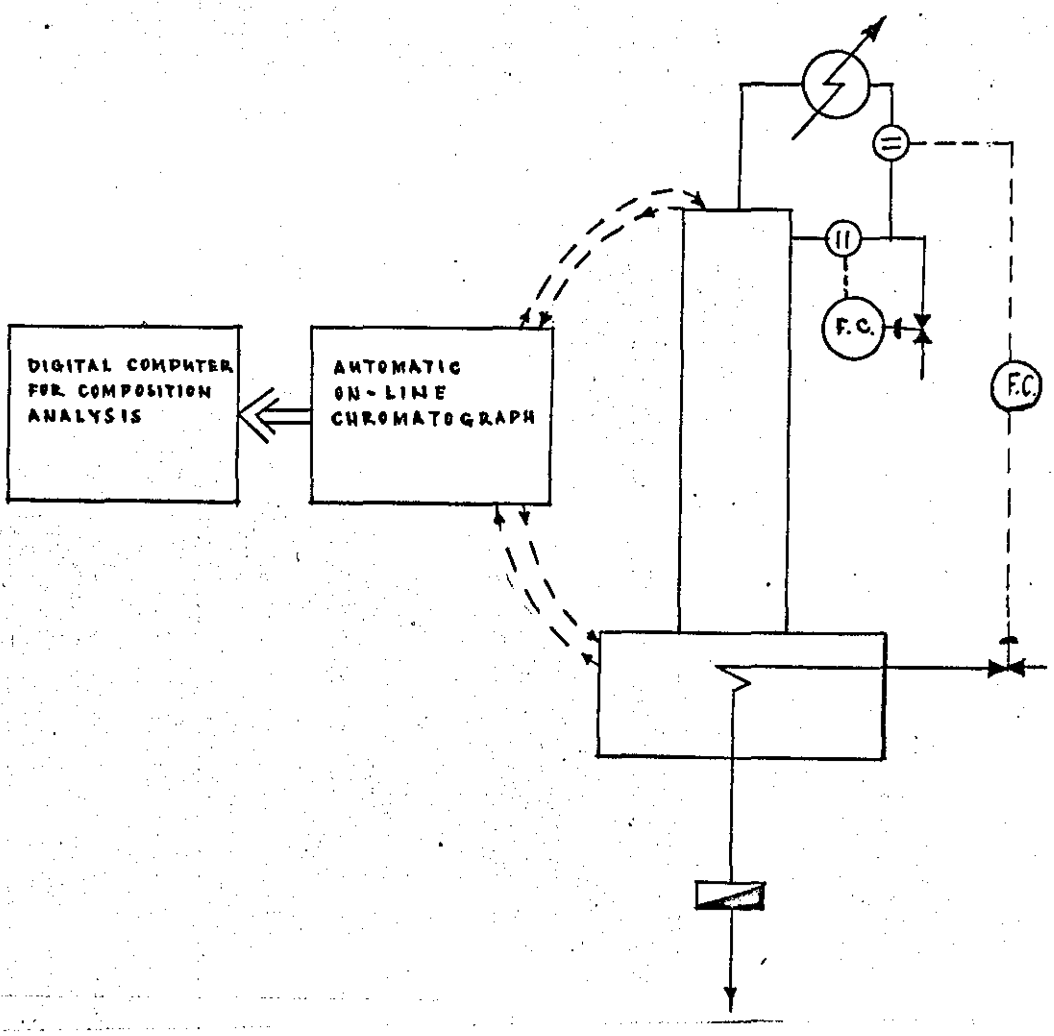


FIGURE 4.1

BLOCK DIAGRAM OF EXPERIMENTAL EQUIPMENT

was positioned between the bottom column section and the still. In addition, a special plate was inserted at the flange between the tee-piece and the still. In this way, a small hold-up of liquid is obtained, and from this samples may be taken (Figure 4.2).

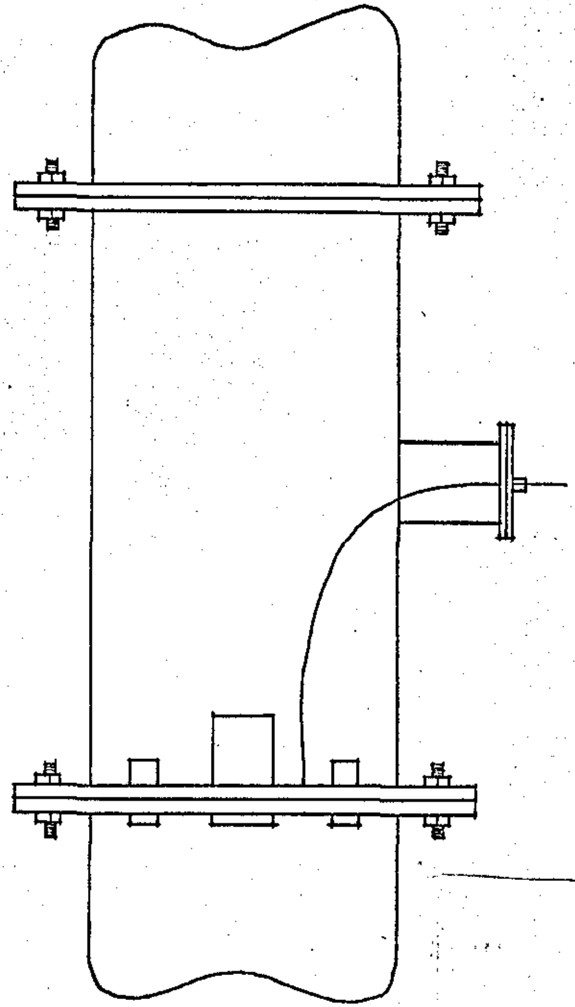


FIGURE 4.2 SAMPLING PLATE

The reflux return to the column was via the arm of another short tee-piece situated above the packed section.

The vapour from the top of the column was fed to the top of a vertical water-cooled condenser, which was vented to atmosphere at the bottom. The vapour leg from the column to the top of the condenser was lagged with a heated coil. This was in order to reduce the amount of vapour which condensed in the line, since it would then run straight back into the column, instead of continuing on to the condenser. (This was important because the condensate rate was considered equal to the vapour rate from the top of the column).

After leaving the condenser, the condensate flowed, under gravity,through a submerged orifice meter. This meter measured the flowrate of liquid from the condenser. The temperature of the condensate was measured here, and the cooling water rate was adjusted manually, in order to keep the temperature constant.

The condensate was then either fed through the reflux return submerged orifice meter and back into the column, or taken off as distillate. In addition, the line leaving the first submerged orifice meter was vented to atmosphere. The proportion of liquid returned to the column was dependent on the setting on the reflux return valve, which was positioned in the distillate off-take line.

The distillate could be collected via a series of steps designed to allow its collection rate to be measured accurately. (A detailed description of this is included in Section 4.7).

Alternatively, to simulate a continuous column, the distillate could be returned to the still. Since the column holdup was small compared with the contents of the still (less than 10%), for a given charge the still composition could be considered to be independent of the column hold-up composition.

In addition, sensing lines were attached between the column and a manometer, such that the pressure drop across the packing might be measured. The high pressure impulse line from the manometer was connected into the vapour space above the liquid in the still, and the low pressure impulse line to the empty column section above the packing.

Finally, liquid samples could be withdrawn from the bottom of the condenser, the plate at the bottom of the packing, or from the still. These samples could then be pumped to the chromatograph for analysis.

4.2 CONTROL LOOPS OVER THE COLUMN.

It may be illustrated, that for a given piece of distillation equipment, two control loops must be fixed to define the operation of the system. Actually, for a two component mixture, seven variables exist, but certain of the variables may be considered as being held constant, and not affecting the process - e.g. the reflux return temperature (74).

Hence, the control of the distillation column may be considered as the control of the following two variables.

1. Boil-Up Rate
2. Reflux Return Rate

The first loop may be considered to control the column capacity, whilst the second loop controls the separation achieved by the column. However, it must be remembered that the two loops are not independent, and a degree of interaction exists between them.

For the initial work, Taylor 3-term pneumatic controllers were used to control these loops. The imput to these controllers must be in the range 3-15 p.s.i.g. For this reason, the signals from the measured variables were first converted to this range.

This was achieved, with respect to the readings from the submerged orifice meters, by using Taylor, fixed-range, Differential Pressure transmitter. These were set up to operate over the range 0-20 inches of water. The pressure drop across the column was also considered as a control variable, and this was converted to an air signal of the appropriate range of the use of a Differential Pressure transmitter with an operating range of 0-20 inches of water.

The outputs from the pneumatic controllers actuated valves in the controlled variable lines. Hence, the reflux return rate was governed by a control valve in the product off-take line, whilst the boil-up rate was governed by a valve in the steam input line to the reboiler.

In addition, since both the pressure drop across the column and the condensate rate from the column, could be used as a measure of the boil-up rate, provision was made to enable these two measured variables to be used in conjunction with one another to form a cascade loop. Since the reflux loop governed the separation achieved in the column, and the chromatograph measured this separation, it is reasonable to consider the readings from the chromatograph as the measured variable for the reflux loop. However, in practice, it was found that the shortest possible sampling period of 2 mins. (governed by the analysis time) was too infrequent to control the loop satisfactorily.

However, it could be used in conjunction with the measurement of the reflux return flowrate to form a cascade loop to control the reflux return rate. This was achieved by including a digital control routine with the analysis programme. The output from this control routine could then be cascaded to the Taylor controller measuring the reflux return rate.

The procedure for controlling the system using digital controllers was similar, but first the signals from the measured variable had to be converted for input to the computer.

This was achieved by feeding the signals from the differential pressure transducers through Taylor pneumatic to electrical transducers, which converted the signal from a range of 3-15 p.s.i.g. to a range of 0-10v.

The controller outputs from the computer installation were in the range 0-10v. These were converted to pneumatic signals of the appropriate range, using Taylor electrical to pneumatic transducers. The outputs from these transducers were then connected to the pneumatically operated control valves which governed the flow of steam to the reboiler, and the reflux return flow-rate.

The digital computer used for the control work was a Ferranti Argus 108, and the analogue to digital/digital to analogue peripheral equipment was considered to be an integral part of the unit.

4.3 THE ANALYSIS TECHNIQUE.

The mass transfer measurements were taken using an on-line chromatograph built in the department. This consisted of an automatic sampling valve, a chromatographic column, and a thermal conductivity detector. These were all mounted inside a thermostically controlled oven. The composition of the liquid in the still and at the column head was pumped continuously through stainless steel tubing, and intermittently injected into the chromatograph. A magnetic valve, actuated by a cam-operated timing device, was mounted on the outside of the oven, where it controlled the air signal to the sampling valve.

The analysis was processed automatically by feeding the output signal from the chromatograph into the computer.

The composition analysis was printed out at the end of each two minute sampling period.

Stainless steel was used in the sampling lines, the sampling valve, and the chromatograph detector, to minimize corrosion.

4.3.1 ARRANGEMENTS FOR SAMPLING FROM THE DISTILLATION UNIT.

The sampling streams were withdrawn continuously from the still and column head and pumped, using micro-metering pumps manufactured by Hughes, through $\frac{1}{8}$" O.D. stainless steel capillary tubing to the sampling valve. They were then returned to points close to the positions from which they had been withdrawn. The use of the capillary tubing kept at a minimum the time lag between taking the samples, and their withdrawal into the chromatograph sensing system.

4.3.2 OPERATION OF THE SAMPLING DEVICE.

The sampling valve has facilities for alternatively taking samples from two different streams. In order to take samples from either of the two streams, a slider in the sampling device was moved between two positions. The movement of the slider was actuated by a compressed air signal, which was controlled by the solenoid valve. The sampling unit is illustrated in figure 4.3. The timing device mentioned above operated a micro-switch in the magnetic valve circuit, and the position of the valve allowed either the points A and B (Figure 4.3) to be subjected to the compressed air signal, or just the point B whilst A was vented to atmosphere.

It can be seen from the illustration, that there are three streams flowing through the sampling device and across the slider. Two are carrying the sampling streams, and the third the carrier gas nitrogen. As the slider alternates between its two positions, it carries a 10μlitre sample from one of the two liquid streams into the path of the nitrogen. The liquid sample is vapourised, and carried through the chromatograph column and detector. It is then vented to atmosphere.

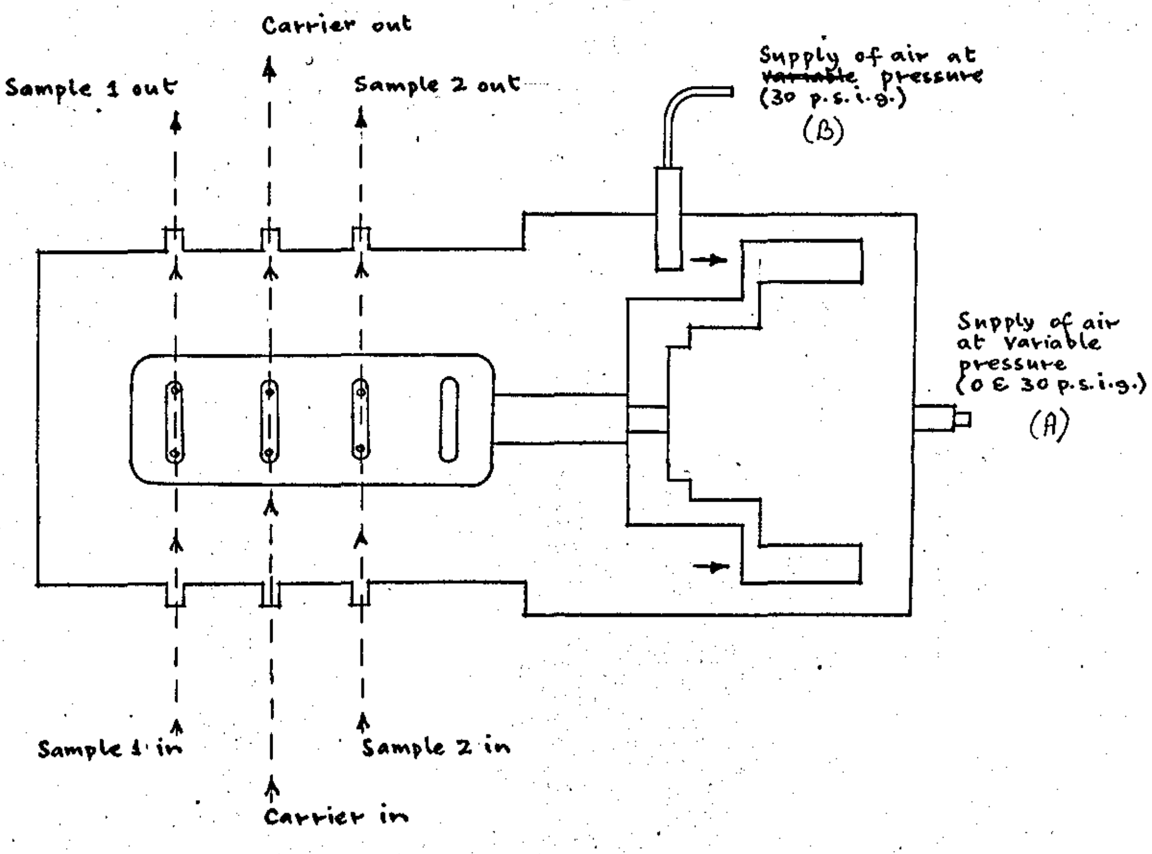


FIGURE 4.3

AUTOMATIC SAMPLING DEVICE USED IN ON-LINE CHROMATOGRAPH

4.3.3 SETTINGS FOR THE CHROMATOGRAPH.

The samples of methyl cyclohexane and toluene that had to be analysed varied widely in composition. Hence, the chromatograph was tuned to settings which were suitable for the analysis of both high and low concentrations of methylcyclohexane. In addition, it was imperative that the two peaks were distinctly resolved, since the analysis programme always allowed the same time for the peaks to emerge, irrespective of the sample composition.

The oven was thermo-statically controlled at 75^{o}c., and the carrier gas flow rate was fixed at 75 ml/min. The current through the thermal conductivity detector was 100 milliamperes, whilst the packing in the column was Polyethylene glycol succinate ester on Chromosorb P (Perkin Elmer DE 113 (P). With this arrangement, the analysis time for each sample was two minutes.

4.3.4 PROCESSING OF THE SIGNAL.

The bridge current power supply and control unit employed in the chromatograph, was manufactured by the Shandon Scientific Company Ltd., London. The output voltage from the detector was fed to a 12" chart recorder with a full scale deflection range of 0-1 mv. Here the resolved components were registered as separate peaks on the chart. In addition, a potentiometer, manufactured by Colvern Ltd. of London, was attached to the recorder. A potential of 10 volts from a stabilised voltage supply was applied to its ends, and it was mounted such that its wiper-arm was rotated by the slide-wire of the recorder. In this way, voltages in the range 0-10 volts, and proportional to the position of the recorder pen, could be fed to the computer installation.

This signal from the chromatograph was processed by the computer, which was programmed to print out the ratio of the peak areas, and the mole fraction of methyl cyclohexane. (Appendix 1 contains details of the programme).

4.3.5 CALIBRATION OF THE CHROMATOGRAPH.

Standard mixtures of toluene and methy cyclohexane were weighed out to an accuracy of the nearest milligramme. These were then analysed on the chromatograph. Repeatability, using the settings indicated previously, was found to be within 0.5% for the mole fraction analysis (Table 4.1)

In addition, the toluene and methyl cyclohexane was tested for purity by analysing them on a laboratory chromatograph fitted with a flame ionisation detector. The methyl cyclohexane used was found to be slightly contaminated with toluene, and the percentage impurity was calculated from the chromatograph assuming that the areas of the peaks were proportional to the number of carbon atoms. A further impurity detected was present in a quantity of the order of 0.1%. This was neglected. The toluene showed no appreciable traces of impurities.

4.4 MEASUREMENT OF LIQUID PHASE HOLD-UP.

The liquid hold-up in the column and auxiliary equipment was measured using a mass balance technique.

The still was charged with a measured quantity of the

Mol. Fraction of M.C.H. in Sample	Area Fraction for M.C.H. peak
4%	0.025
14%	0.1
26.7%	0.2
39%	0.3
51%	0.4
61.75%	0.5
72.0%	0.6
81.5%	0.7
90%	0.8
97%	0.88

TABLE 4.1

CHROMATOGRAPH CALIBRATION CHART

(The Samples were mixtures of Methyl Cyclohexane and Toluene, and were analysed every two minutes).

standard binary mixture Methyl Cyclohexane/Toluene. To this was added a measured quantity of a non-volatile liquid. In this case, di-n-butyl phthalate was chosen.

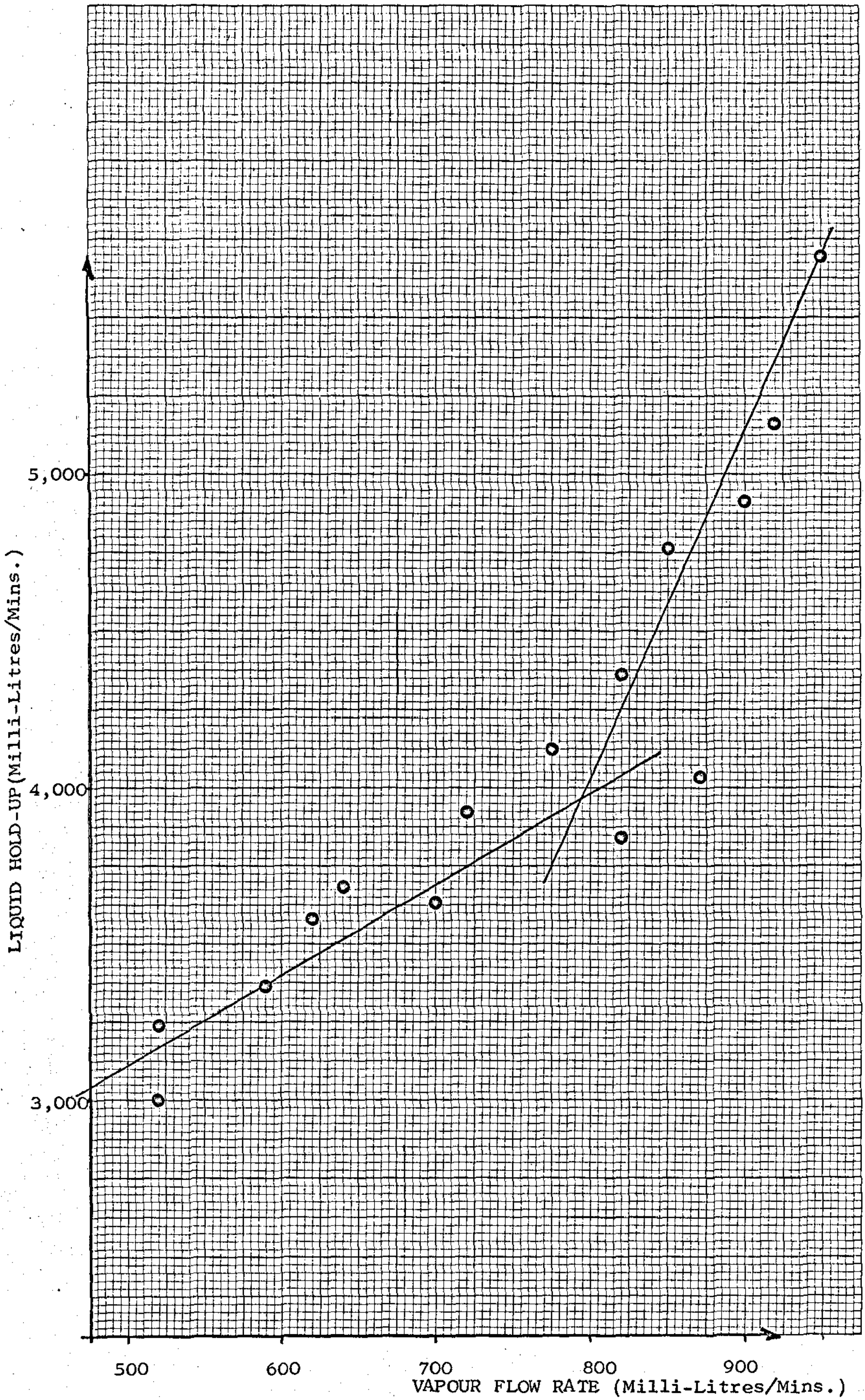
The column was then run under total reflux conditions. It is reasonable to assume that all the non-volatile component remained in the still. Therefore by measuring its new concentration in the still, the volume of liquid which has left the still, and is now in the column, condenser, flowmeter and auxiliary equipment, may be calculated by a simple mass balance.

The hold-up at various different boil-up rates was measured using this technique, and the results are outlined as a graph of boil-up rate versus hold-up. (Graph 4.1 'a').

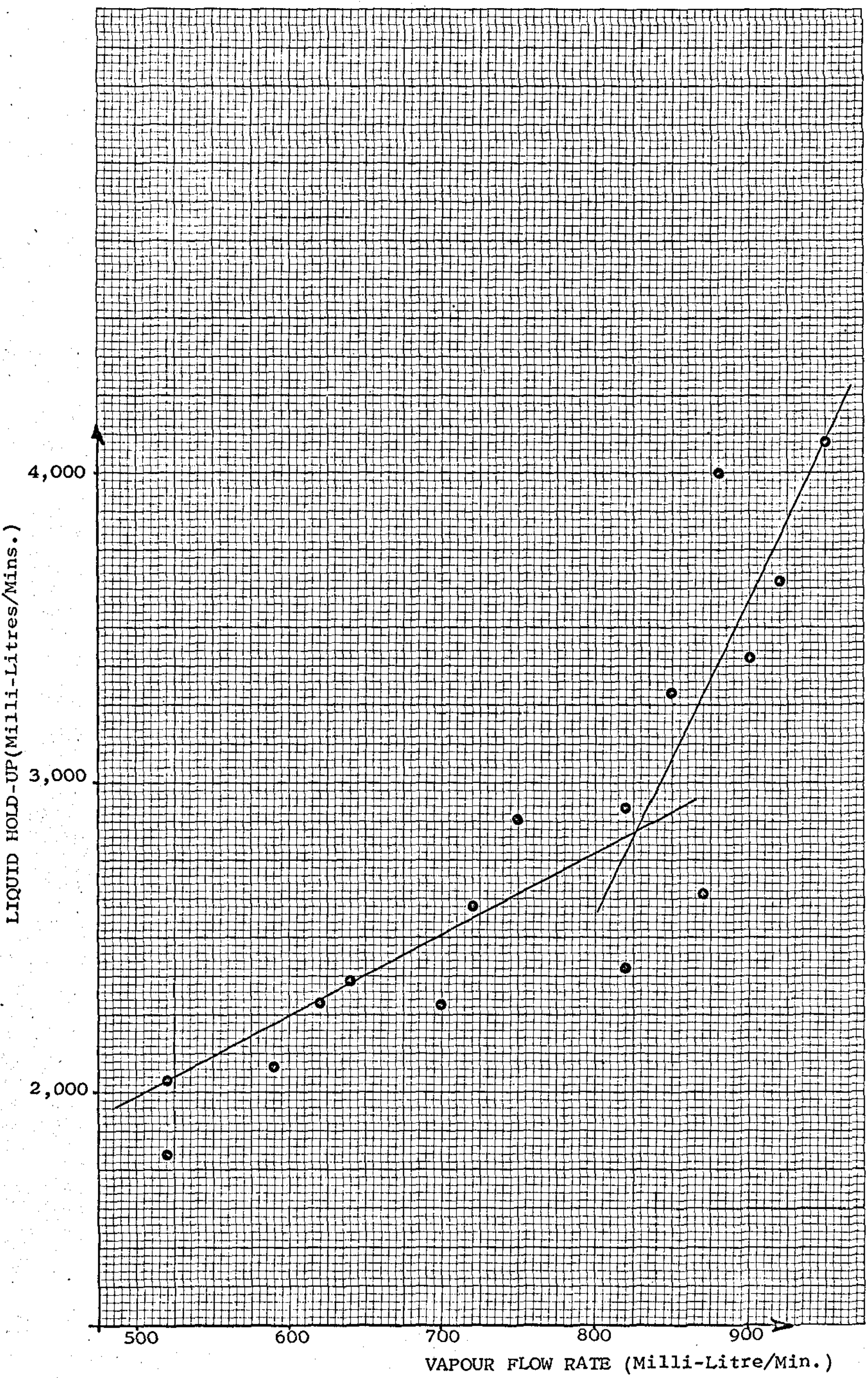
In addition, the hold-up in the packing may be assessed by subtracting from the total hold-up, the contents of the two flowmeters. (The hold-up in the condenser is small enough to be neglected). Using this technique a graph of actual column hold-up for a variety of boil-up rates may be plotted. (Graph 4.1 'b').

The method employed to measure the concentration in the mixture of the non-volatile liquid, was to evaporate off at 70° C. the low boiling mixture of Methyl Cyclohexane and Toluene from a weighed sample, and then to weigh the residue.

By reference to Graph 4.1 'b', it can be seen that the points may be represented by two straight lines, and that the break point is at a boil-up rate of 825 mls/min. This corresponds



GRAPH 4.1(a) TOTAL LIQUID HOLD-UP IN THE EQUIPMENT VERSUS BOIL-UP RATE



GRAPH4.1(b) LIQUID HOLD-UP IN THE COLUMN EXCLUDING AUXILIARY EQUIPMENT

well with the loading point, as found from the pressure drop measurements.

This is in agreement with previous investigators (75), who have found that liquid phase hold-up in packed columns is a linear function of the liquid rate up to the loading point, but that above the loading point, the relationship is different since the gas rate becomes an important variable.

4.5 CALIBRATION OF FLOW-METERS.

The flowrates of the condensate and the reflux return were measured using submerged orifice meters. Initially, a constant head unit was employed in order to obtain a constant flow-rate through the meters. They were then calibrated by measuring these flow-rates for a number of different pressure heads.

The equipment was arranged as indicated in Figure (4.4). The column was run at total reflux with the condensate temperature adjusted to 40^{o}c. Initially the column was controlled at a low boil-up rate. Once the boil-up rate was reasonably constant, the controller on the boil-up loop was switched from automatic control to manual. With the level in the flowmeter held constant, by the use of the constant head apparatus, the output from the condensate flow-meter was collected over a timed interval. Several samples were collected in this way, and the average collection rate determined.

With the system on total reflux, the heights in the two flow-meters were recorded. The boil-up rate was then increased, and the procedure repeated until the flow-meters had been

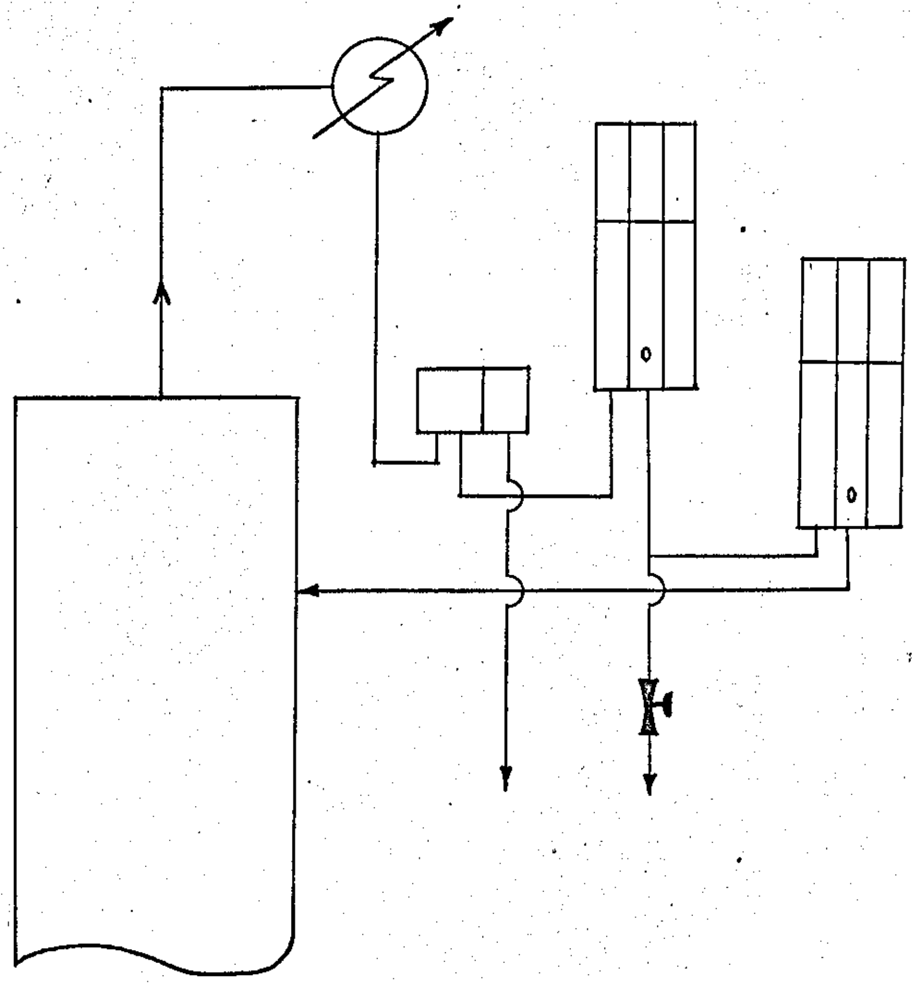


FIGURE 4.4

BLOCK DIAGRAM OF ARRANGEMENTS FOR FLOWMETER CALIBRATION calibrated over their entire operating range.

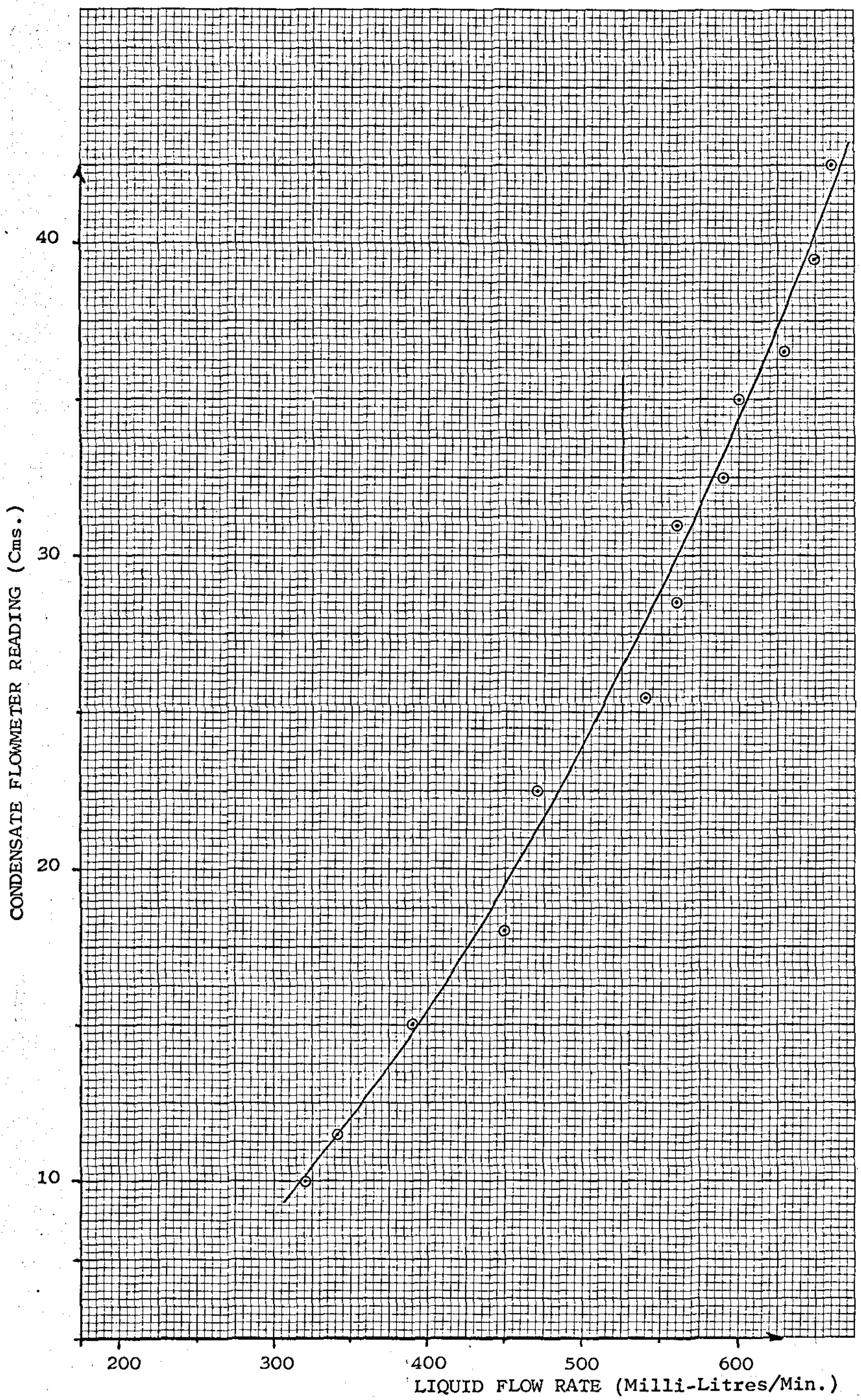
The results have been plotted on graphs (4.2 'a') and (4.2 'b'). It should be noted however, that the actual vapour rate up the column is greater than the condensate rate. This is because some of the vapour is condensed by the return of sub-cooled reflux.

It was found that fluctuations in the flowrates led to experimental scatter of the results. These errors were particularly severe with the reflux flow-meter. In addition, the reflux flow-meter was calibrated using different reflux ratios, but any changes in the calibration curve were not detectable.

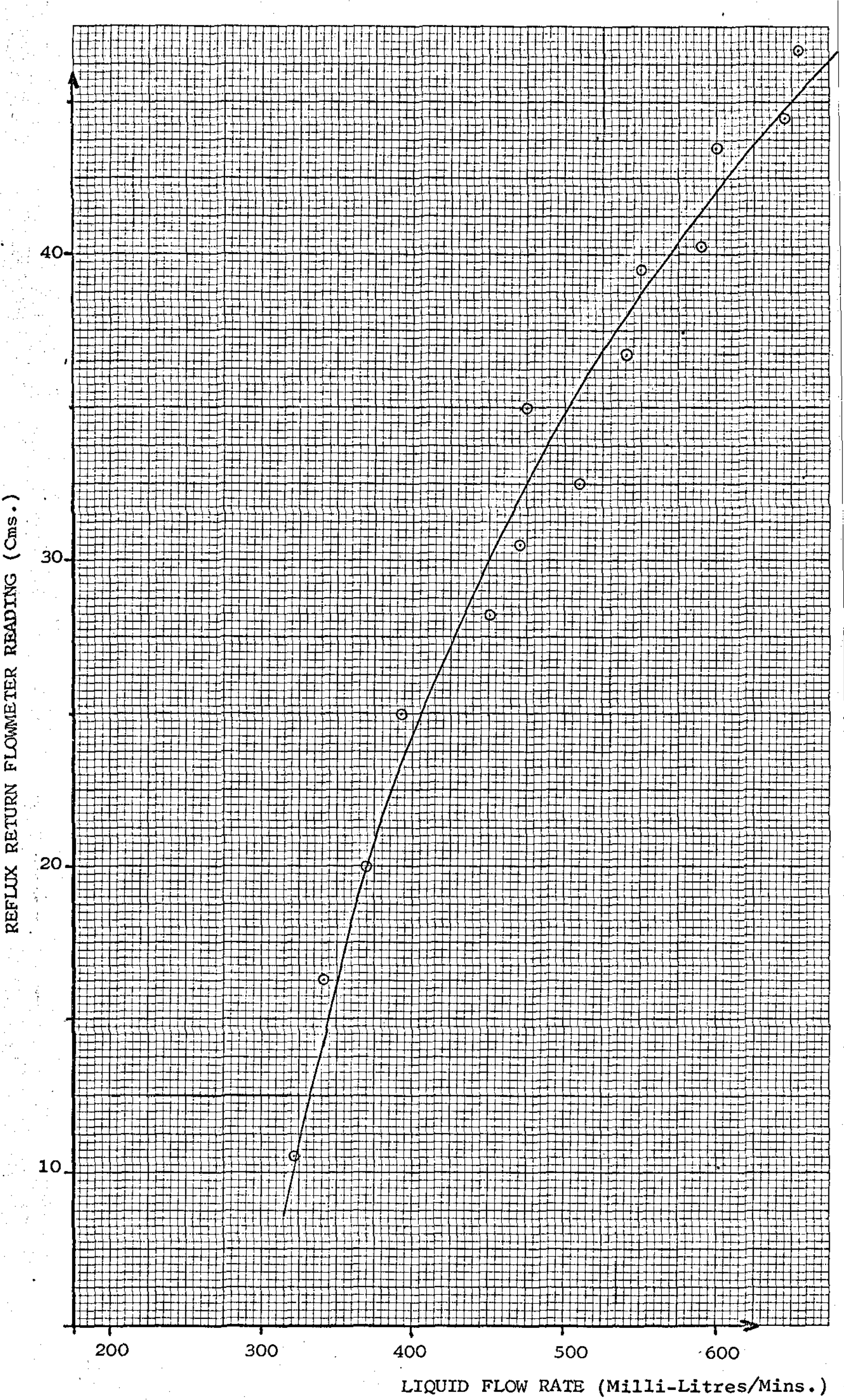
4.6 MEASUREMENT OF PRESSURE DROP ACROSS THE COLUMN PACKING.

A water manometer was used to measure the pressure-drop across the column packing. The high-pressure impulse line was connected into the vapour space above the liquid in the still. The low pressure impulse line was connected to the empty column section above the packing.

Using this configuration, the pressure-drop across the column for a variety of different boil-up rates was measured. During the investigation, it was found that the pressure-drop signal was rather noisy. Infact, the scattering of the results was due in part to the reason that it was impossible to read the signal to better accuracy than ± $\frac{1}{2}$ cm of water. A variety of mechanisms were employed in an endeavour to dampen these signals, but none worked satisfactorily.



GRAPH 4.2(a) CALIBRATION CHART OF CONDENSATE FLOWMETER



GRAPH 4.2(b) CALIBRATION CHART OF REFLUX FLOWMETER

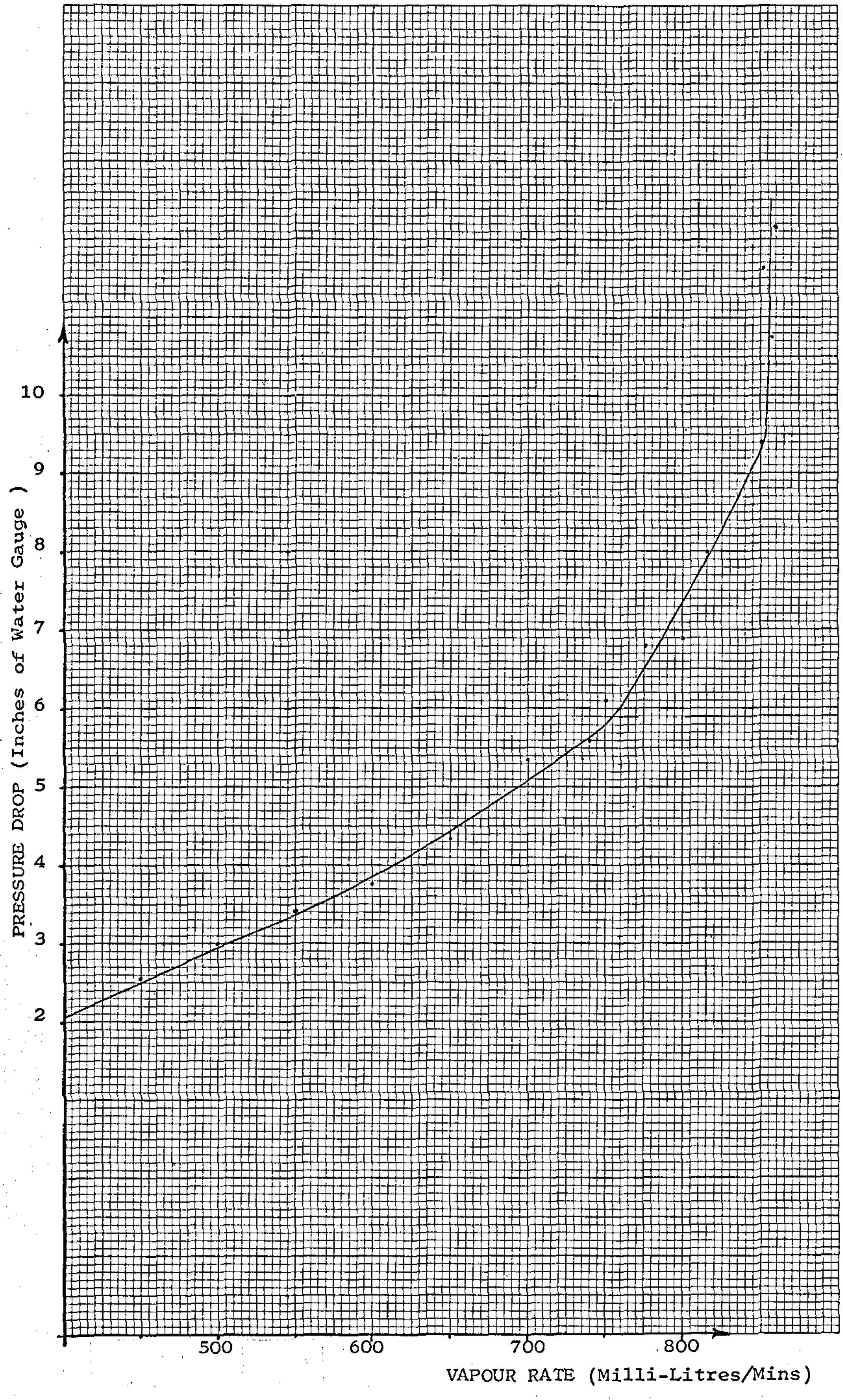
The pressure drop versus boil-up rate curve, at total reflux and using the M.C.H/Toluene mixture, is shown in (Graph 4.3). It can be seen that two breaks in the curve are distinguishable. These are the loading point and the flooding point.

A further analysis was carried out using different reflux ratios. However, the influence of reflux ratio on the pressure-drop across the column, at a given boil-up rate, was found to be small for the range of reflux ratios examined. (These were between total reflux, and the ratio of $^L/D = 1$).

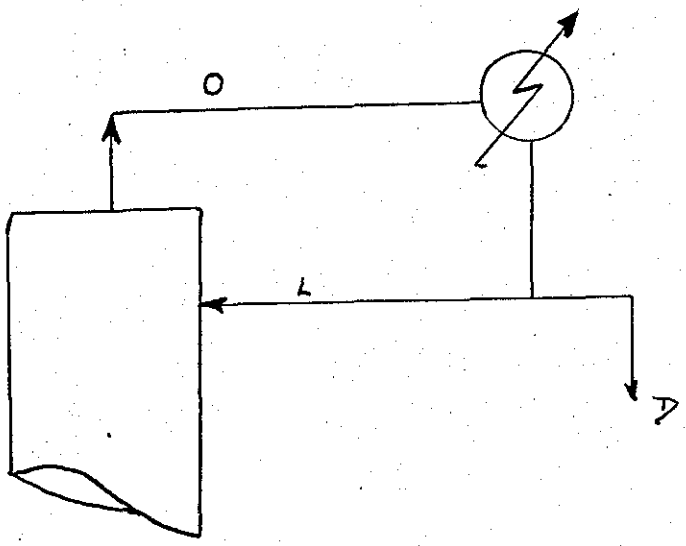
4.7 MEASUREMENT OF DISTILLATE COLLECTION RATE.

Initially, an endeavour was made to measure the product collection rate by merely differencing the apparent boil-up rate and the reflux return rate. However, this was found to be unsatisfactory, because the signals from the flow meters measuring these two variables were subject to fluctuations because of the inherent presence of surging within the equipment. (This surging is illustrated by reference to the analysis of the measurement of pressure drop across the column).

As a result of the properties of these two measuring loops, it was only possible to control and measure the two flow rates to within ± 3%. As a consequence, if the only control action taken was an endeavour to maintain these two flow rates at the requisite values, then measurement of distillate flow rate would be subject to significant errors. This may be illustrated by considering typical flow rates through the system.



GRAPH 4.3 PROFILE OF PRESSURE-DROP WITH VAPOUR RATE CHANGES UP THE COLUMN



D = O - L (4.1)

$= 600\ cc/_m - 550\ cc/_m$ (4.1 'a')

$= 50$

The accuracy to which O and L may be measured is ± 3%

hence,

by inserting the experimental errors into equation (4.1), we get,

$D = O - L$

$= (600 \pm 18) - (550 \pm 17)$

i.e. the values which D might have are

$15\ cc/_{min.} \rightarrow 85\ cc/_{min.}$

hence the distillate rate may be specified as D ± 70%.

It should be noted however, that these two variables do not fluctuate independently.

As a result, the problem is not quite as large as it might at first appear.

In order to overcome this problem, a system must be designed which will measure the low distillate flow rates accurately. A variety of schemes including submerged orifice meters and turbometers were considered. Finally, it was decided that the most convenient practical solution was a form of level tank device.

It should be noted however, that the reflux ratio was measured by an analysis of the mean values of the flow rates of the two main control variables - apparent boil-up rate and reflux return rate.

The equipment employed in the level tank device is illustrated in Figure 4.5, and the principle governing its operation is as follows.

The distillate was allowed to collect for two minutes in a cylindrical vessel, and then flow into the vessel was stopped. Ten seconds were allowed for the level in the cylinder to settle and the height of liquid in the cylinder then measured using a differential pressure transducer. This height measurement was converted into a voltage and transmitted to the computer. Thereupon, the cylinder was emptied, and the procedure started once more. The computer processed the signal by means of a calibration curve held in its store, and from this obtained the

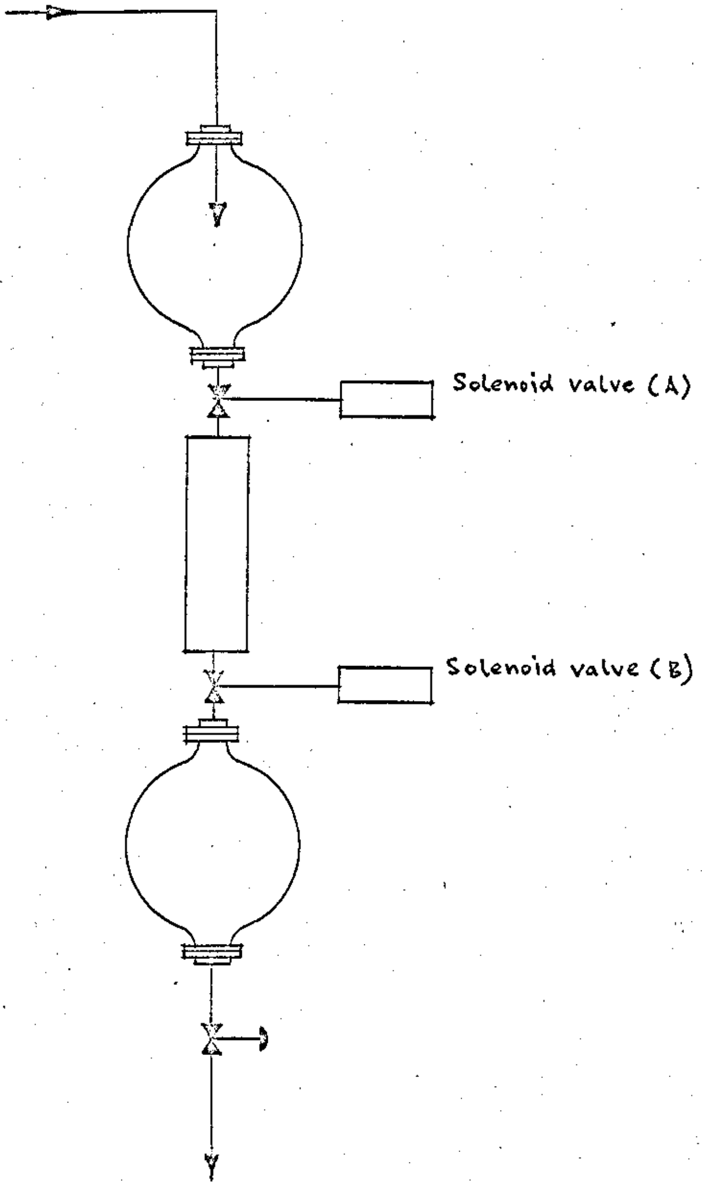


FIGURE 4.5

EQUIPMENT FOR DISTILLATE COLLECTION MEASUREMENT corresponding distillate flow rate.

In addition, a subroutine was included in the programme in order to convert the flow rate signal to moles of product distilled from the system. This was achieved in conjunction with use of the on-line chromatograph.

The practical arrangements for the distillate collection, and the requisite programming subroutines are outlined in Appendix 2.

Hence, using this arrangement, the computer is able to assess and control the mean value of the reflux ratio, and to measure the total quantity of distillate removed to date, whilst it is also measuring the composition profiles during the experimental run.

A typical programme printout is as indicated in Figure 4.6.

4.8 CALIBRATION OF AIR/ELECTRICAL AND ELECTRICAL/AIR TRANSDUCERS.

The transducers were linear over their whole operating range, and were designed to receive or transmit signals in the ranges 0 → 10 V. and 3 → 15 p.s.i.g. However, usually the plant signal with which they were associated, gave non-linear characteristics. (Typical of this, was the flowrate to voltage conversion, where the flowmeters were non-linear).

4.8.1 AIR-ELECTRIC TRANSDUCERS.

These were on the measured process variable lines, and

```
222.910                 046.400

000.8277                000.8989


000.67                  016.75

013.8


098.135                 248.286

000.2833                000.3231


000.71                  017.46

013.4
```

Area of Peak 1	Area of Peak 2
Area Fraction of Peak 1	Mol. Fract of Component 1
Mols. of distillate removed in 2 minute interval.	Total distillate removed to date.
Mean Value of Reflux Ratio in 2 minute interval.	

FIGURE 4.6

TYPICAL ANALYSIS PROGRAMME PRINT-OUT DURING A BATCH DISTILLATION RUN.

were calibrated by first maintaining a constant input signal to the transducers. (This was achieved with the flowmeters by the use of constant head apparatus). The resultant voltage signal at the computer installation was read, and the procedure repeated over the whole experimental operating range.

4.8.2 ELECTRIC-AIR TRANSDUCERS.

These transducers were calibrated over their experimental operating range by sending out to them fixed voltage signals from the computer installation. The transducers actuated the control valves on the steam input lines and the product off-take line.

For each voltage signal, the corresponding boil-up rate or ratio between reflux return and distillate collection rate was determined.

ANALYSIS OF THE FEED-BACK CONTROL LOOPS

SECTION 5: ANALYSIS OF THE FEED-BACK CONTROL LOOPS.

CONTENTS

ANALYSIS OF THE FEED-BACK CONTROL LOOPS.

5. INTRODUCTION.

In this Chapter, the control of the individual feedback loops has been considered. Both analogue and digital controllers have been employed, and the performances of a number of control schemes were investigated.

In addition, the application of certain digital cascade control schemes has been investigated. As a result of the analysis, digital cascade controllers which led to improved column performance were devised.

5.1 FEEDBACK CONTROL ALGORITHMS.

5.1.1 PNEUMATIC CONTROLLERS.

Both boil-up and reflux return loops were controlled using Taylor pneumatic controllers. These controllers nominally have a control algorithm described by the equation

$$P = K_c \left(1 + T_s + U/s\right) \quad E \qquad (5.1)$$

The actual pneumatic controller characteristics may be approximated by equation (5.2), which was supplied by the manufacturer (76)

$$P = K \left(\frac{1 + UT}{1 - UT}\right) \left(1 + \frac{T}{1 + UT} S + \frac{U}{1 + UT} \cdot \frac{1}{S}\right) \qquad (5.2)$$

The recommended procedure for setting these controllers is to obtain a step response with a 4:1 decay ratio. For the digital controllers the integral of the error squared. The derivative action of the pneumatic controller was found to be unnecessary for satisfactory control, and was therefore not included in the digital control experiments.

5.1.2 DIGITAL CONTROLLERS.

Three algorithms were tested, the first two, the position and velocity algorithm were suggested by T.J. Williams (39) and the third, which made use of a truncated logarithmic quantisation was proposed by Peatman and Mergler (77).

The position algorithm represents a three-term controller analogous to that given by equation (5.1) and is given by equation (5.3) in finite difference form.

$$P_n = K_p e_n + K_D \left(\frac{e_n - e_{n-3} + 3e_{n-1} - 3e_{n-2}}{6\ t} \right)$$

$$+ \ K_I \sum_{t=o}^{t=n} e_t \Delta t + P_o \qquad (5.3)$$

In the application of this control algorithm to the plant, the derivative constant, K_D, was set to zero. In this case the control algorithm is equivalent to that described in reference (30). The programme developed to control the plant according to this algorithm is explained in detail in Appendix 3.

The velocity algorithm is given by

$$V_n = P_n - P_{n-1} \qquad (5.4)$$

$$\text{i.e. } V_n = K_p \left(e_n - e_{n-1} \right)$$

$$+ \ \frac{K_D}{6\Delta t} \left(e_n + 2e_{n-1} - 6e_{n-2} + 2e_{n-3} + e_{n-4} \right) + K_I \ e_n \Delta t \qquad (5.5)$$

Williams (39) stressed the fact that in using equation (5.5) it was always necessary to include some integral action, i.e. $K_I > 0$, as this is the only term which includes a measure of the set-point for the controller.

When used on the distillation unit, the velocity algorithm was also used with K_D set to zero.

The velocity algorithm was further developed to include the use of non-linear action to improve the performance of the controller. At any interval, the present error signal was compared with the previous error signal from the plant, and the appropriate action taken according to the trajectory of the measured process variable.

The programme developed to control the plant according to the simple velocity algorithm is explained in Appendix 4, and for the non-linear algorithm in Appendix 5.

It can be seen from equations (5.3) and (5.5), that the integral and derivative terms are simply expressed by making the sample interval (Δt) equivalent to the 'dt' in the standard continuous sampling algorithm. In addition, the controller constants are all independent of the sample size, and hence, the controllers must be tuned to a given sampling frequency.

The final digital control algorithm which was examined, was specifically developed such that it could be employed by the use of a simple piece of digital logic. The basis of the algorithm is that the truncated logorithmic value of the process error is employed when formulating the controller output. The equation for the controller is given by:-

$$\Delta M_n = K_E^* \; E_n^* + K_{\Delta E}^* \left(E_n^* - E_{n-1}^* \right) \qquad (5.6)$$

* denotes use of the truncated logarithm.

The operations implied by equation (5.6) are carried out in such a fashion that core store requirements are reduced and multiplication is simplified. The operations may be formally defined by the expression.

$$\langle \mathrm{LOG}\ E_n \rangle = \begin{cases} 0 & E_n = 0 \\ (\mathrm{SIGN}\ E_n) \langle 1 + \mathrm{LOG}_2 |E_n| \rangle & E_n \neq 0 \end{cases} \qquad (5.7)$$

The brackets $\langle\ \rangle$ are used to indicate that the error signal is truncated at the decimal point e.g. 2.96 is represented by 2. Therefore, in binary form, $1 + \mathrm{LOG}_2\ |E_n|$, is a number equal to the position of the most significant digit. The simple logical operations required for the controller are illustrated in Figure (5.1).

It can be seen from experimental results (Graph 5.4), that adequate control was possible using this algorithm. In addition, it should be noted, that as one might expect, the tuned constants of the truncated controller were greater than those of the standard algorithms by a factor of approximately $\sqrt{2}$.

5.2 OUTLINE OF EXPERIMENTAL PROCEDURE.

Control of the reflux return rate was considered first, and then control of the boil-up rate.

The first loop may be considered as determining the separation achieved by the column, whilst the second loop controls the column capacity. However, it must be realised that there is inter-action between the two loops, and they are not independent.

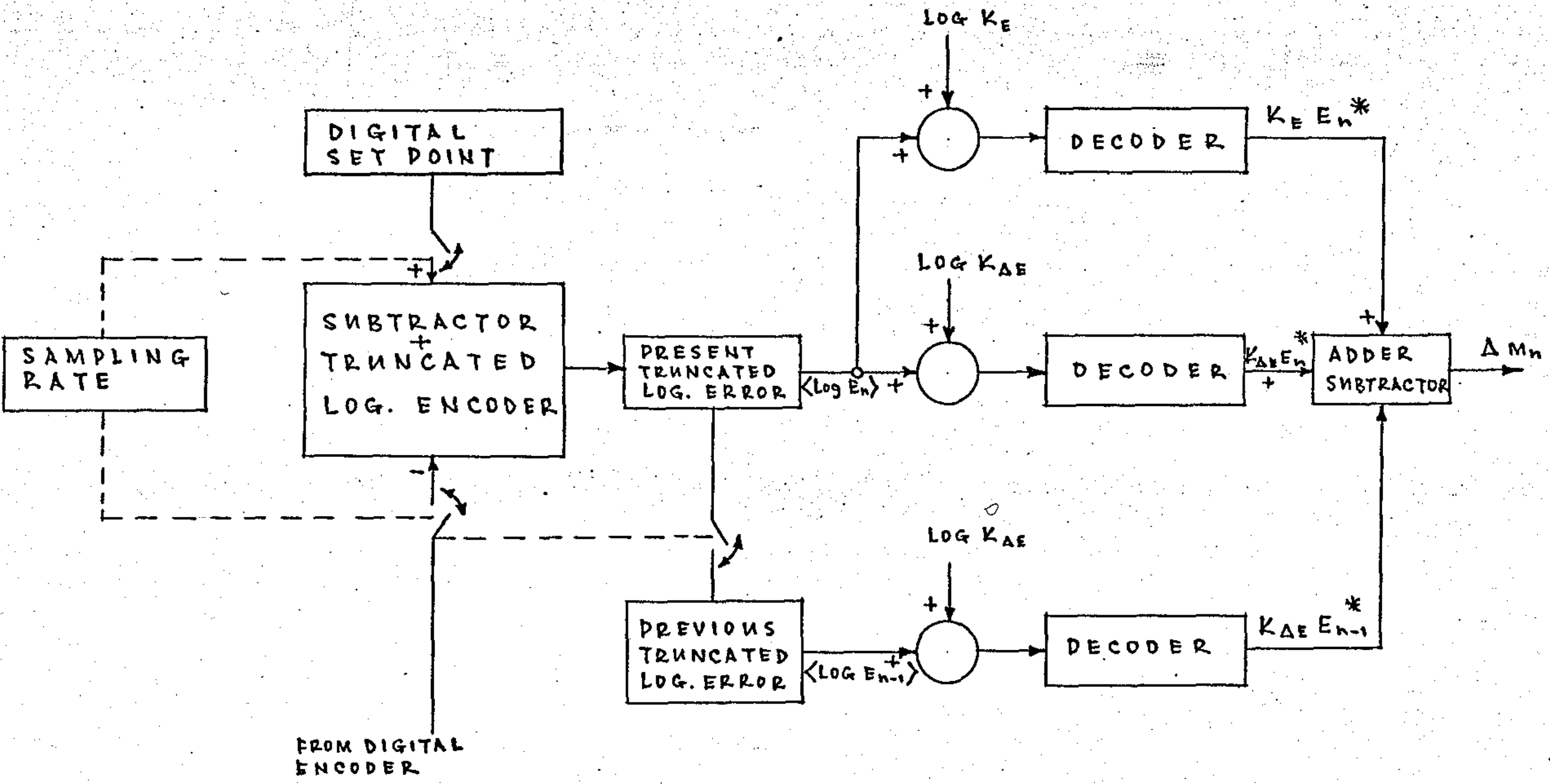


FIGURE 5.1 LOGIC FOR TRUNCATED LOGARITHMIC CONTROLLER

The control loops which are examined in detail for the purpose of comparing the various controllers are as outlined in Figures (5.2 'a') and (5.2 'b').

The digital controllers may be used to control each loop separately, but in addition they may be combined such that the control of the unit becomes an integrated problem.

The mixture used throughout the experimental examination was Methyl Cyclohexane/Toluene, and it was decided to fix the initial still composition at 30% M.C.H. throughout the experimental work.

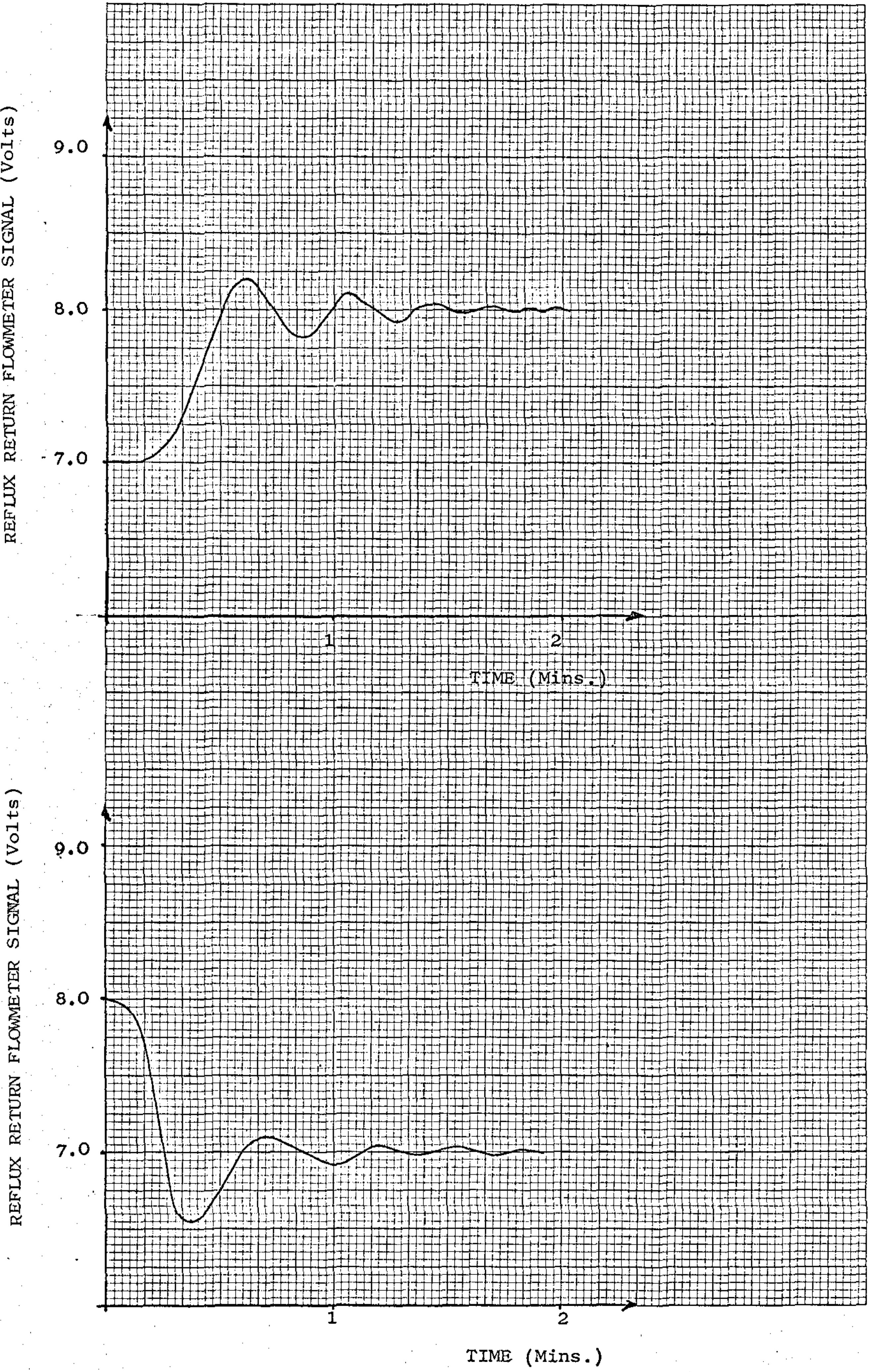
5.3 EXAMINATION OF THE ANALOGUE CONTROLLER SYSTEMS.

Using the pneumatic analogue controllers, the control loops were tuned approximately using the procedure recommended by the manufacturer. The loops were then further adjusted by using the simplex technique as described in Appendix 6. The value of $\int e^2 dt$ was taken as the performance index throughout.

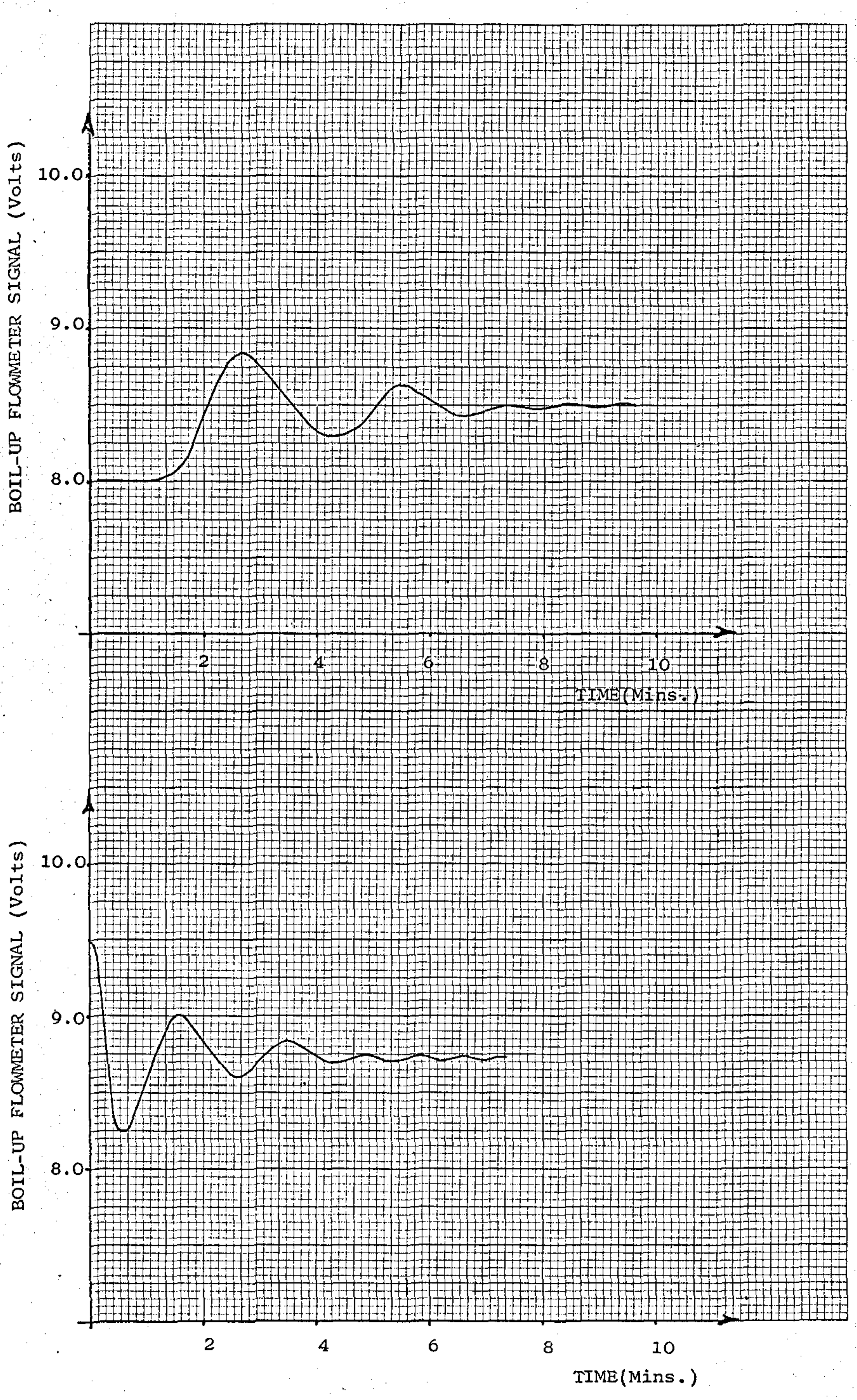
The response of the reflux flow loop is shown in Graph (5.1) and that of the boil-up loop, using the condensate flow-rate as the measured variable, is as shown in Graph (5.2).

An endeavour was made to use the raw pressure drop signal as the measured variable for the boil-up loop.

However, it was found that the signal noise ratio was such that satisfactory control was not possible. In addition, the



GRAPH 5.1 REFLUX FLOW LOOP RESPONSE WHEN USING AN ANALOGUE CONTROLLER



GRAPH 5.2 RESPONSE OF BOIL-UP LOOP WHEN USING ANALOGUE CONTROLLER

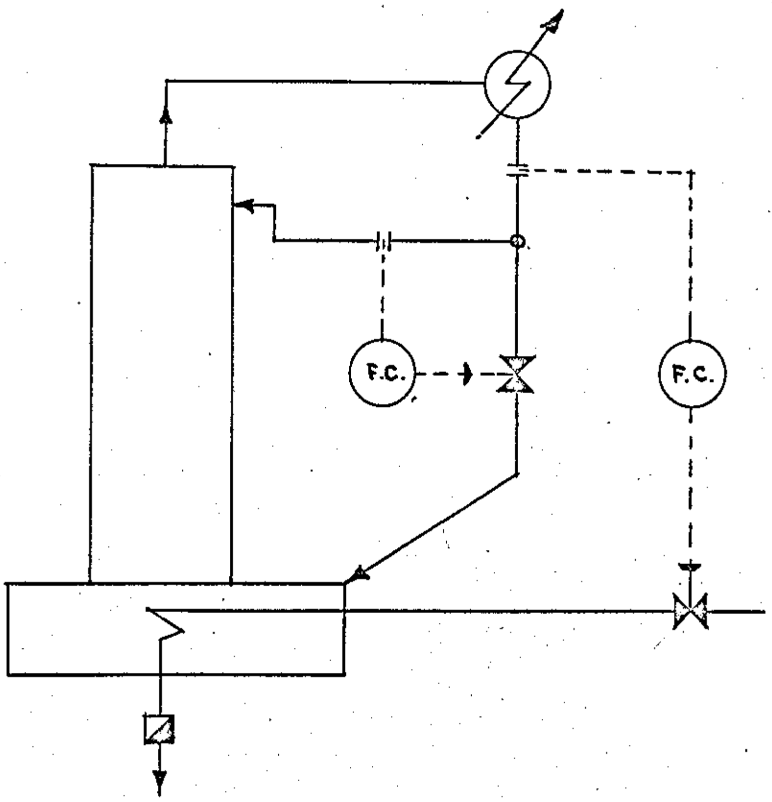


FIGURE 5.2'a'

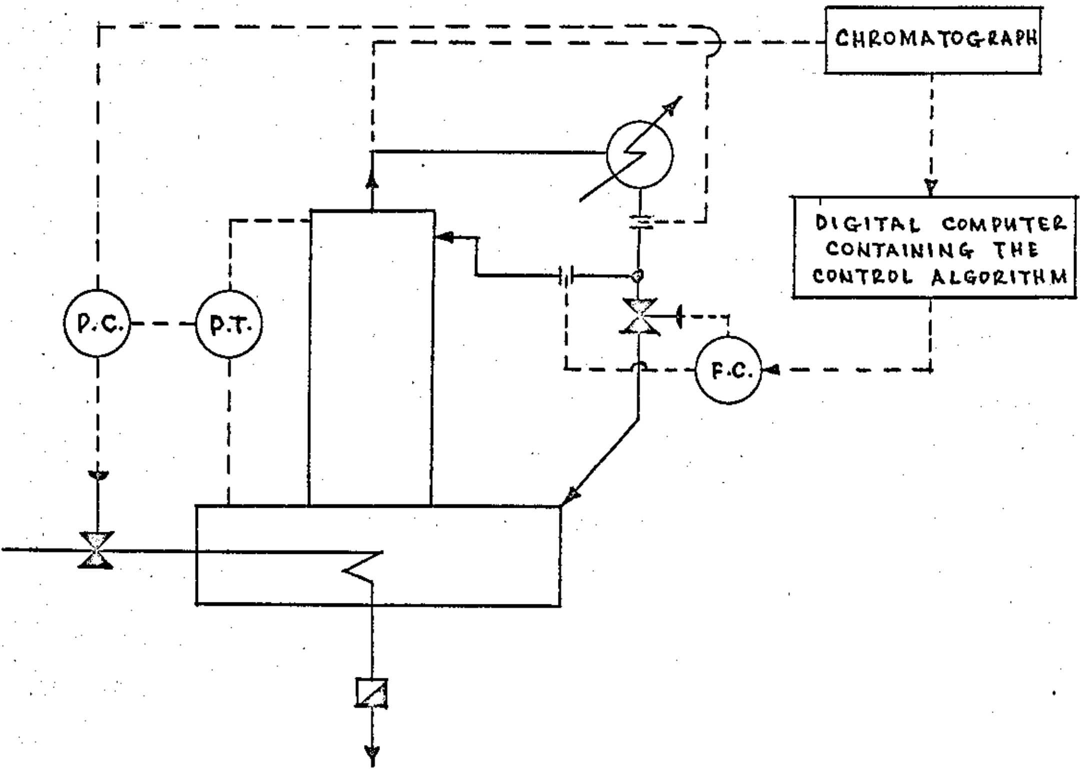


FIGURE 5.2'b'

FIGURE 5.2

OUTLINE OF CONTROL SCHEMES EXAMINED DURING THE PRACTICAL INVESTIGATIONS non-linear behaviour of the loop was such that temporary instability usually resulted from a disturbance of any magnitude.

The response of the system, when controlled by an analogue controller and using the pressure drop as its measured variable, is outlined in (Graph 5.3).

Because the pressure signal was extremely noisy, the readings have been smoothed before plotting them on the graph.

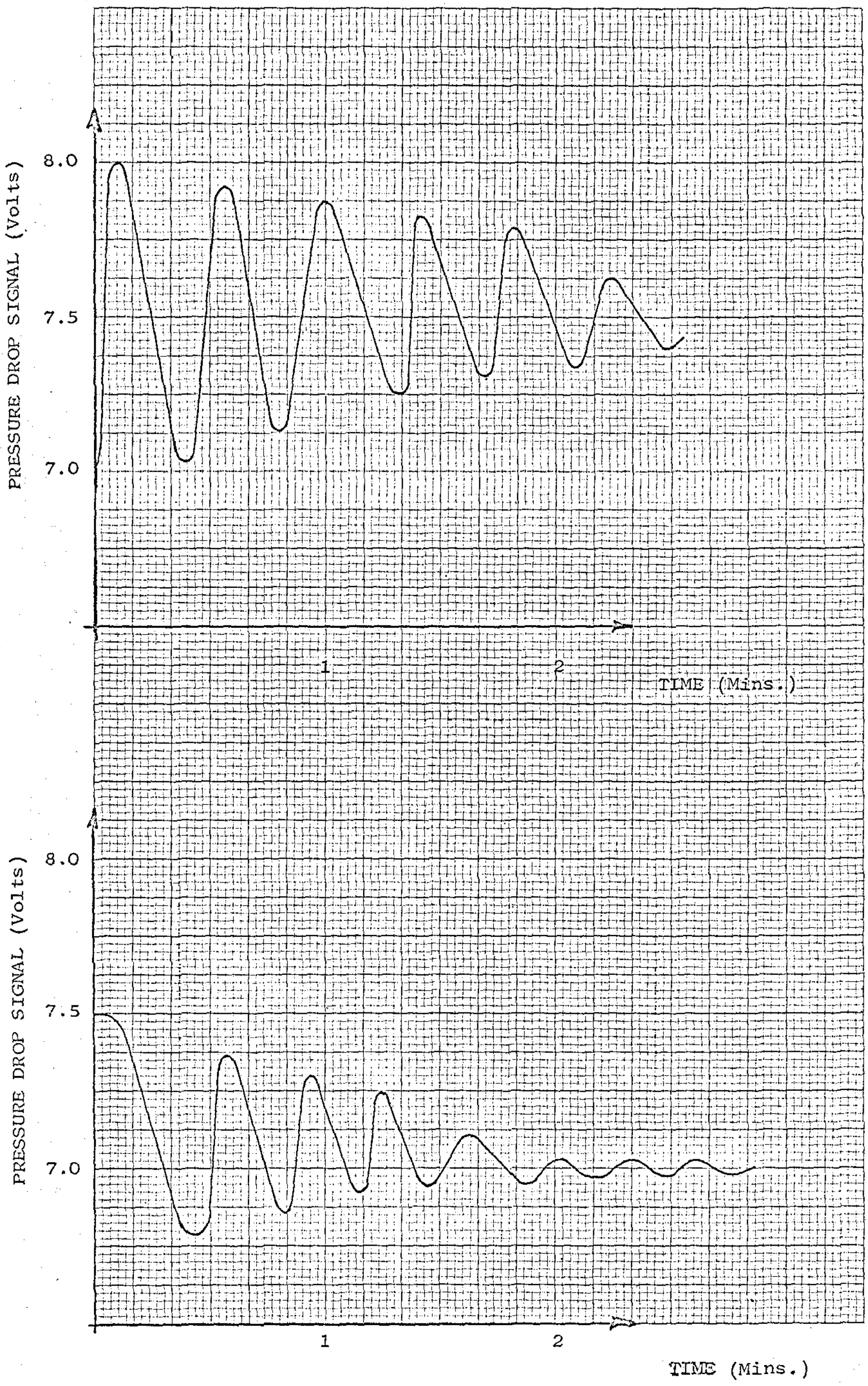
5.4 INITIAL DEVELOPMENT OF THE DIGITAL CONTROLLERS.

A model of a third order process was constructed on the analogue computer (a PACE TR 10). This model is indicated in Figure (5.3).

The open loop response of the model to set-point changes and load disturbances was first examined.

The digital control algorithms described by equations (5.3), (5.5) and (5.6) were tested initially using this simulated process. The effects of changing the sampling frequency, the controller constants, and the time constants of the process, were all examined using the analogue computer. Once the digital programmes for the controllers were functioning satisfactorily, and an indication of the performance of the controllers had been obtained, the controllers were employed on the distillation unit control loops.

The tuned controller settings for all the digital controllers were made on the basis of minimizing the integral of the error



GRAPH 5.3 PRESSURE DROP LOOP RESPONSE WHEN USING ANALOGUE CONTROLLER

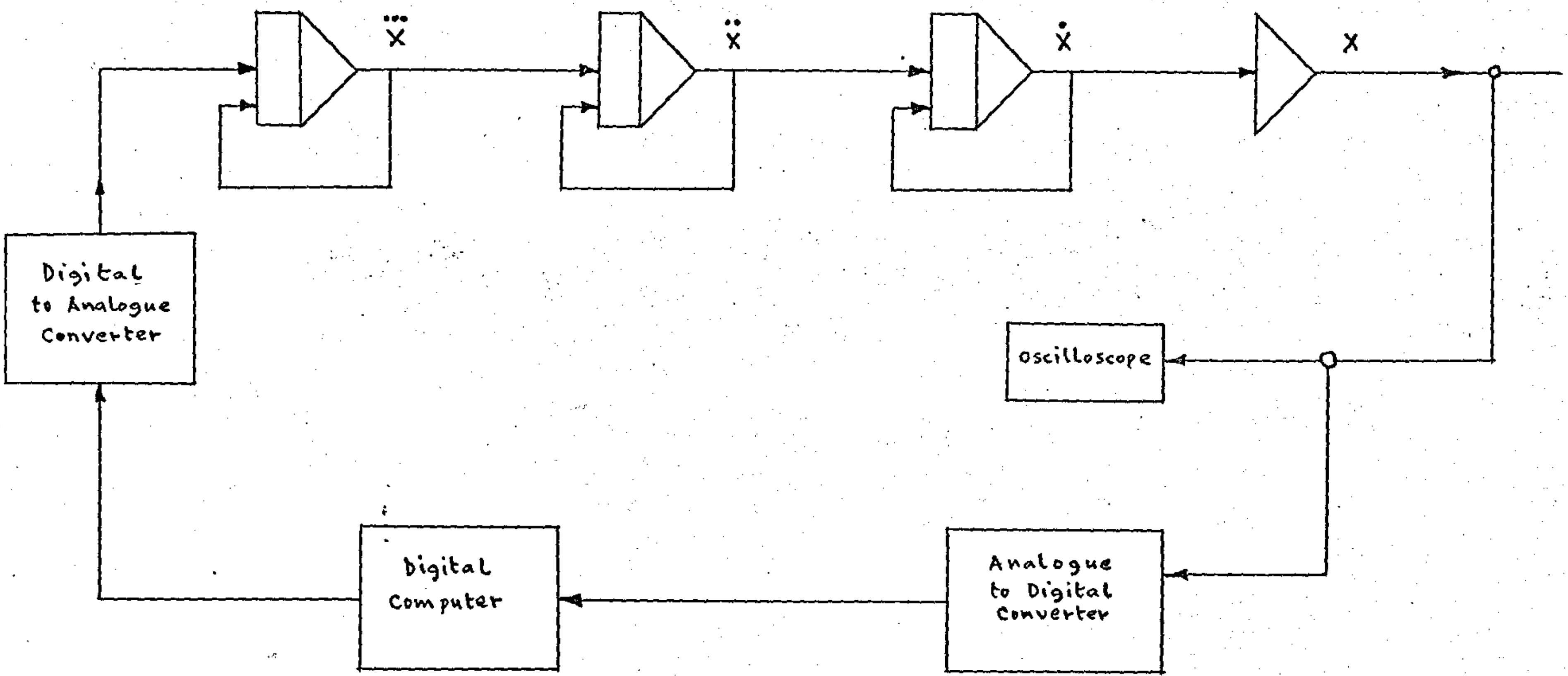


FIGURE 5.3

ARRANGEMENT OF 3RD. ORDER PROCESS ON THE ANALOGUE COMPUTER BY THE DIGITAL COMPUTER squared, and using the simplex technique described in detail in Appendix 6. In carrying out this integration, the maximum sampling rate was used throughout.

5.5 CONSIDERATION OF SAMPLING FREQUENCY.

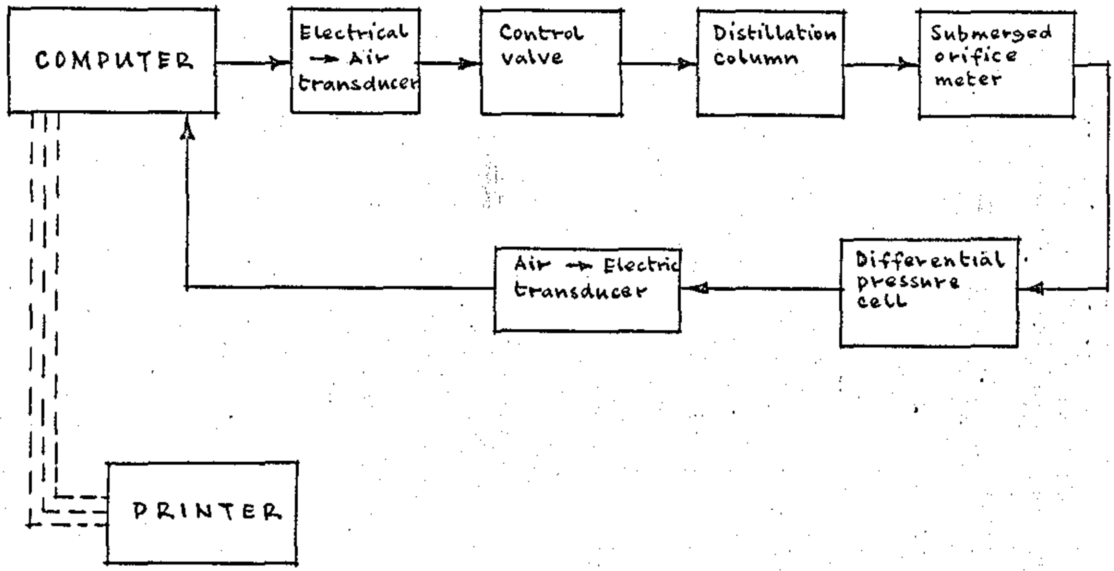
Choosing a suitable sampling interval is an important variable with digital controllers and there are different rules to determine the best sampling rate (46, 51 and 52). However, the rules may be generalized thus. If the time constant of the process is small, then generally a sampling interval of one third the time constant is required for adequate control. With larger time constants, it is necessary to decide what load disturbances are liable to occur, and what will be their effect on the process. However, generally, a sampling interval of less than a third of the time constant is required.

5.6 EXAMINATION OF THE OPEN LOOP RESPONSES OF THE SYSTEM.

Approximate values for the controller constants, and of the required sampling frequency were determined from the open loop responses of the systems. For the purpose of the experimental investigation, they were considered to include their peripheral equipment (Figure 5.4). The characteristics of the process were investigated by making step changes in the valve position.

The computer was used to actuate the valve, and the open loop response of the system logged. The responses for the reflux loop were plotted linearly and on a log-linear plot, and the results have been summarised in Table 5.1. It can be seen that the time constants and gains of the loops varied non-linearly over the whole range of operation.

In addition, it was found that for both the reflux flow loop and pressure drop loops, the time constant of the electrical to pneumatic transducers had a significant effect on their response.



Note. Output from Printer

1. Voltage Signal from Process Variable.

2. Voltage Signal Transmitted to Valve.

FIGURE 5.4

Arrangement Used to Determine the Open Loop Response of the Control Loops.

Initial Signal on Valve	Final Signal on Valve	v on Valve	v on Process	Dead Time on Process	Time Constant of Process	Gain
Volts	Volts	Volts	Volts	Secs.	Secs.	-
6.0	7.0	1.0	.94	2	88	.94
7.0	8.0	1.0	1.07	6	144	1.07
8.0	9.0	1.0	0.61	11.5	111	0.61
5.55	4.8	0.75	1.10	2	107	1.42
6.3	5.55	0.75	1.11	8.0	150	1.48
7.05	6.30	0.75	0.51	10.0	96	0.68

TABLE 5.1

OPEN LOOP RESPONSE OF THE REFLUX RETURN FLOW LOOP.

5.7 OUTLINE OF EXPERIMENTAL PROCEDURE FOR DIGITAL CONTROL OF THE DISTILLATION UNIT.

Once the controllers had been found to function satisfactorily with the model on the analogue computer, they were employed on the distillation unit.

It was found that the control algorithms had to be modified to handle the effects of the process dead time, and the noise level on the signal.

The effect of the dead time was that care had to be taken that integral saturation of the controller did not take place. This was easily dealt with by setting a maximum value to the integral action.

A variety of filters were employed in an endeavour to overcome the problem of process noise (Appendix 7). However, generally, a simple averaging filter was found to be satisfactory. In addition, a routine was included in the controllers, which processed the signals to avoid unnecessary fluctuations in the controller output when the process was at a steady state condition. This was achieved by taking corrective action only if the error signal was above a certain magnitude (this being fixed by the characteristics of the control loop (Figure 5.5).

The experimental procedure used to tune the controllers was as follows.

The plant was first controlled at a given set-point. Once steady state had been reached, a step change in the desired value

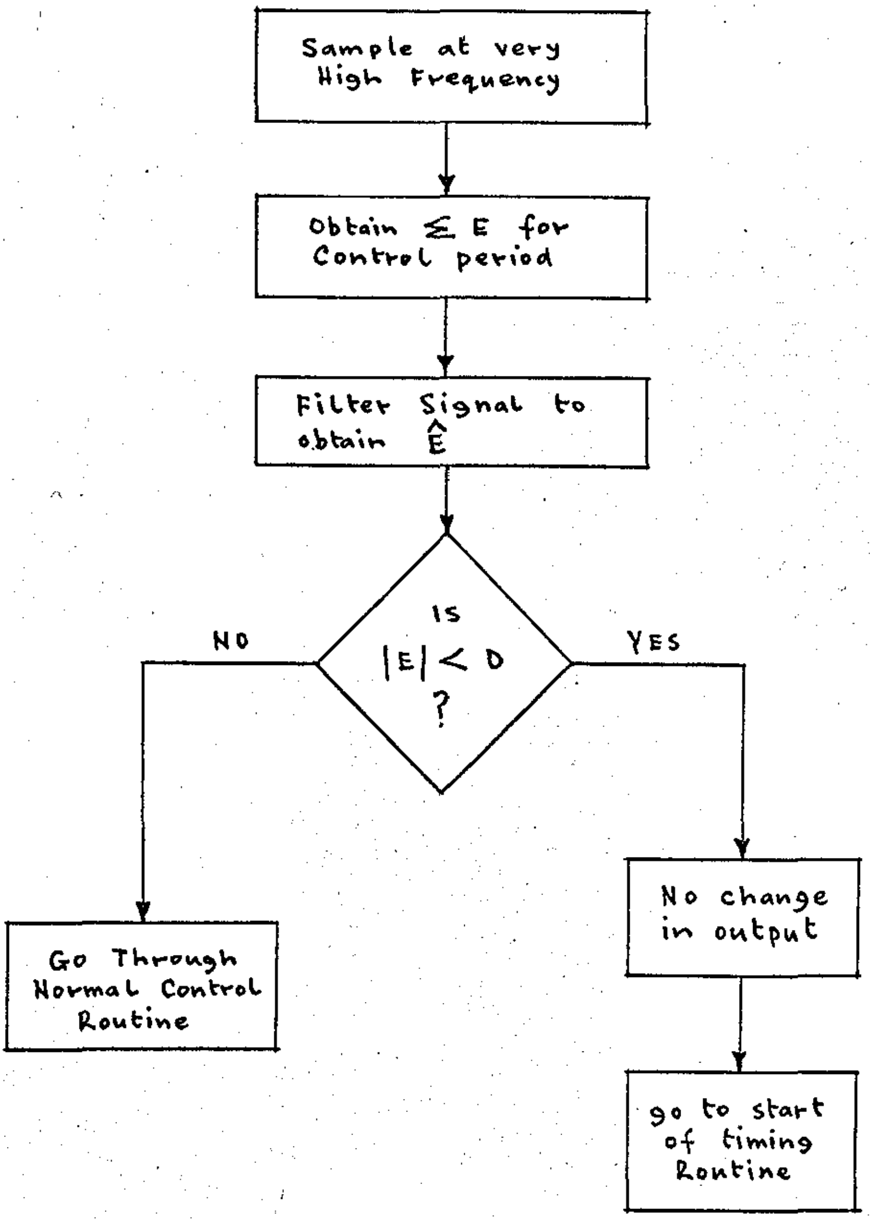


FIGURE 5.5

FLOW DIAGRAM INDICATING LOGIC TO AVOID UNNECESSARY FLUCTUATIONS IN CONTROL OUTPUT of the measured variable was made. As the controller brought the process to the new set point, the error in the measured variable was recorded at 0.1 sec. intervals. Subsequently, the value of the terms $\int E^2 dt$ $\int Edt$ and $\int Edt$ were obtained for an appropriate time interval. At first, before the controller constants had been approximately tuned, the values of $\int E^2 dt$, for periods of approximately two and four times the loop time constants, were determined.

This was because initially the loop took some time to reach steady-state, whilst, when the controller had been tuned, care had to be taken not to saturate the results from the step change by noise whilst the process is at steady-state.

This experimental step was then repeated with a change in set point back to the initial value of the measured variable.

The performance index was determined by summing the respective integration values for the step changes in each direction. This technique was employed to overcome the non-linear effects of the control loops. For the initial work, the performance index of $\int E^2 dt$ was chosen.

5.8 EXAMINATION OF THE REFLUX FLOW LOOP.

The open loop response work on the reflux flow loop indicated a time constant of the order of 100 secs. However, its time constant appeared to vary over the operating range of the loop.

It was decided to investigate the reflux flow loop by using three sampling periods.

(1) 0.1 secs. :- this was as fast as the interrupt routine would allow, and hence approximate towards continuous sampling.

(2) 2 secs. :- frequent sampling, but allows other loops to be controlled as well. Appears to be the most satisfactory sampling frequency.

(3) 30 secs. :- considered as the largest sampling period which could control the process satisfactorily.

The simplex operation outlined previously was first used to determine the best controller constants at the various sampling intervals, using the velocity algorithm. The results, when using a sampling interval of 0.1 second, have been outlined in Table 5.2'a'. Those from using sampling intervals of 2 seconds and 30 seconds are outlined in Tables 5.3 and 5.4 respectively.

Once the investigation with the standard algorithm had been completed, the effect of using a rate restraint to govern the valve movement was considered. Various values for the rate restraint were employed in conjunction with certain specified controller constants that had already been examined. Their effects on the performance index was then determined by comparison (Tables 5.2'b' and 'c').

It can be seen from these results that the use of the

Point No.	Value of K_1	Value of K_2	E^2 dt
* 1	10	.006	762,122
2	8	.008	1,971,846
3	7	.004	1,479,134
4	9	.002	935,056
* 5	12	.004	1,391,211
6	13	.008	1,527,261
7	10	.006	623,296
* 8	11	.010	1,672,763

TABLE 5.2'a'.

Point No.	Value of K_1	Value of K_2	E^2 dt
1(a)	10	.006	1,498,151
5(a)	12	.004	722,151
8(a)	11	.010	628,869

TABLE 5.2 'b'.

Point No.	Value of K_1	Value of K_2	E^2 dt
1(b)	10	.006	1,273,468
5(b)	12	.004	1,191,381
8(b)	11	.010	1,241,985

TABLE 5.2'c'.

TABLE 5.2 :-

Results of Simplex Analysis Using Velocity Algorithm to Control Reflux Flow Loop (Sampling Interval 0.1 sec.).

(a) Without rate constraint

(b) Rate constraint of 2% value movement per sample period

(c) Rate constraint of 1% value movement per sample period

Point No.	Value of K_1	Value of K_2	E^2 dt
1	11.0	.11	1,204,654
2	10.0	.07	1,200,093
3	8.0	.10	544,726
4	7.0	.06	480,396
5	5.0	.09	720,595
6	4.0	.05	884,682
7	7.0	.06	640,240
8	5.5	.055	710,971
9	6.0	.075	887,841
10	6.5	.04	800,559
11	8.0	.045	890,453
12	8.5	.065	885,265
13	7.5	.08	640,997

TABLE 5.3.

Results of Simplex Analysis Using Velocity Algorithm to Control Reflux Flow Loop (Sampling Interval 2.0 sec.).

Point No.	Value of K_1	Value of K_2	E^2 dt
1	5.0	1.0	4,501,154
2	3.0	0.8	2,643,421
3	4.0	0.4	3,531,986
4	2.0	0.2	5,251,385
5	1.0	0.6	5,062,384
6	2.5	1.5	2,631,391

TABLE 5.4.

Results of Simplex Analysis Using Velocity Algorithm to Control Reflux Flow Loop (Sampling Interval 30 sec.).

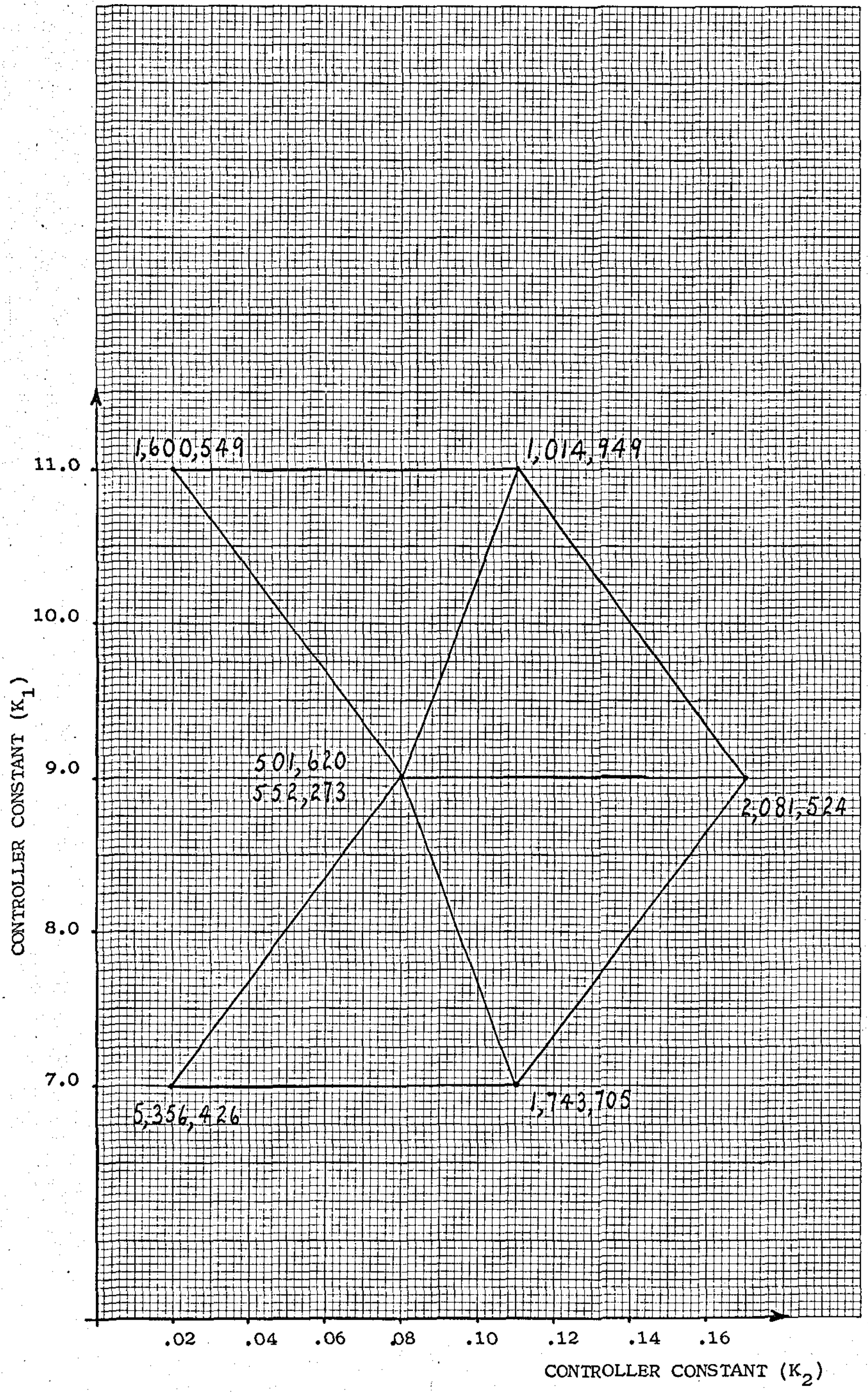
restraint tends to result in greater stability of the process over a wider range of controller constants. Generally, however, a slightly higher gain is required than when the restraint is not included. The restraint has two effects. Firstly, it stops too drastic corrective action initially, and secondly, it slows down the rate at which the controller is able to take corrective action.

The procedure was repeated using the other controller algorithms, and the effects of tuning the controllers are outlined in Graphs 5.4 and 5.5.

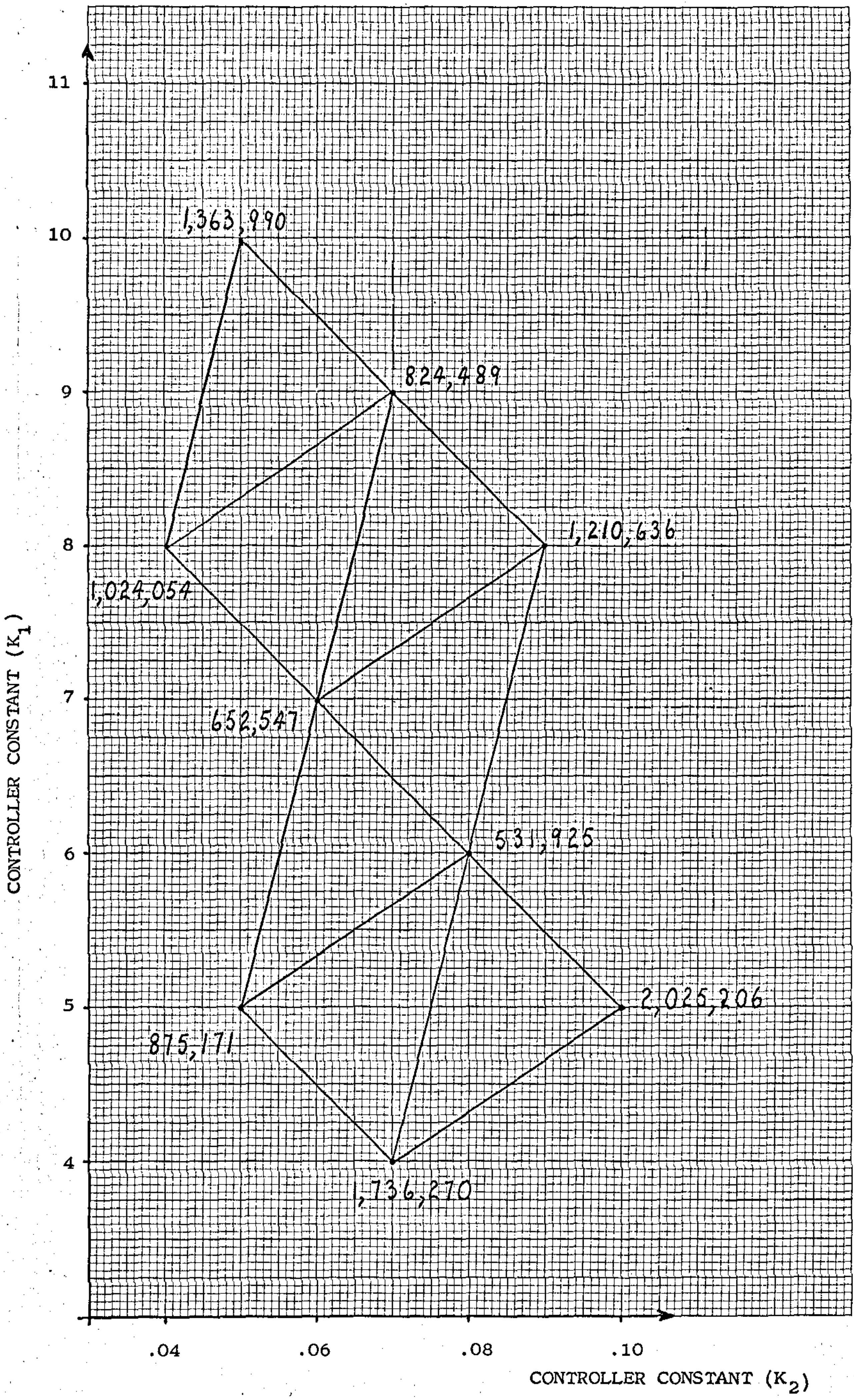
Finally, the effect of using the non-linear controller was considered. This controller has been outlined in more detail elsewhere (Section 5.1.2 and Appendix 5). Here it will suffice to list its three main advantages.

1. It accelerates the corrective action when the process variable is some distance away from the desired set-point.
2. The corrective action it takes is dependent on the direction of the process variable velocity.
3. Decreased controller gain around the set-point.

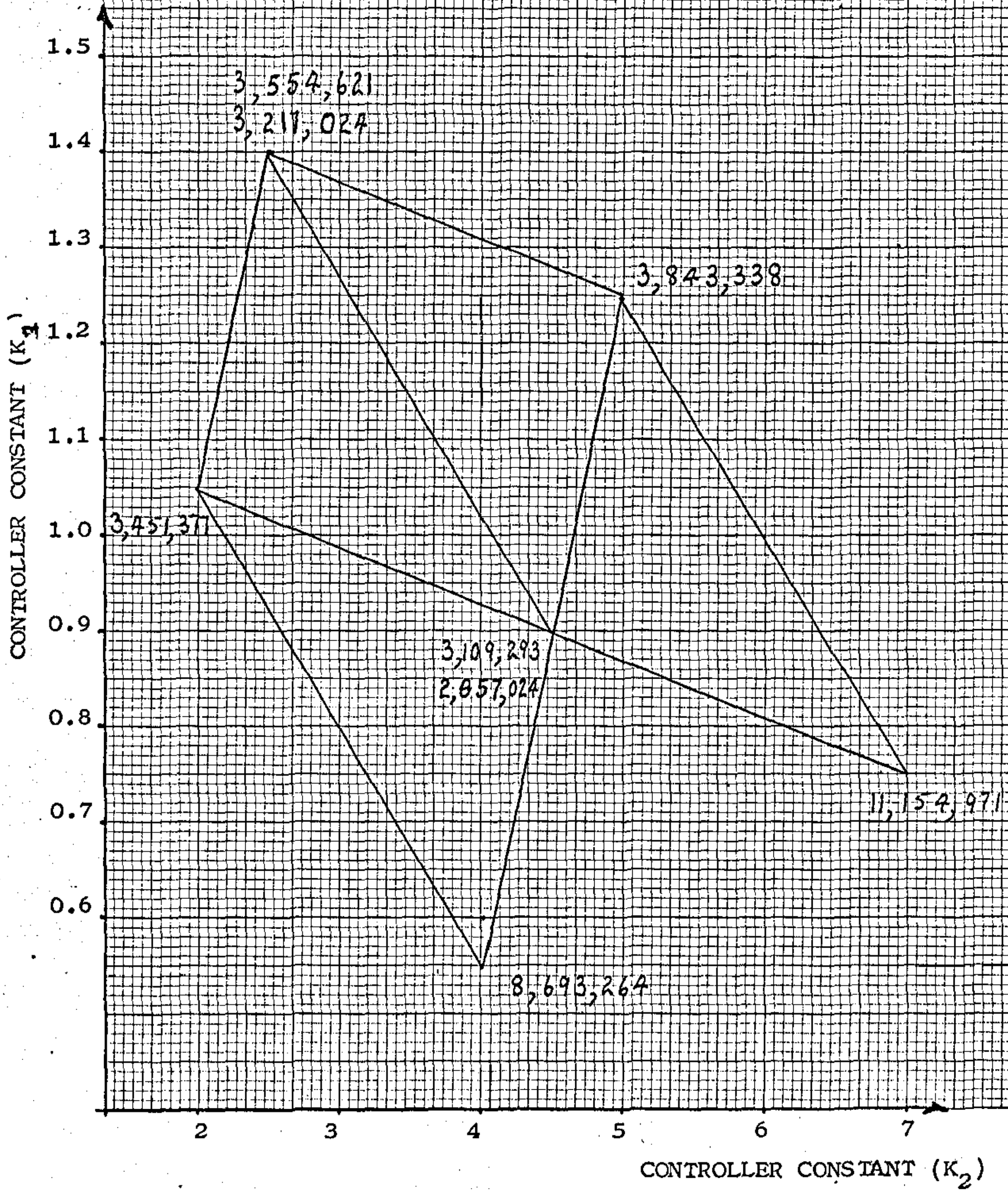
The controller was first examined on the reflux control loop using the same relatively small set point changes that had been employed previously ($v = \pm .75v$). The results of this examination are outlined in Table 5.5. However, since the reflux flow loop was relatively straightforward to control, and the disturbances considered were quite small, this was not a very satis-



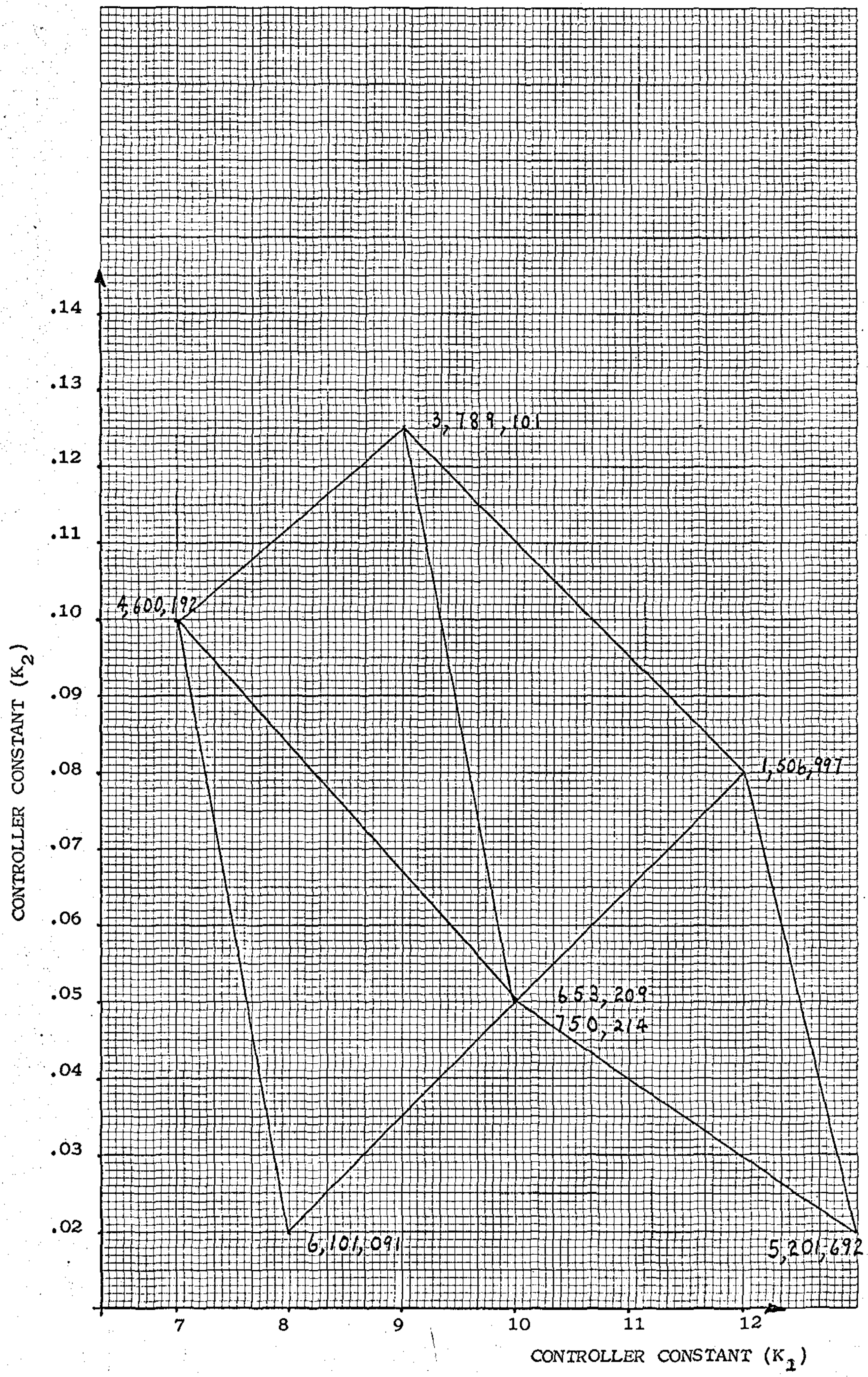
GRAPH 5.4(a) SIMPLEX USED TO TUNE TRUNCATED CONTROLLER (ΔT=2Sec.)



GRAPH 5.4(b) SIMPLEX USED TO TUNE POSITION ALGORITHM (ΔT=2 Sec.)



GRAPH 5.5(a) SIMPLEX USED TO TUNE VELOCITY ALGORITHM (Δ T=30 Sec.)



GRAPH 5.5(b) SIMPLEX USED TO TUNE TRUNCATED CONTROLLER (Δ T=0.1Sec.)

Point No.	Value of K_1	Value of K_2	E^2 dt
1	7	.06	480,396
2	8	.10	544,726
3	7.5	.08	640,997

TABLE 5.5 'a'.

Linear Velocity Controller.

Point No.	Value of K_1^1	Value of K_2^1	Value of K_1^2	Value of K_2^2	E dt
1	8	.07	4	.03	380,010
2	12	.10	5	.04	340,674
3	10	.08	4	.05	485,631

TABLE 5.5 'b'.

Non-Linear Velocity Controller, Special Action Band-Width .75v.

TABLE 5.5

Results of Analysis Comparing Control of Reflux Flow Loop Using Linear and Non-Linear Velocity Controllers (Sampling Interval 2.0 secs.).

factory test of the controller. The controller was also employed on the boil-up loop, and the results of this examination were more interesting (Section 5.9.1).

5.9 EXAMINATION OF THE BOIL UP LOOP.

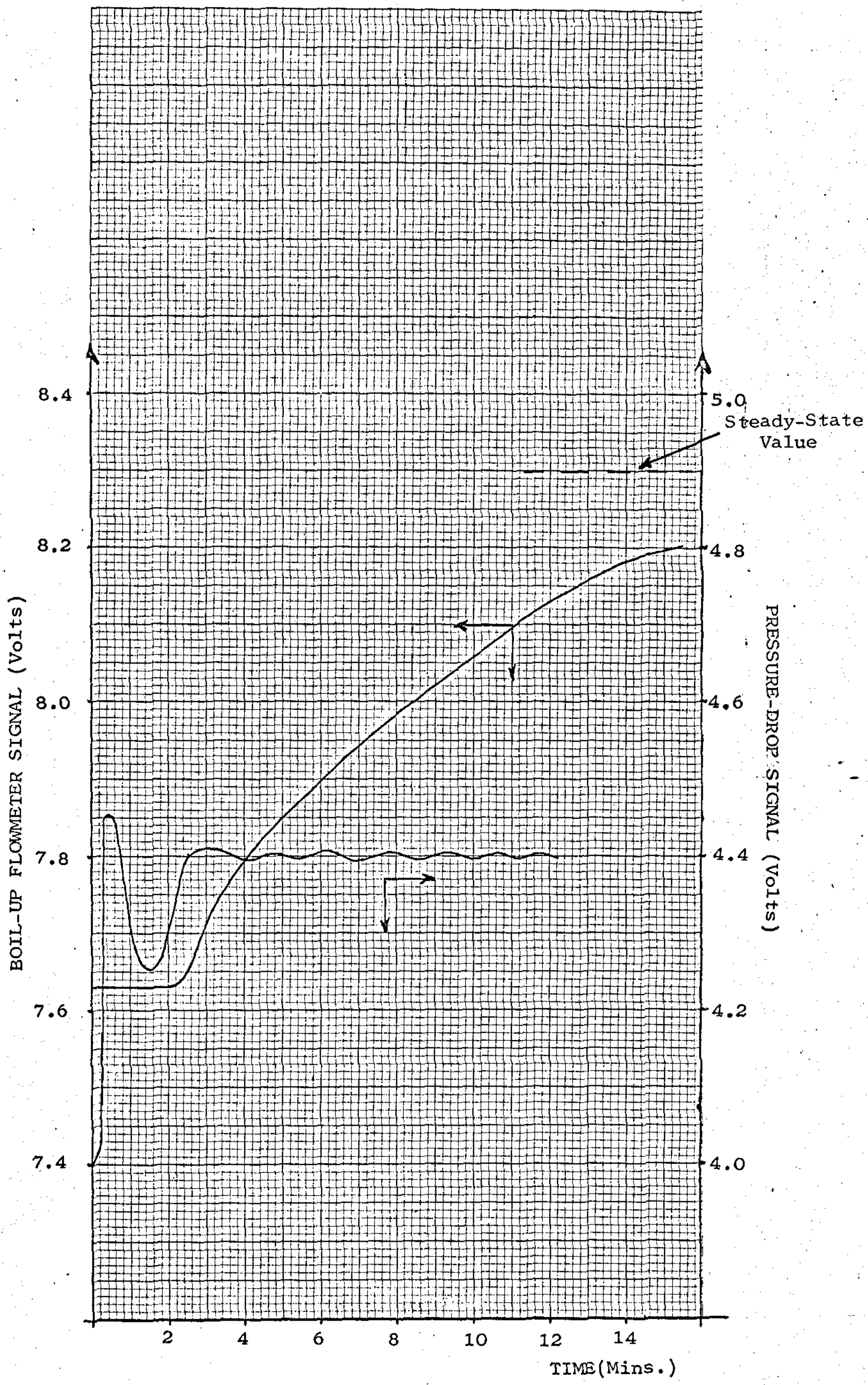
5.9.1 BOIL UP/FLOW METER LOOP.

The flow meter is a measure of the condensate leaving the condenser. The vapour rate up the column is this, plus the vapour condensed by the cool reflux. Because of the hold-up in the column and auxiliary equipment there is a substantial time lag in the response of the system.

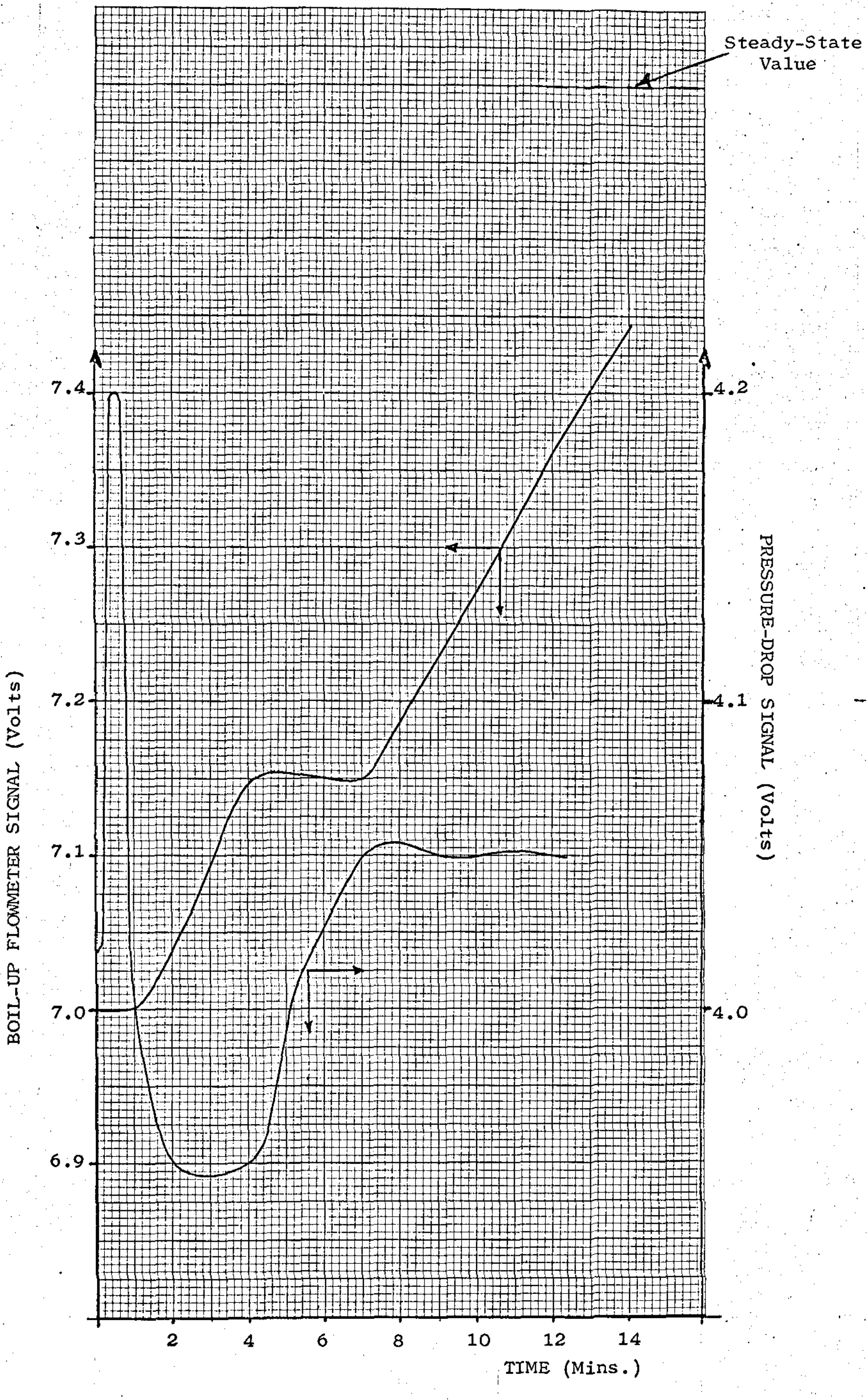
To examine this system its open loop response was first determined.

From Graph 5.6 ~~(a)~~ it can be seen that the system is non-linear, and in addition, there are significant differences in its characteristics depending on the direction of the forcing function.

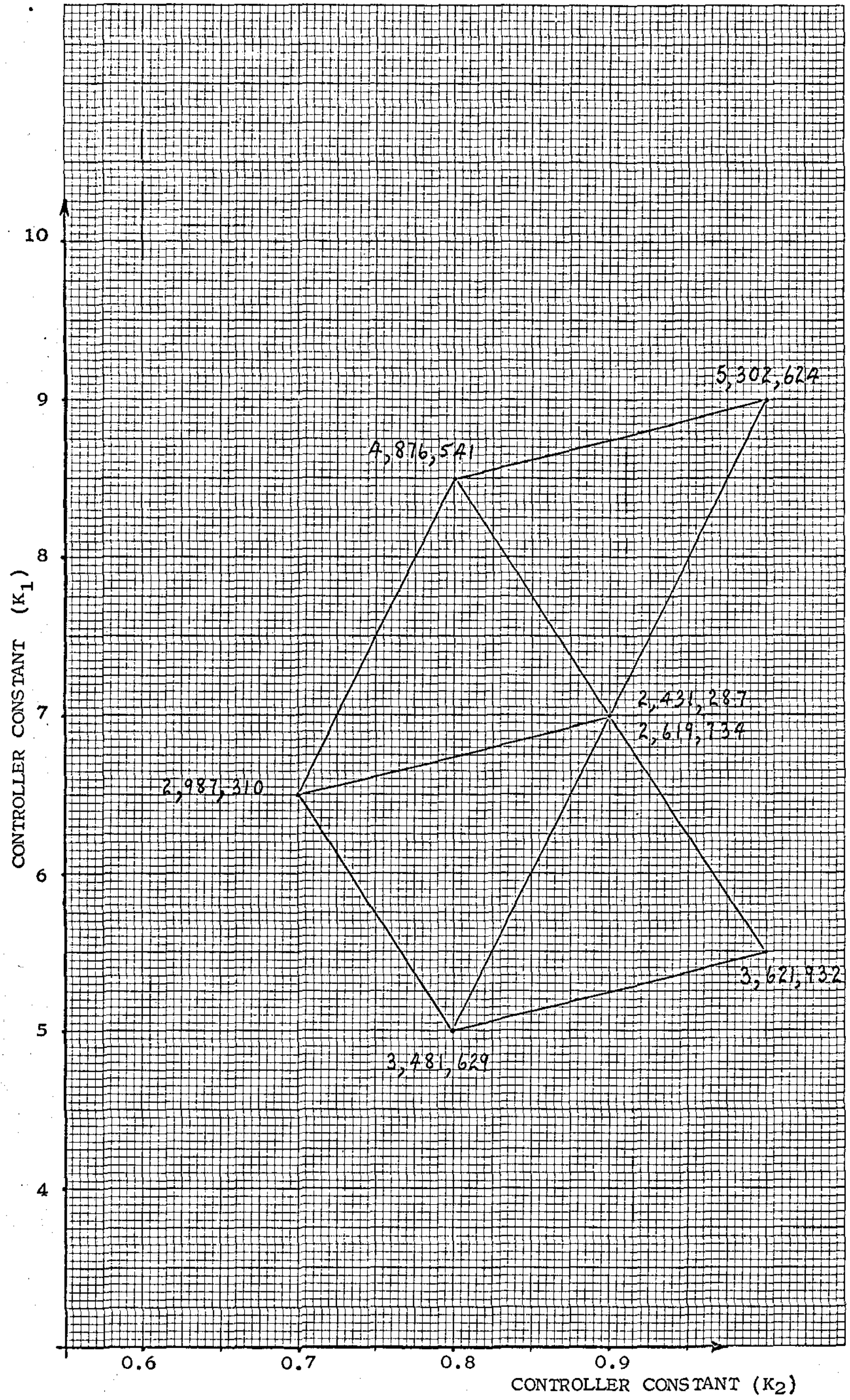
The system may be examined in a similar manner to that outlined for the reflux flow loop. Typical results are outlined in Graphs 5.7 & 5.8, and in Table 5.6.



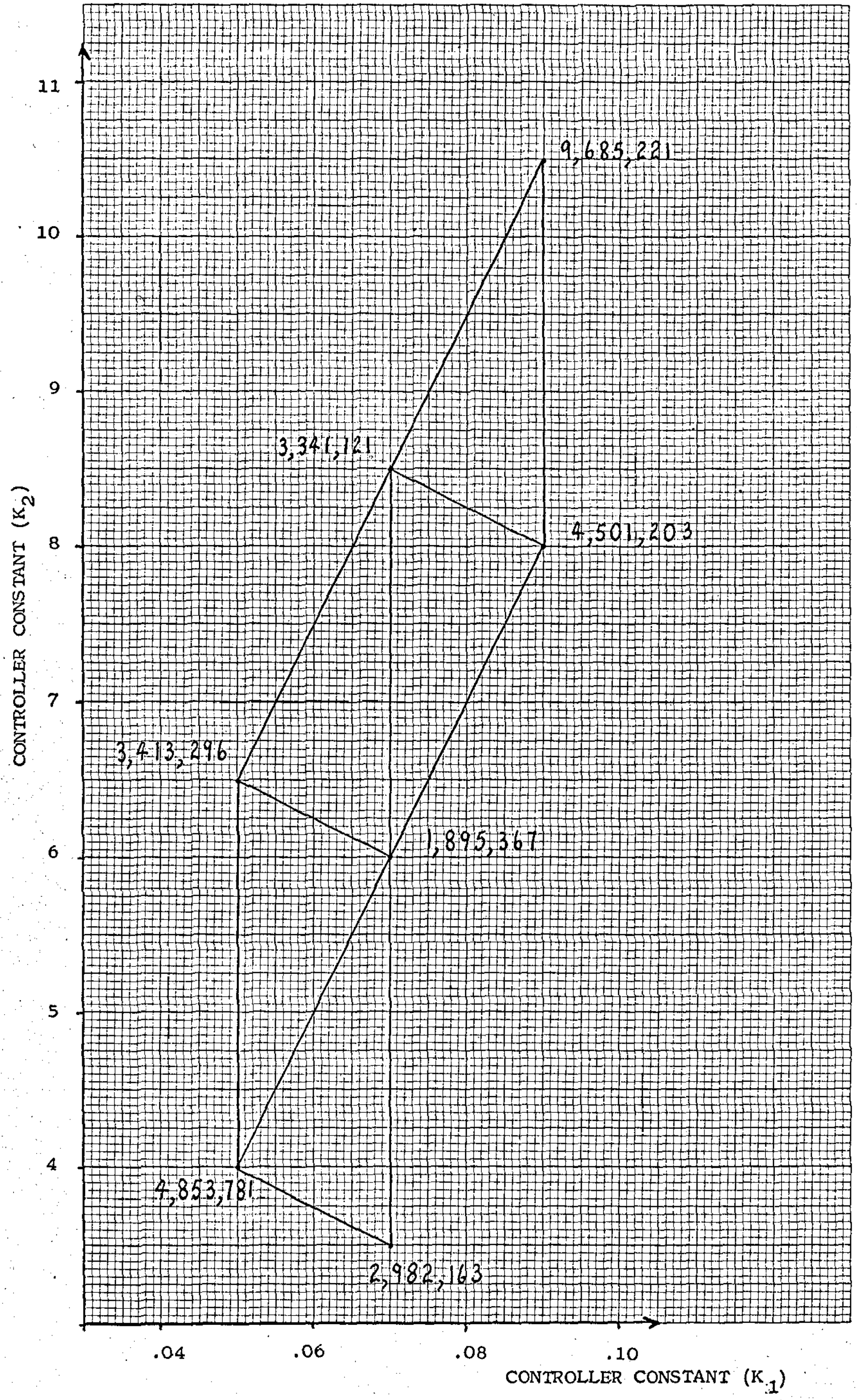
GRAPH 5.6(a) OPEN LOOP RESPONSE TO A CHANGE IN BOIL-UP RATE



GRAPH 5.6(b) OPEN LOOP RESPONSE TO CHANGE IN BOIL-UP RATE



GRAPH 5.7 SIMPLEX USED TO TUNE POSITION ALGORITHM (ΔT = 30 Sec.)



GRAPH 5.8 SIMPLEX USED TO TUNE VELOCITY ALGORITHM (ΔT=2.0 Sec.)

Point No.	Value of K_1	Value of K_2	E^2 dt
1	6	.07	1,895,367
2	8.5	.07	3,341,121
3	8	.09	4,501,203

TABLE 5.6 (a)

Linear Velocity Controller.

Point No.	Value of K_1^1	Value of K_2^1	Value of K_1^2	Value of K_2^2	E^2 dt
1	8	.07	5	.04	1,781,232
2	9	.06	4	.04	1,501,044
3	10	.07	6	.05	1,671,673

TABLE 5.6 (b)

Non-Linear Velocity Controller Special Action Band-Width .75v

TABLE 5.6.

Results of Analysis Comparing Control of Boil-Up Flow Loop Using Linear and Non-Linear Velocity Controllers (Sampling Interval 2.0 secs).

5.9.2 BOIL-UP/PRESSURE DROP LOOP.

The open loop response of this system was examined in the manner previously explained. Typical results are illustrated in Graph 5.6(b).

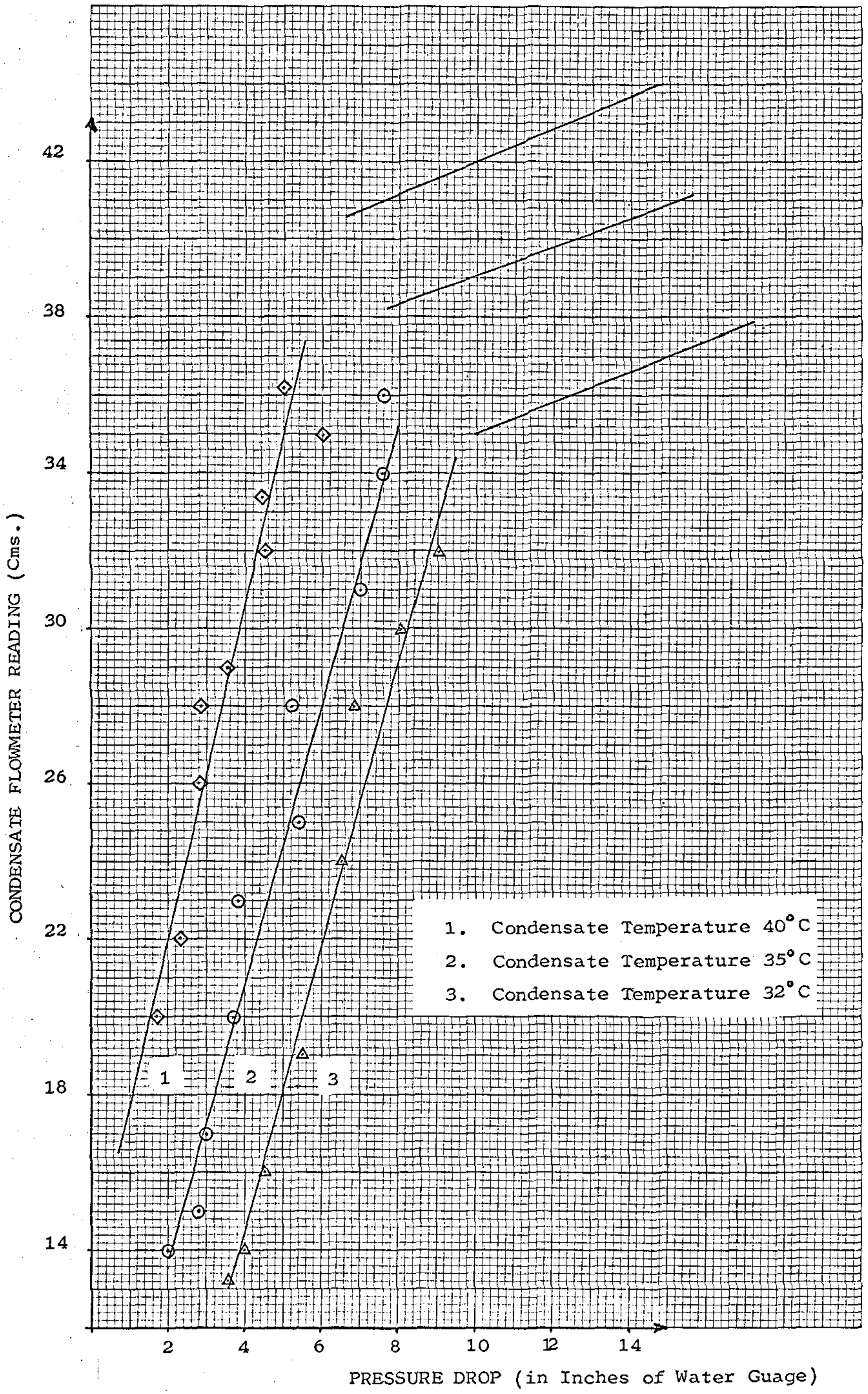
The relationship between the flow meter reading and the pressure drop is illustrated by the plot of the steady state flowmeter and pressure drop readings (Graph 5.9).

Once again the measured variable is the vapour rate up the column but in this case it is assessed by determining the pressure drop across the column. For this reason, the dynamic response is a measure of the balance between vapour rate up the column, liquid rate down the column, and the column hold-up.

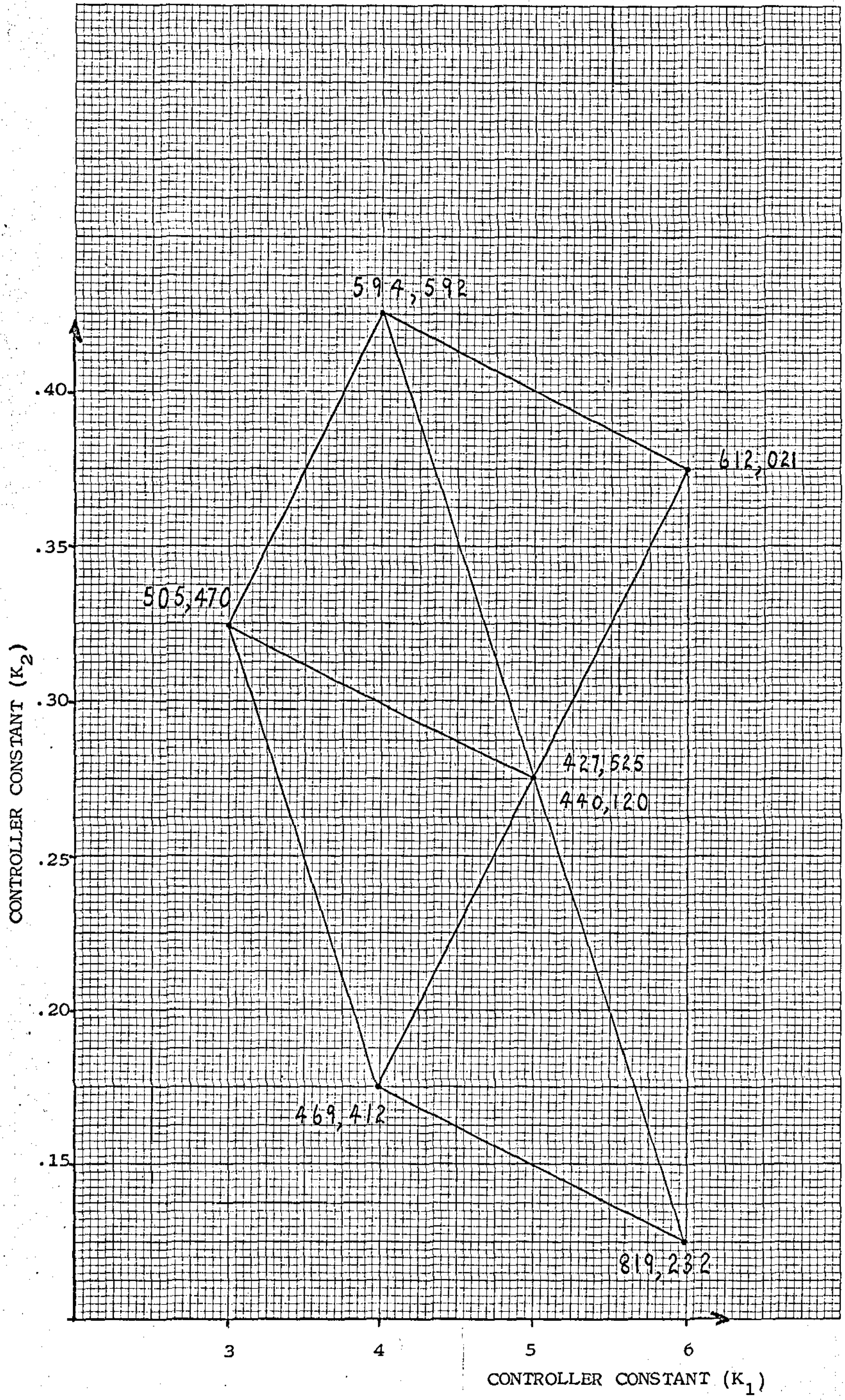
The advantage of the loop is that its time constant is small. However, its disadvantage is that the ~~gain~~ signal/noise ratio is quite low. In addition, its response to a load change may include pulsations. Because of this pulsation a large sampling interval would be desirable. However, the small time constant of the process does not permit this.

The controller constants were again tuned by use of the Simplex Evolutionary Technique (Graph 5.10).

However, because of the non-linear characteristics of the process, and in particular the pulsations that result from an increase in vapour rate, satisfactory control was difficult to obtain. The effects of these characteristics on the controlled



GRAPH 5.9 PLOT OF FLOWMETER READING AGAINST PRESSURE DROP ACROSS COLUMN



GRAPH 5.10 SIMPLEX USED TO TUNE POSITION ALGORITHM (ΔT=2 Sec.)

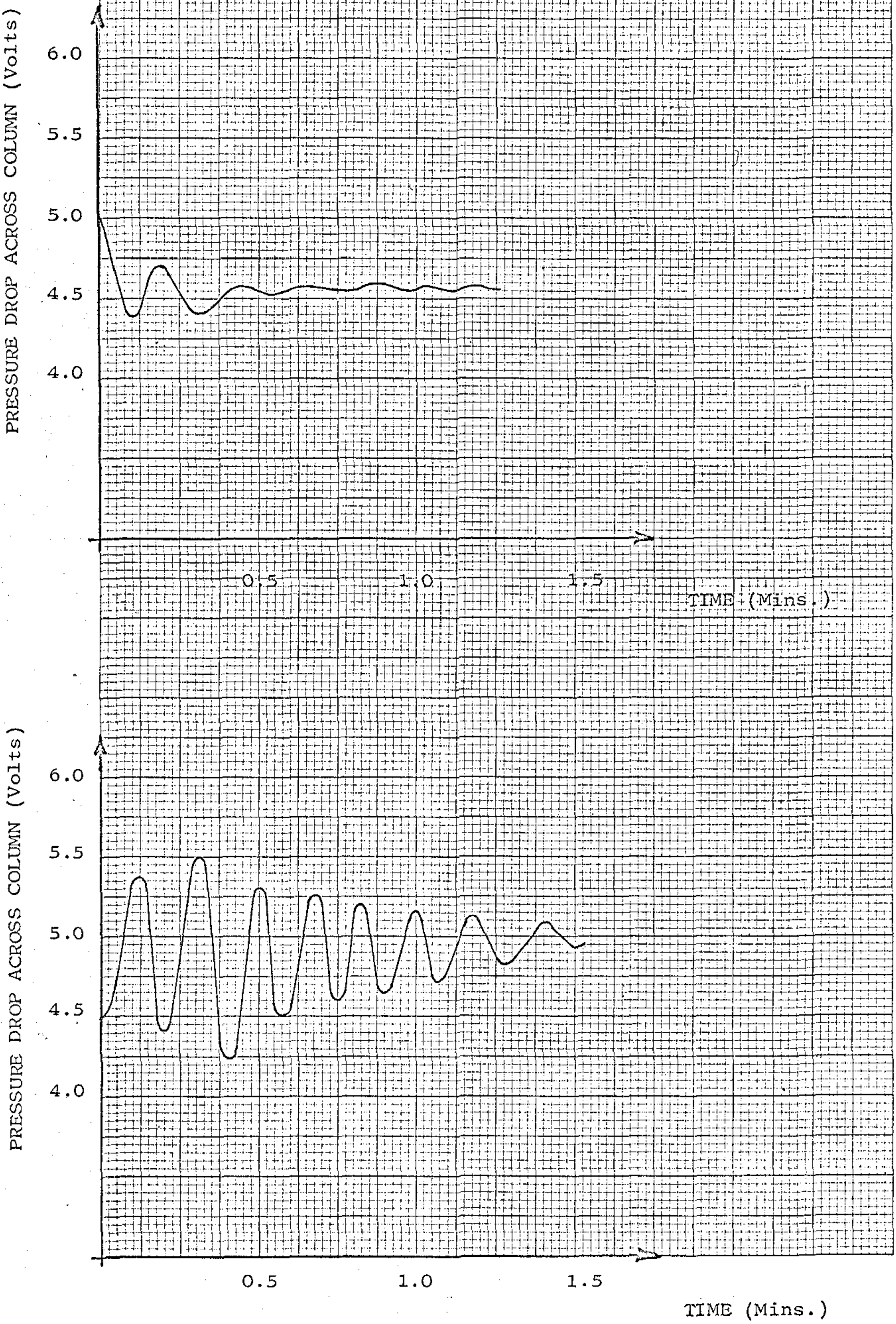
response of the loop are illustrated in Graph 5.11. It may be seen from these response curves, that satisfactory control of the loop is not possible when using a simple controller algorithm. However, by employing a more sophisticated controller, it is possible to utilise the measurement of this process variable (Section 5.10.1).

5.10 THE APPLICATION OF CASCADE CONTROL LOOPS TO THE DISTILLATION UNIT.

5.10.1 CASCADE CONTROL LOOPS APPLIED TO THE CONTROL OF THE BOIL-UP RATE.

The control of the boil-up loop under a cascade system was with the condensate flow-rate measurement used as the measured variable to the master controller, and with the pressure drop signal as the measured variable to the secondary controller which actuated the steam valve.

The through-put of a distillation column, for a given separation, is primarily a function of the boil-up rate. Hence, endeavours were made to develop a system to allow the column to be operated as near its flooding point as possible, and this requires close control of the boil-up loop. Graph 5.6 indicates the open loop responses of the flow-meter loop and the pressure-drop loop. Both of the process variables are a measure of the vapour velocity up the column. However, the pressure drop is a measure of the instantaneous vapour velocity, where-as the condensate flow meter is a measure of the vapour leaving the top of the column at any time. As a result, there was a dead time of several minutes before



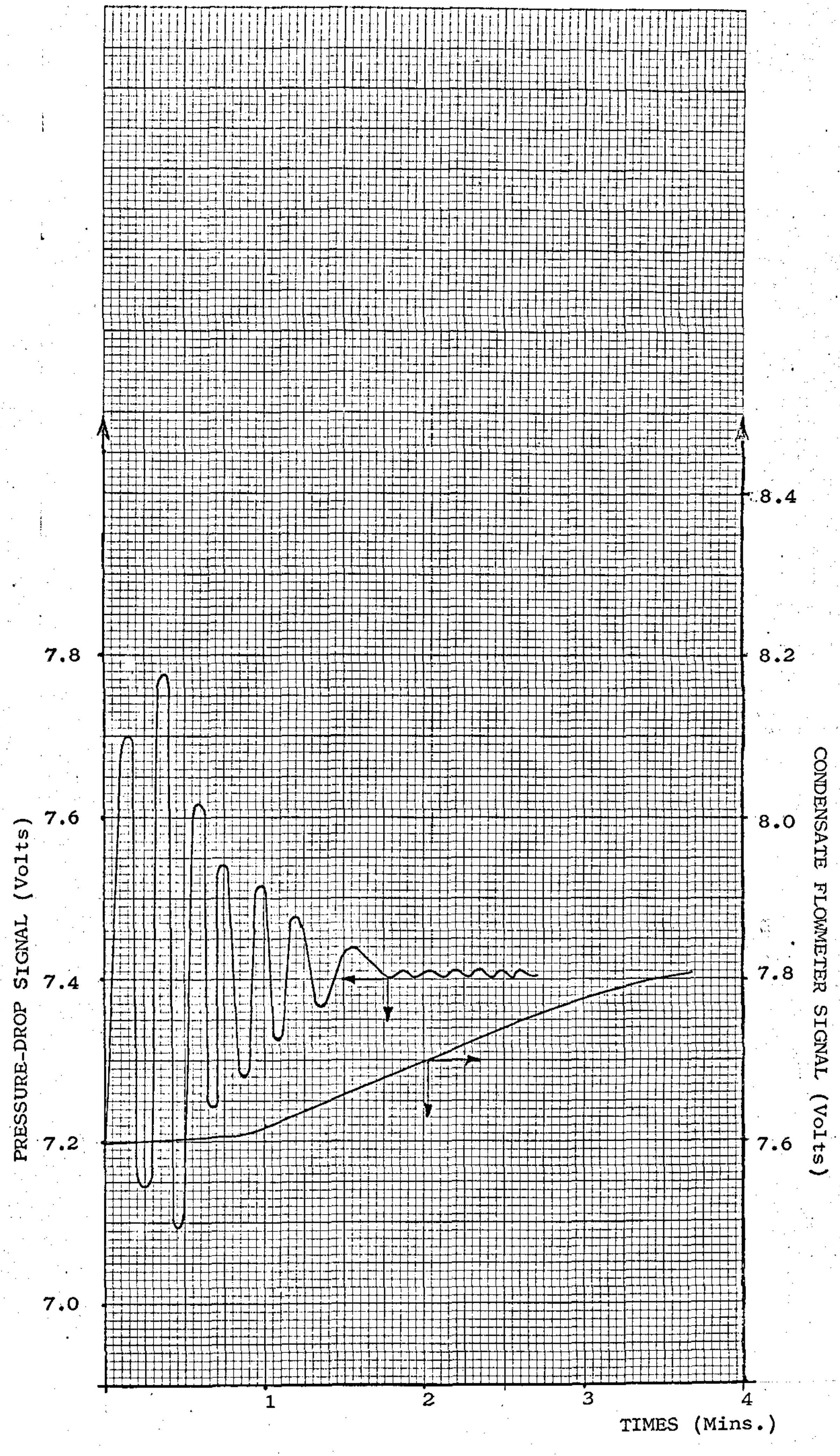
GRAPH 5.11 RESPONSE OF PRESSURE DROP LOOP USING VELOCITY ALGORITHM

a change in vapour rate was detected by the flowmeter loop, the hold-up of the condenser and flowmeters, which was of the order of 1.5 litres, and that of the column, which was about 2.5 litres, caused it to have an open loop response time of the order of 15 minutes.

However, that of the pressure drop loop was about 3 minutes. Because of the basis of the pressure drop loop, it was found that a sudden increase in hot vapour flow increased the pressure drop across the column immediately, but sometimes resulted in transient flooding, without, however, inducing permanent instability. For this reason, the pressure-drop sensor may be used to detect a change in the dynamic equilibrium of the system almost immediately. However, whilst the flowmeter loop detects some pulsations of the vapour up the column, in the main they are damped out.

If only the pressure drop across the column were used as the controlled variable, then the response of the system to a step change is as indicated on Graph 5.12. It can be seen that adequate control of the boil-up loop was not possible. This was especially true when the colum was operated near its flooding point. The use of the pressure drop loop to control the vapour rate through the column is explained in more detail in Section 5.9.2.

Adequate control of the boil-up could be obtained by using the flow meter loop, providing the boil-up rate was well below the flooding rate. Unfortunately, it was found that when the flowmeter loop was used to control the column just below the flooding



GRAPH 5.12 PRESSURE-DROP CONTROL ON BOIL-UP LOOP, FLOW RATE UNCONTROLLED

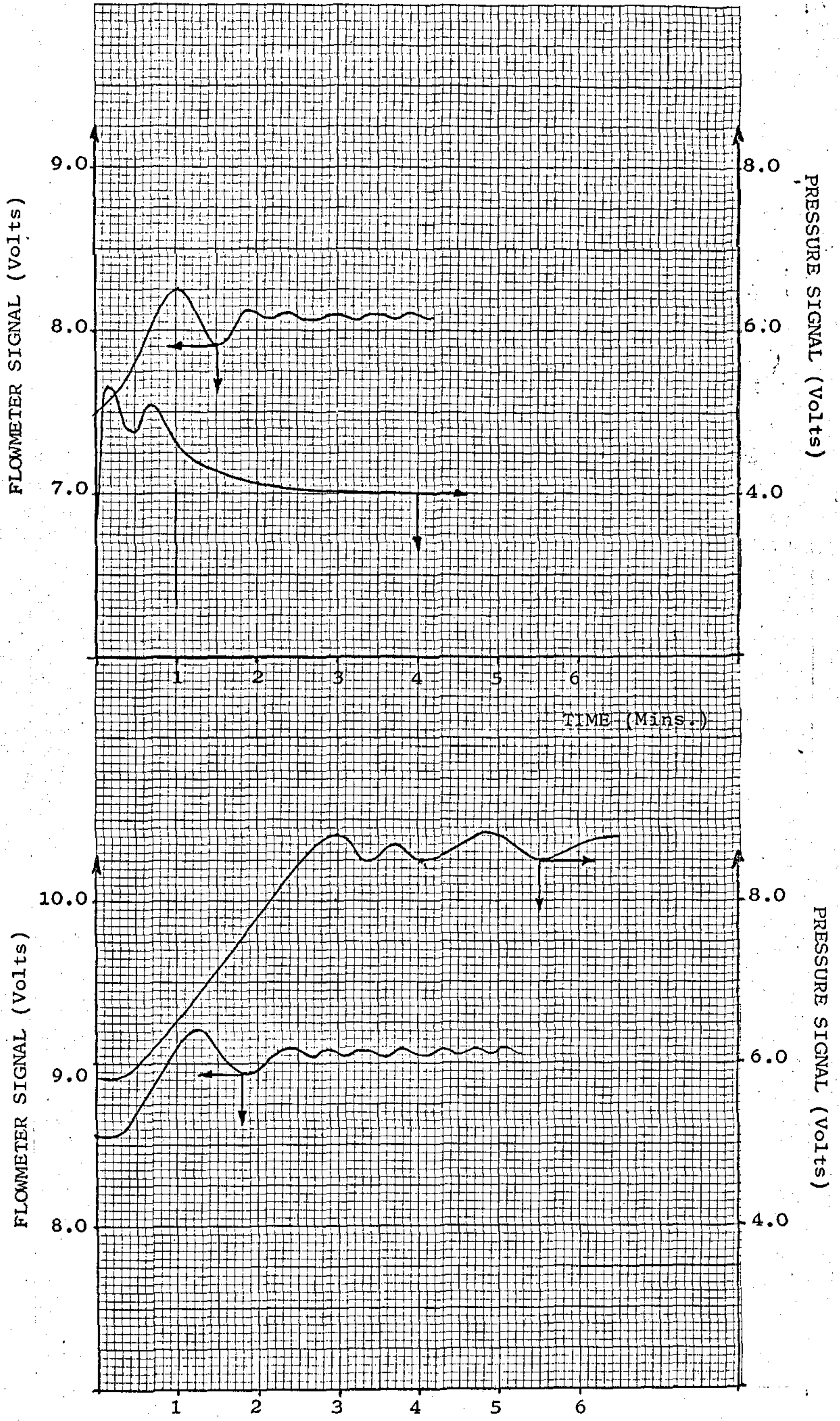
point, the system was unstable (Graph 5.13).

Observation of the dynamic pressure drop across the column suggested that a rate constraint on the steam valve movement could be used to avoid flooding (Graph 5.6). This was because the instantaneous pressure drop was particularly susceptible to changes in the steam signal. Restriction on the movement of the steam valve was therefore included in the controller programme.

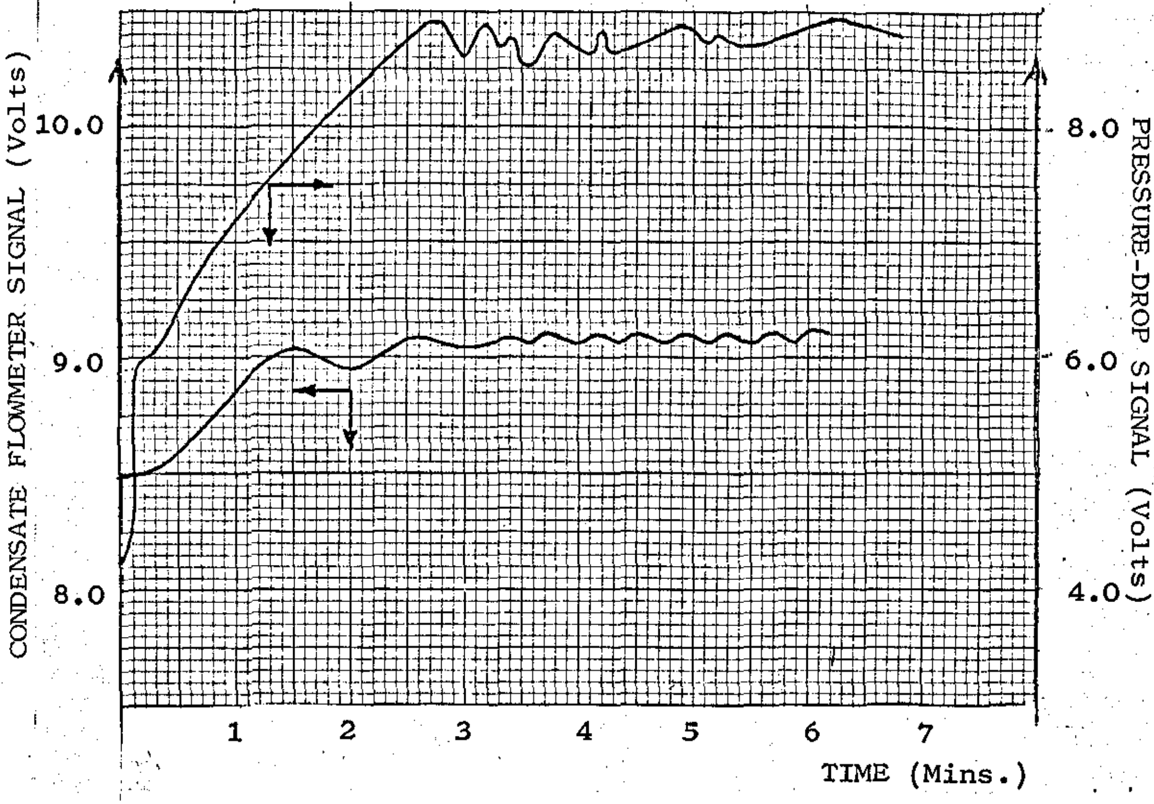
By referring to Graph 5.14 it can be seen that the rate of valve movement had to be restricted to 4% per minute, before an appreciable improvement was detected. However, even then, satisfactory control of the system was not obtained, since the process was still liable to become unstable.

Although a change of controller constants gave an improved response, the control of the boil-up rate was still not satisfactory (see Graph 5.14 (d)).

As a result of the work outlined above, it was decided that to operate the column near its flooding point it would be necessary to control both the pressure drop across the column and the boil-up flow meter reading. It is felt that by using both the variables, the rate of off-take at the top of the column could be controlled at its maximum. The measurements of the fast response pressure drop loop ensured that pressure surges through the column could be detected, and dealt with, before they caused flooding to occur within the column. The use of the flow-meter as the master controller,



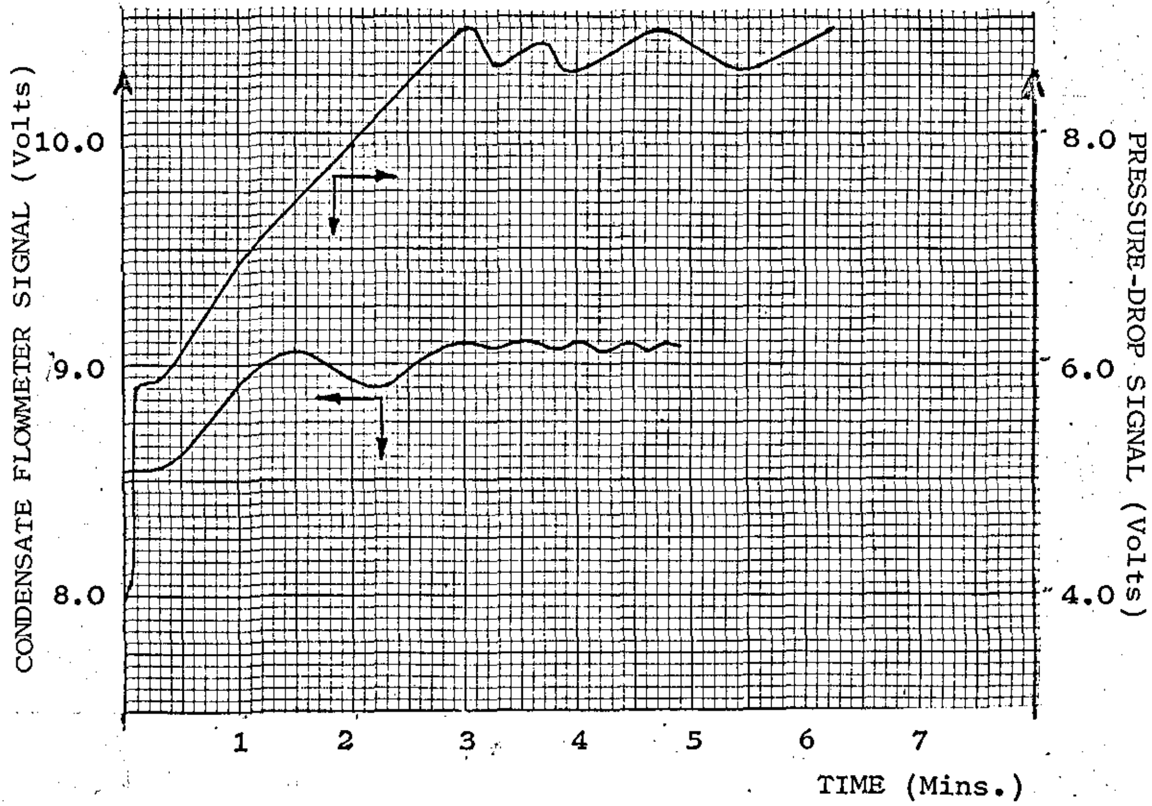
GRAPH 5.13 FLOW RATE CONTROL ON BOIL-UP LOOP, PRESSURE-DROP UNCONTROLLED



Controller Constants; K_1=7.0, K_2=0.25

No Restraint on Valve Movement, and Column Unstable

(ΔT=15 Sec.)

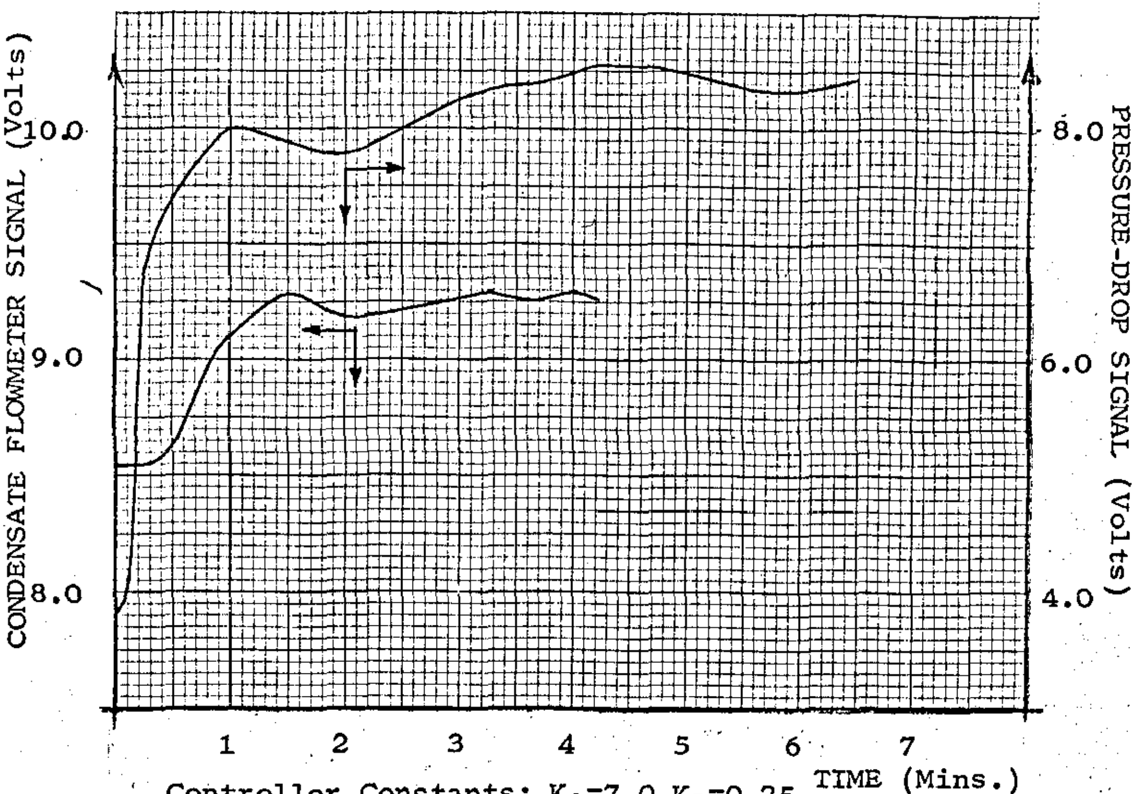


Controller Constants; K_1=7.0, K_2=0.25

Max. Valve Movement — 10%, and Column Unstable

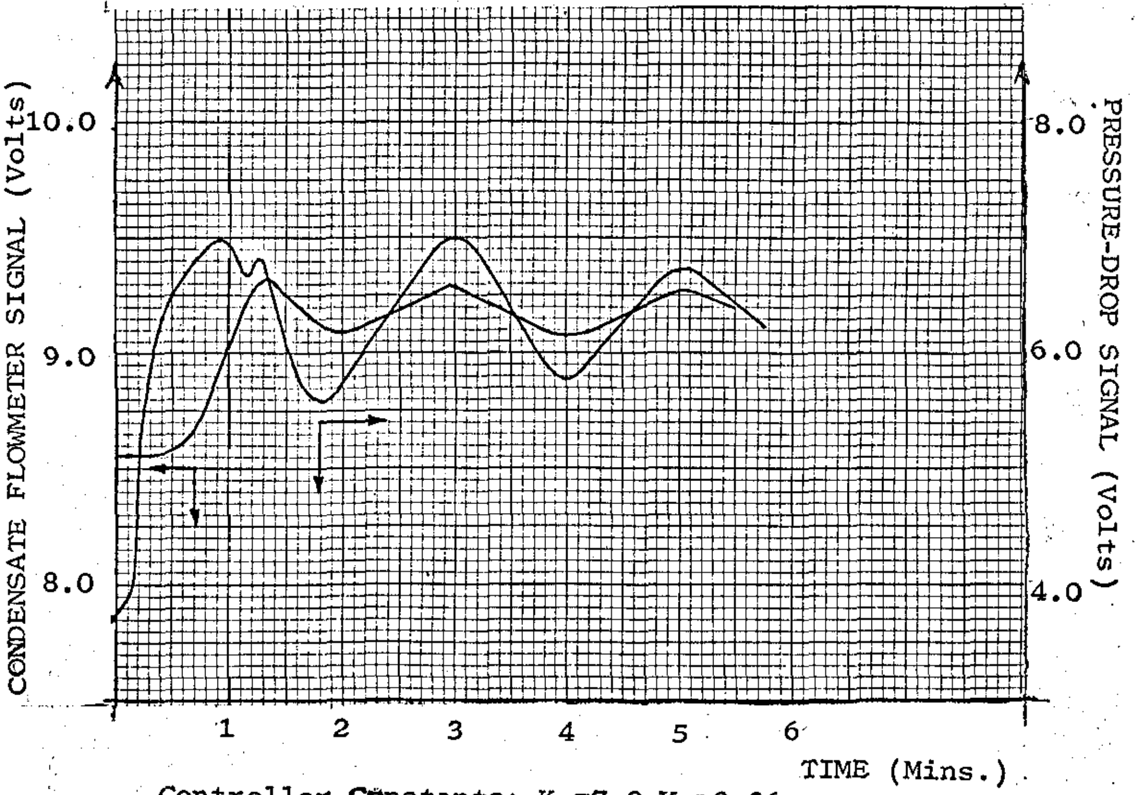
(ΔT=15 Sec.)

GRAPH 5.14(a&b) FLOW CONTROL OF BOIL-UP RATE WITH RESTRAINED STEAM VALVE



Controller Constants; $K_1=7.0, K_2=0.25$

Max. Valve Movement - 4%, and Column Stable (ΔT=15 Sec)



Controller Constants; $K_1=7.0, K_2=0.06$

Max. Valve Movement — 4%, and Column Just Unstable

(ΔT=15 Sec.)

GRAPH 5.14(c&d) FLOW CONTROL OF BOIL-UP RATE WITH RESTRAINED STEAM VALVE ensured that the flow of vapour from the top of the column was maintained at the appropriate level.

The system configuration is outlined in Figure 5.6. However, certain modifications were included in the digital cascade programme to allow for the individual characteristics of the system. The noise level of the flow loop was low, and therefore no filtering of the process signal was included. In addition, the reliability of the reproducability for the requisite steady-state reading was high, and therefore, the take-off rate at the top of the column could be specified precisely by the flow controller.

However, it was decided that a rate limitation on the output signal from the master controller should be included. Satisfactory control of the pressure loop was more involved. The pressure signal from the plant was read by the computer, and because of the noise level a moving average of the signal was used. The error signal was then computed, and if it was below a specified value, then no corrective action was taken by the controller. This was to prevent unnecessary oscillations of the control valve; in particular those oscillations that could result from a noisy signal. In addition, if the signal from the plant was above a specified value, then flooding conditions were considered to be on the point of occurring. As a result, an alarm message is sent out, and the signal to the steam valve is set to zero. In addition the output from the master controller (i.e. the set point to the secondary loop) was not allowed to exceed the pre-flooding pressure for the column. Finally, the

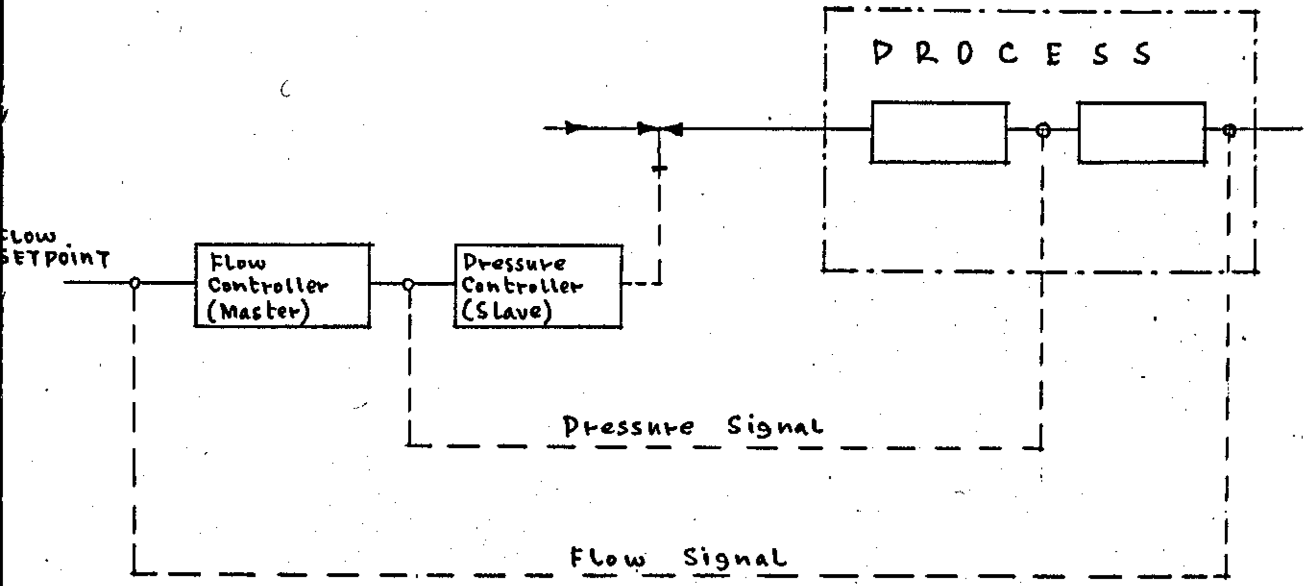


FIGURE 5.6

SYSTEM CONFIGURATION FOR THE CASCADE LOOP CONTROLLING BOIL-UP RATE

rate of valve travel was constrained to reduce the occurrence of pressure surges within the column.

As has been mentioned previously, the pressure loop is particularly sensitive to flooding conditions within the column. Together with its quick response time, this meant that its use in the cascade system, and supplemented with the steps outlined previously, it allowed the column to be operated successfully near its flooding point.

The sampling intervals for the two loops were independent. After some investigation, it was found that sampling rates of 15 secs. for the flow loop, and 2 secs. for the pressure loop handled the system satisfactorily.

The cascade controller was tuned to control the process satisfactorily using the information gathered from the open loop responses and the control of the two loops separately.

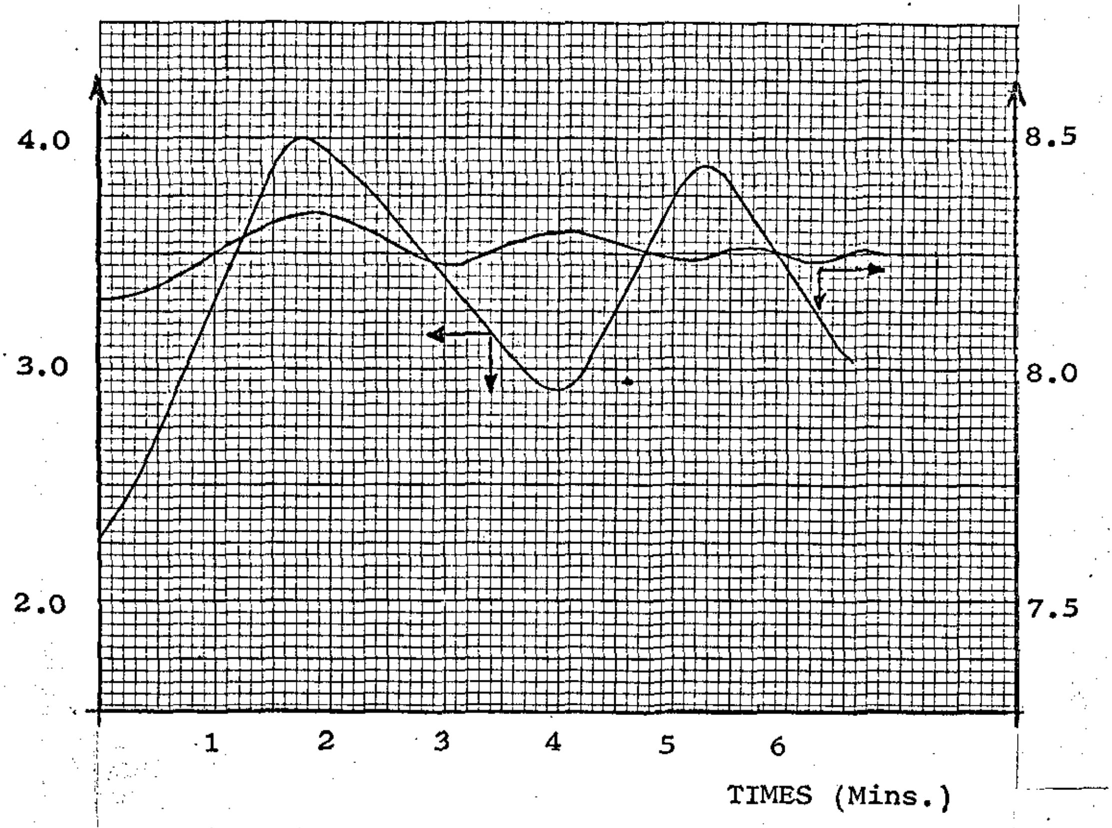
Suitable rate constraints for the outputs from the primary and secondary control loops were determined experimentally.

Typical response curves are presented in Graph (5.15).

From these results it can be seen that by use of the cascade system, the boil-up loop may be operated satisfactorily near the flooding point of the distillation column.

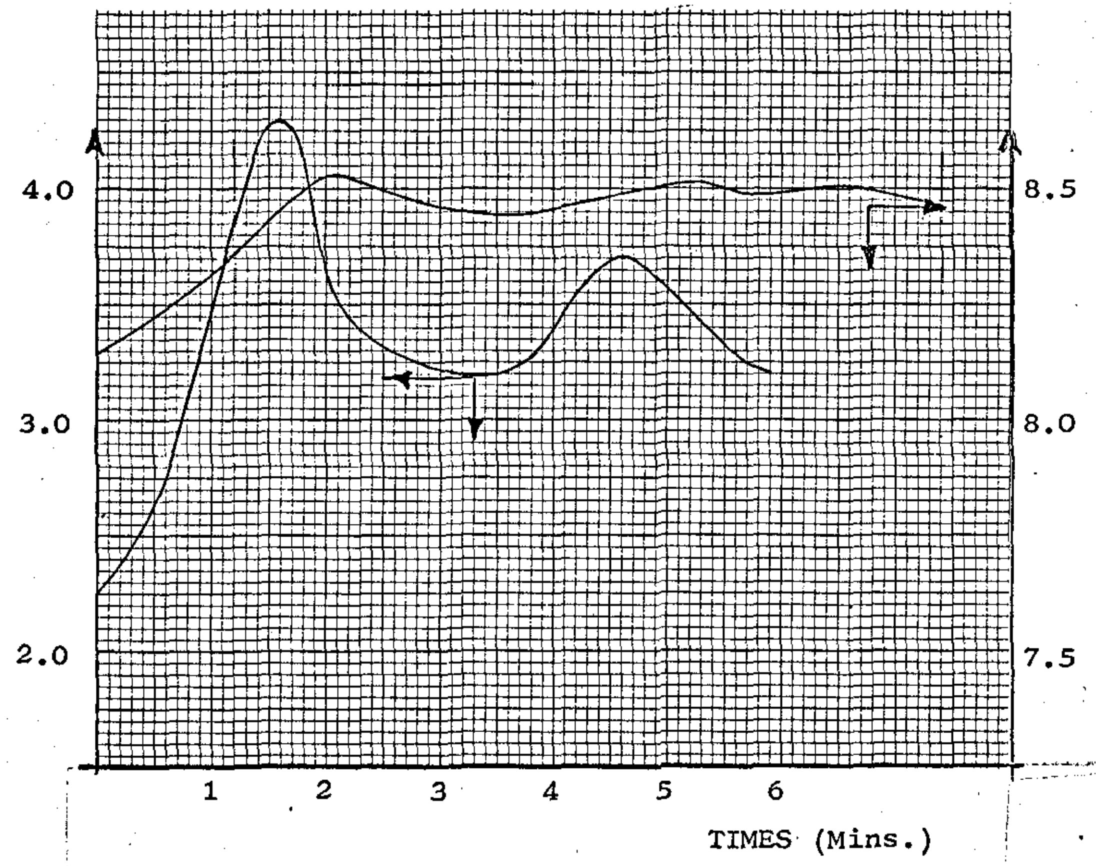
5.10.2 CASCADE CONTROL LOOPS APPLIED TO THE CONTROL OF THE COMPOSITION AND REFLUX FLOW-RATE.

The reflux loop is controlled by a valve in the product off-



Maximum Allowable Change in Valve Position = 12%

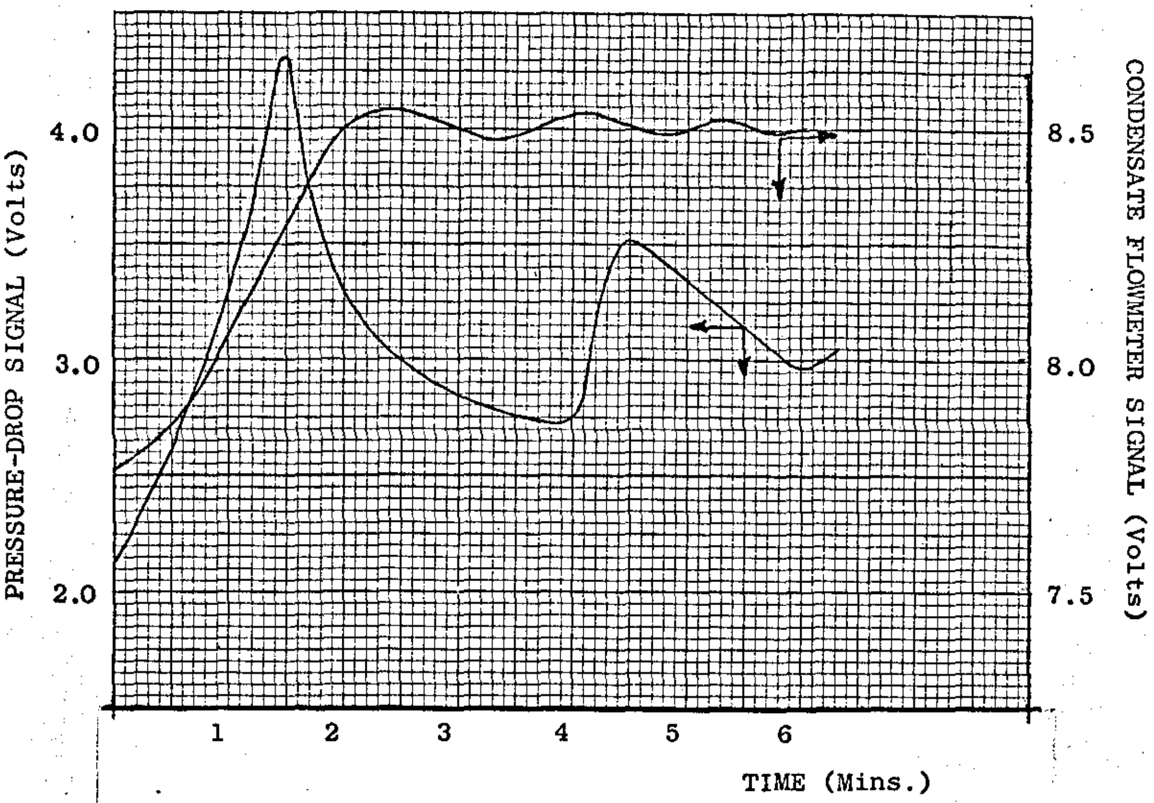
Process Stable



Maximum Allowable Change in Valve Position = 18%

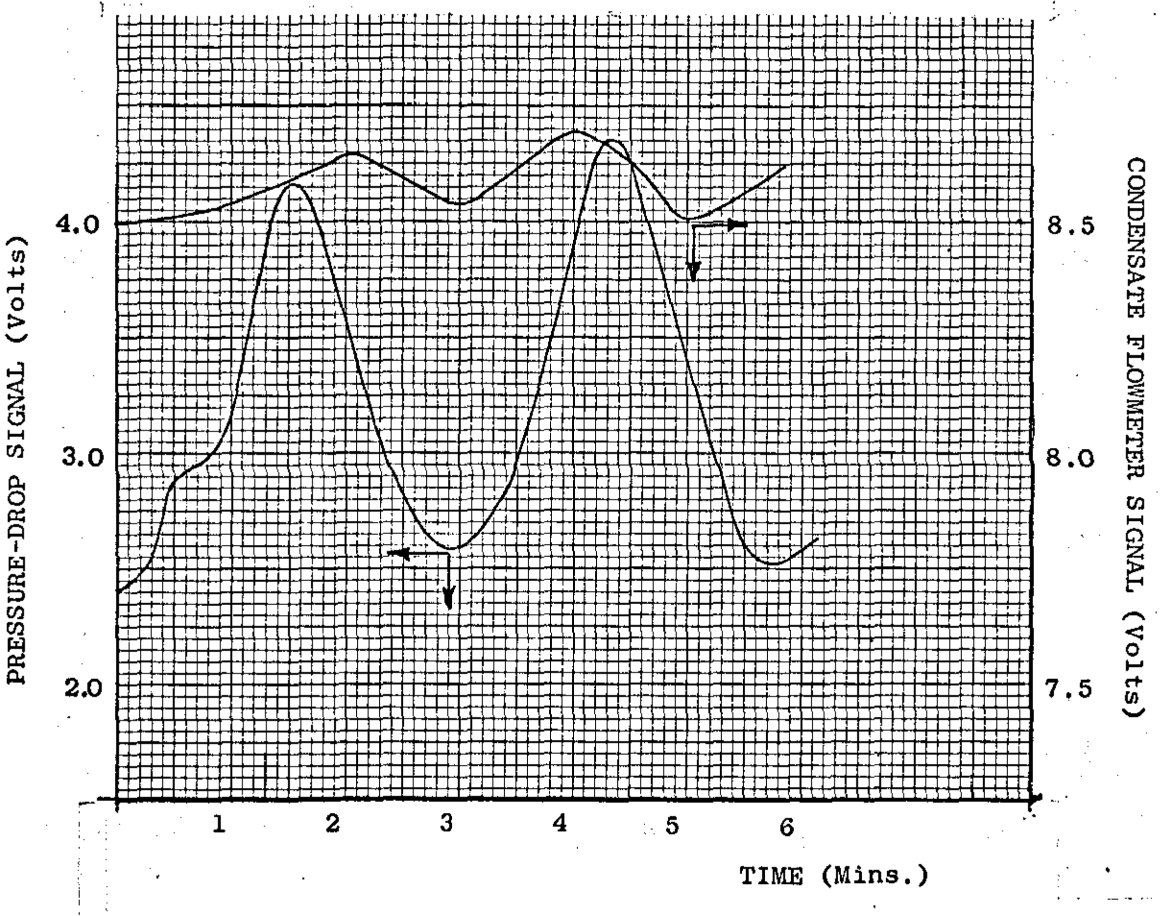
Process Stable

GRAPH 5.15(a) RESPONSE OF BOIL-UP LOOP USING CASCADE CONTROLLER



Maximum Allowable Change in Valve Position = 24%

Process Just Stable



Maximum Allowable Change in Valve Position = 36%

Process Just Unstable

GRAPH 5.15(b) RESPONSE OF BOIL-UP LOOP USING CASCADE CONTROLLER take line. For the simple control systems this was governed from the reading taken at the submerged orifice meter in the reflux return line.

If the desired top product composition were controlled instead by a composition controller governing the reflux return flow loop, then the error signal could only be updated every two minutes. Although this would give some control, it would mean that significant disturbances would be given the time to disturb the process before corrective action were taken. This is particularly true since it might be some time before the effects of the disturbances were felt by the composition controller, unless it had been situated near the source of the disturbances.

However, since the reflux return flow rate is also a primary variable for the column separation, and because it detects changes in the column dynamics much faster than the composition analyser, it is normally used preferentially. (The characteristics of the reflux return flow loop are a 'Dead - Time' of about 10 seconds, and a 'Time Constant' of about 100 seconds). However, there are certain difficulties if only single loop flow control is employed.

To perform the distillation task one must first calculate, off-line, what reflux ratio is required to produce product of the given specification. One must then specify the boil-up rate and from that the flow rate of condensate to be returned to the column. The product composition may be sampled and analysed off-line, and appropriate manual corrections made to the set-point of the reflux flow controller. However, there is no automatic feed back from the product analysis to overcome errors as they occur. In addition there is no automatic correlation between boil-up rate changes and reflux ratio.

The purpose of the cascade loop on the reflux control, is to allow the distillation operation to be carried out with constant control on the top product composition. When a step change in the reflux ratio is made, with respect to the composition, the system has a dead time of several minutes and the complete response is of the order of thirty minutes, whereas that of the flow loop is two minutes.

Hence, by using the scheme outlined in Figure (5.7), one may specify a desired top product composition, and keep the plant at that composition using the composition controller, whilst maintaining the appropriate mass balance over the column, by use of the reflux return loop, with its relatively small time constant.

The cascade control programme for this loop is relatively straight forward. The output from the chromatograph must be converted to an analysis, and the resulting composition value filtered for spurious readings. This filtered value is then used in the composition controller and the output from the controller used as the set point to the secondary controller. The secondary controller is merely a straight forward flow control loop explained in detail in Section (5.8). Satisfactory control of the reflux loop was possible using this configuration, and typical results are as indicated (Graph 5.16).

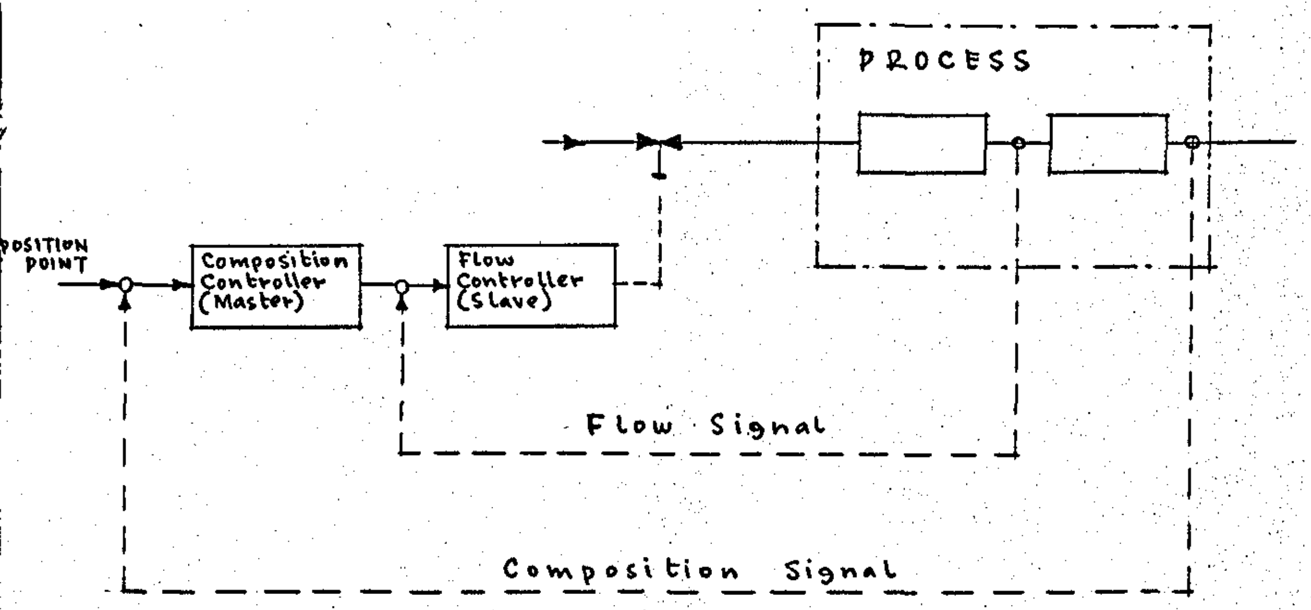
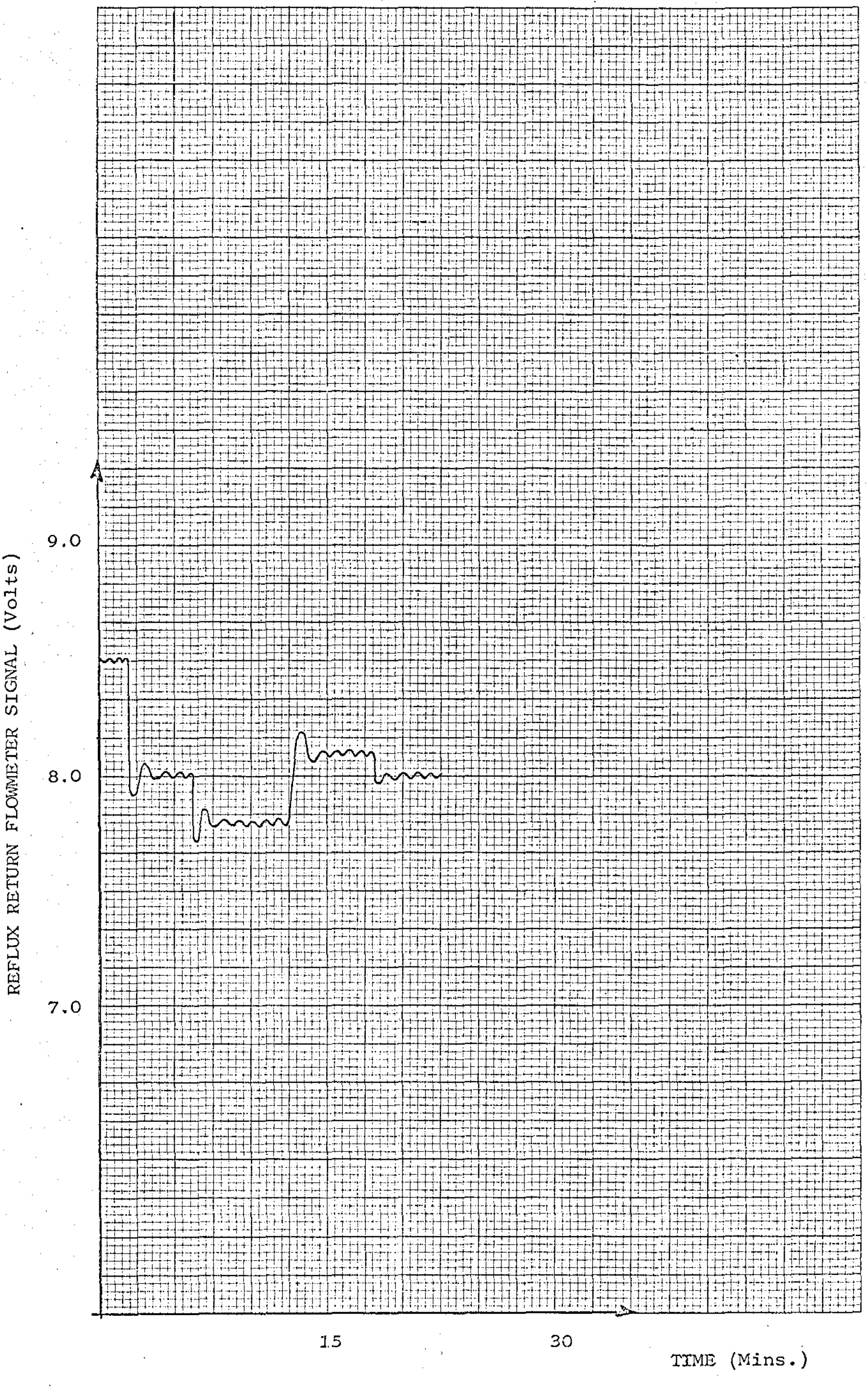


FIGURE 5.7

SYSTEM CONFIGURATION FOR THE CASCADE LOOP CONTROLLING REFLUX RETURN RATE



GRAPH 5.16 CONTROL OF REFLUX LOOP, INCLUDING COMPOSITION CONTROL

THEORETICAL ANALYSIS OF OVERALL COLUMN OPERATION AND PERFORMANCE

SECTION 6:- THEORETICAL ANALYSIS OF OVERALL COLUMN OPERATION AND PERFORMANCE.

CONTENTS Page

THEORETICAL ANALYSIS OF OVERALL COLUMN OPERATION AND PERFORMANCE

6. INTRODUCTION.

The control of a distillation column must satisfy two requirements, these are, the control of the column hydraulics, and the control of the mass transfer.

For the equipment used in the experimental work, the column hydraulics could be used to control both the boil-up and the reflux return. The equipment included an on-line chromatograph, and measurements from this were required to give an indication of mass transfer within the column. This information could be used to operate the column according to an optimal strategy.

An analysis of the problem of determining an overall operating strategy for distillation columns has been carried out by a number of authors (21, 23, 78 and 79). However, this work may be sub-divided into the two main sections of 'continuous' and 'batch' columns.

For the continuous distillation unit one may state the normal steady-state operating conditions required in the unit. If there are any changes in the feed composition or imput rate, or in the desired column products, a steady-state model may be employed to predict the new requisite steady-state operating conditions.

This technique may be modified such that the change from one steady-state condition to another follows an optimal trajectory.

The path of this trajectory will be governed by the nature of the disturbance and the definition of 'optimal' operation. (Factors which must be considered include,should the product be brought precisely to specification as quickly as possible, irrespective of the disturbances produced as a result, or would it be better if it took longer, with smaller disturbances?). To predict this trajectory, it would be necessary to employ a dynamic model of the column.

The problem of optimising the performance of continuous distillation units has been well illustrated in a paper by Archer (80).

We will consider now the operation of batch distillation columns. Although their performance is of a necessity governed by dynamic behaviour, steady-state models are often used to give an insight to batch distillation operations.

Typical of the work that has been carried out is that of Price (23) and Mitten (21), who calculated optimum reflux trajectories for batch distillation systems. Both authors ignored column hold-up and assumed steady-state conditions. In addition, Price (23) recognised that the inclusion of additional column dynamics would considerably complicate the optimization routine. A further fact which emerges from the analysis of optimal batch distillation unit operation, is that the optimum trajectory for a batch distillation task is determined solely by the reflux strategy used. With regard to the boil-up rate, it is merely necessary to hold it at its maximum permissable value.

Finally, the operation of distillation units in non-conventional modes was considered. Two techniques were investigated, cyclic operation and snatch operation. The first is based on frequent periodic controlled disturbances of the process, and the second on operating the column mainly at total reflux, where distillate removal takes the form of quick snatches of distillate from the reflux drum.

6.1 CONVENTIONAL BATCH DISTILLATION TECHNIQUES.

Conventionally, batch distillation units are operated in one of two ways. They are normally either run at a constant reflux ratio, with a variable distillate composition, or with a constant distillate composition and a variable reflux ratio.

With the former technique, the reflux ratio required to obtain the appropriate distillate is determined off-line beforehand. The distillation task is then carried out with the reflux ratio maintained at this level, and the distillate collected has an average composition of the appropriate value.

This method is often the more efficient of the two, since towards the latter part of the run, when the separation is more difficult, lower specification material may be collected. In addition, the hydraulic behaviour of the system is static, which means that operation and control of the column is relatively simple.

The alternative conventional technique is to maintain the distillate composition at the desired value. This is achieved by operating the column with a variable reflux ratio.

The desired distillate composition is specified beforehand, and may be maintained by a feed-back loop on the reflux ratio (by measuring either the top product composition or say the temperature near the top of the column). Alternatively, the reflux ratio profile may be determined off-line beforehand, and then controlled at the requisite value by a flow controller.

This technique is particularly suited to processing where separation is relatively easy. It also has the advantage that initially, the distillate may be withdrawn very quickly. However, as the separation becomes more difficult towards the end of the run, in order to maintain the requisite distillate composition, very high reflux ratios may be required. For this reason, the technique is not very suitable for distillation tasks where the separation is relatively difficult. A further problem of this method is that operation and control of the column is more difficult than when operating at a fixed reflux ratio. This is because neither the hydraulic behaviour, nor the material balance within the column, are maintained at constant values.

6.2 OPTIMISATION OF THE BATCH DISTILLATION TASK USING DYNAMIC PROGRAMMING.

Dynamic Programming is an optimising technique which is suitable for calculating the most economical conditions for the operation of a multistage batch process. This is because it relies upon decision making at each stage of the problem, rather than trying to solve the entire N-stage optimisation problem simultaneously. Hence, it is extremely convenient for optimising the operation of a batch unit. Infact, its flexibility is illustrated, in that it is possible to use other optimisation techniques for the optimisation of each stage. Finally, it can easily cope with problems which include constraints and discontinuity in the objective function. Because of the suitability of this technique, it was decided to use it to investigate the relative merits of optimal operation, compared with conventional operation.

If one considers the normal operation of a batch distillation unit, it is usual to operate it in one of two ways. These are, to either hold the reflux ratio constant and collect distillate with an average composition of the appropriate concentration, or to hold the distillate at the desired composition by increasing the reflux ratio as the run progresses.

Dynamic programming optimisation was implemented by considering that the operation of the column had been split into a number of discrete stages, during each of which, the reflux ratio was held constant. The purpose of the optimisation routine was to determine the optimum values of the reflux ratios (i.e. Optimise the vector FR_n, where $FR_n = R_1, R_2, R_3 \ldots\ldots\ldots R_n$).

An endeavour was made to compare the performance of the batch distillation unit when operated in the manner designated by these various techniques.

For the purpose of the computer routines, the consumption of steam was considered as the criterion of performance. As an alternative, the more valid experession given by the value of outgoing process streams minus the values of the incoming and utility streams, and minus the other operating costs allowing for labour etc. could have been minimized instead.

However, the consideration of just the steam consumption is sufficient to illustrate the application of dynamic programming, and to compare the column's performance when operated according to this technique with its performance when operated in any other manner.

For the purpose of the computation, it was considered that the steam consumed was a direct proportion to the amount of liquid that was converted to a vapour, irrespective of its composition or temperature.

During the initial work, the hold-up in the column and auxiliary equipment was considered to be negligible. In addition, simple equilibrium equations were used to define the behaviour of the system. Hence, the contents of the equipment were considered always to be at equilibrium. This implies that the column is always at steady state in both an hydraulic sense, and in a qualitative sense. As such, it is assumed that not only does the column react instantaneously to changes in reflux ratio, but in addition, as the distillate is removed, its contents re-adjust to the new equilibrium condition immediately. With regard to the first effect, one could increase the number of product off-take stages, with each at a different reflux ratio. Smaller disturbances would result from the reduction of the magnitude of the change in reflux ratio. However, the computation time is approximately linearly related to the number of these stages.

It is clear that theoretically, the best way to operate the column would be to have its operation optimised for an infinite number of stages with a continuously changing reflux ratio. (This differs from the fixed top product method of operation in as much as only the average distillate composition would be fixed beforehand. Infact, the distillate composition profile during the batch runs would be fixed by the computation).

6.3 USE OF THE CYCLIC TECHNIQUE IN THE OPERATION OF A DISTILLATION UNIT.

In recent years, the idea of deliberately operating certain process units in a controlled unsteady-state manner has been suggested (28). This is contary to the popular concept that continuous, steady-state operation is the ultimate goal towards which one should aim (this is illustrated by the endeavours to convert from batch to continuous processes).

Results from both simulations and experimental work have indicated that considerable benefits can accrue from imposing a repetitive discontinuity on an otherwise continuous operation (28, 29).

In general, the capacity and efficiency of any operation may be improved by unsteady-state operation. In practical terms this means a greater amount of a better quality product from a given piece of equipment.

To illustrate this with a distillation unit, the cycle operation would consist of two stages. The first is the vapour flow period, during which no liquid flows down the column, and the second is the liquid flow period, during which no vapour rises. Since no vapour is flowing during the liquid flow period, the liquid is able to flow through the normal vapour channels. For ideal operation this liquid flow must take the form of 'plug' flow.

The main effects of operating a given piece of equipment in this way, are that it has a greater capacity and more flexibility of operation. This flexibility includes the fact that the additional variables of cycle period and amplitude are now available. V.N. Schrodt et al (29) carried out a practical investigation into the benefits of cyclic operation with a 12" diameter column which had 15 plates. Results of their study indicated that during cyclic operation, not only was the possible column through-put substantially increased, but also the efficient operating range of the column increased significantly. Although their work indicated a lower maximum average plate efficiency with cyclic operation, it must be realised that the column had not been modified in any way to obtain conditions more suitable for cyclic operation.

The actual improvements possible are dependent on a variety of factors. However, results indicate that for both batch and continuous columns, with plate and packed columns, the throughput can be trebled. The seperation in plate columns show substantial improvements, whilst the changes in packed columns are usually marginal (28, 29).

However, before cyclic operation may be applied to industrial columns there are certain practical aspects that must be overcome. Probably the major problem is that the pressure drop across the column results in the bottom plates being emptied preferentially. As a consequence with the larger columns, the bottom plate has run dry before liquid flow has started on the top plates. In addition, considerable mixing takes plate on the plates. Although a cyclic column may be of a simpler design (in so much as the plates do not require down-comers etc.), the cyclic operation demands certain additional features. Firstly, between the units extra surge tanks would have to be installed, and secondly, the control aspects would create additional problems.

With this form of operation, there are two aspects of the control variable. The first is the instantaneous values of the process variables for which instruments with very quick response are essential (this is in order to maintain correct instantaneous individual material and heat flows), and the second is the average readings from the process variables (required to maintain the overall control strategy).

Finally, there is the physical problem of handling the instantaneous on/off aspect of the operations on a large scale (with respect to both design of say the condenser, and the problem of flow control).

6.4 USE OF THE SNATCH TECHNIQUE IN THE OPERATION OF A DISTILLATION UNIT.

The operation of distillation unit according to the snatch technique was another mode of operation which was examined for possible resultant improved plant performance.

This technique involved first running the unit at total reflux for a period, quickly removing some distillate from the top-product reservoir, and then returning the unit to total reflux.

It was felt that particular benefits would accrue from this technique when employed on a batch still, since in normal operation the contents were always in a non-steady state condition. It was believed that benefits could also result from operating continuous columns in this manner.

A particular advantage of this technique is that its practical application does not introduce any significant difficulties. It is merely necessary to install a reflux drum of the appropriate size, control the boil-up rate at the desired level, and to periodically empty the reflux drum quickly.

It is an extremely versatile technique, in so much as, by varying the size of the reflux drum, and the frequency with which distillate is removed, the characteristics of the technique may in turn be altered.

6.5 ANALYSIS OF UNIT OPTIMISATION BASED ON DYNAMIC PROGRAMMING.

It was decided to employ dynamic programming to investigate in some detail the potential of optimising the operation of a batch distillation unit. The investigation consisted of comparing the performance of a given piece of equipment when operated according to an optimal strategy, with its performance when operated conventionally. This was achieved by writing a programme to determine the energy requirements for a given distillation task for each of the techniques considered.

With these programmes, and considering each technique in turn, an analysis of a variety of distillation tasks was carried out.

6.5.1 STATEMENT OF THE OPTIMISATION PROBLEM.

Using the batch distillation equipment detailed previously (Section 4) a total of D moles of distillate is to be produced from a batch of B_o moles of feed stock.

The initial still and overall distillate composition must also be specified.

The distillation task is to be carried out in a series of consecutive steps, during each of which a specified number of moles are collected, and a specified reflux is used.

The reflux ratio may be considered to be represented by the vector $\underline{R}$; where $\underline{R} = R_1, R_2 \ldots . R_n$.

It is desired to predict the optimum vector :-$\underline{R}^*$. For the purpose of this study, the economy was measured as the reboiler heat requirement.

Dynamic programming (84, 85) is a recursive procedure for solving certain types of multistage optimisation problems. The method is based on certain properties of an optimal solution, as described by Bellman's principle of optimality.

In the context of the problem, this principle, may be stated as follows:-

An optimal operating programme has the property that for every stage m (where $1 \leqslant m \leqslant N$), no matter what values X_m & R_m may assume, the decisions R_1, R_2 ... R_{m-1} must constitute an optimal operating programme for the sub-process consisting of stages $1 \rightarrow m-1$ with the input X_o and output X_{m-1}.

This statement may be represented thus:

$$F_m (X_m) = \overset{\min}{R_m} \left\{ C_m (R_m, X_m) + F_{m-1} (X_{m-1}) \right\}$$

$$(m = 1, \ldots N)$$

$$\text{or} \quad F_m (x_m) = \overset{\min}{R_m} \left\{ C_m (R_m, X_m) + F_{m-1} \;\; h_m (R_m, X_m) \right\}$$

The relationships outlined in the previous equations may be represented by the following algorithmic instructions.

1. Choose an admissible value for x_m

   (a) Choose an admissible value for Rm, and compute $C_m (R_m, X_m)$, and $X_{m-1} = h_m (R_m, X_m)$

   (b) Look up the previously computed value of F_{m-1} and add to $C_m (R_m, X_m)$

(c) Repeat this process for all admissible values of R_m, and find the minimum value for the sum $C_m (R_m, X_m) + F_{m-1} (X_{m-1})$.

This is $F_m (X_m)$, and the value of R_m for which the sum is a minimum is $R^*_m (X_m)$.

2. Repeat this process for every admissible value of X_m.

3. To obtain the optimal operating programme $\underline{R}^* = (R^*_1, R^*_2 \ldots R^*_N)$, simply compute recursively the values of $R^*_m = R^*_m (X^*_m)$ and $X^*_{m-1} = h_m \left\{ R^*_m (X^*_m), X^*_m \right\}$ for m = N, N-1 ... 1 starting with $X^*_N = X_N$.

6.5.2 DESCRIPTION OF THE PROGRAMME FOR THE DYNAMIC PROGRAMMING BASED OPTIMISATION.

The optimisation routine used a steady state model of the process, with no allowance for the hold-up in the condenser or column.

For the purpose of the illustrated examples, the number of stages was confined to four, during each of which, an equal amount of distillate was removed. However, the programme was written in a manner which would allow the number of stages to be increased or decreased, depending on the process under investigation.

The programme may be considered to consist of four distinct sections. The first is the model of the process, on which all the comparative separation figures are based. As has been previously mentioned, this is based on steady-state equations.

The next section, considers the separation achieved by the column during the first stage of the distillation. Hence, it determines the performance of the column from the fixed starting point (X_o), to the variety of values of X_1 that correspond to the requisite range of values of R_1. The results of this analysis, a series of values of X_1 and R_1, are stored by the computer.

The third section considers the separation obtained from various starting points (X_{m-1}), with a variety of different reflux ratios (R_m), to give a series of corresponding values of X_m. These results are stored by the computer, in the form, X_{m-1}, X_{m-2}, R_m, for a series of values of R_m.

In addition, each time the computer goes through this section, it combines the results it obtains with those results that have been previously obtained from the earlier computations. In this way, the values of FR^*_m $\left(\text{where } FR^*_m = (R_1 + R_2 \ldots R_m)_{\text{min}}\right)$ for a series of values of X_m are obtained. Hence, the first time section three is used, a series of values of X_2 is obtained. For each of these values of X_2, the corresponding combinations of $R_1 + R_2$ are computated. In each case, the minimum value of $R_1 + R_2$ is then stored, as FR^*_2, along with the corresponding values of R_2 and X_2.

Section three may be repeated as often as is desired by the operator, and depends on the number of stages which he has specified during the formulation of the problem. However, each time section three is employed, use of the routine to determine and store the optimum values of FR_m for a series of values of X_m is included.

With the last section, although the starting point may be from a variety of values of X_{n-1}, and a variety of reflux ratios may be employed, the final still composition is fixed. For this section it is therefore possible to obtain the singular series of reflux ratios and corresponding values of X_{n-1} that satisfy the boundary conditions of the problem.

Various values of X_{n-1} have already been stored, together with corresponding values of FR^*_{n-1} and R_{n-1}. By combining the results from the final section with these results it is possible to obtain the minimum value of FR_n:- FR^*_m (where $FR^*_n = R^*_1 + R^*_2 \ldots R^*_n$).

Finally, the values of the components of the vector FR^*_n are obtained by checking back through the results that have been previously stored. In this way, values of R_m and X_m for each stage may be determined.

The operation of the programme may be considered as the performance of two tasks. The first is an analysis of the performance of the distillation unit, in order to predict the separation of the mixture that it is able to achieve, starting from a given set of initial conditions, and satisfying certain boundary conditions during the separation. The second task is an organisational task. It is to collect a series of results which are subsequently employed in completing an overall picture of the process. In this way, the operation of the distillation unit may be optimised for a given distillation task.

The usefulness of the programme is a function of how closely it represents a real process. With respect to all the initial programming work, the section concerned with the separation obtained in the distillation unit relied on steady-state equations, where a small increment of product was removed during each computation.

A model of the process was available, but the computation time required to reiterate through the model the requisite number of times in order to build up a complete overall picture of the separation problem would be extremely lengthy. However, it is this overall picture which would be required to optimise operation of the unit, using the dynamic programming technique. For this reason, the examination was carried out using the steady-state equations.

The first task which had to be undertaken was to determine the magnitude of the two incremental variables within the programme, namely the magnitude of the distillate off-take increment, and the magnitude of the step changes in distillate composition (Refer to Programme flow diagram - Figure A8).

The theoretical examination of the optimisation technique was based on analysising a number of distillation tasks, and comparing the energy requirements when the plant was operated in an optimal manner, with the energy requirements when it was operated conventionally.

The validity of the theoretical anayses based on the steady-state equations was examined by comparing the results obtained by the computations with those obtained during actual experimental runs (Section 7).

6.5.3 DETERMINATION OF THE REQUISITE MAGNITUDE OF THE INCREMENTAL VARIABLES.

There are two variables which must be examined, the first is the value of the incremental distillate off-take, and the second is the value by which the distillate composition is incremented during the trial and error search. (Refer to Programme flow diagram Figure A8).

It is apparent that both variables have a significant effect on the computational run time, and hence, it was important that their largest permissible values were employed in the computations.

The requisite magnitudes of the incremental steps were determined by an analysis of their effects on the system.

The results from the analysis of the distillate off-take increment have been detailed in Table 6.1. It can be seen that for the analysis in question, the value of ΔD should not exceed 0.3. It is apparent that the true governing criterian is the ratio between the incremental step, and the number of moles of charge left in the still at the end of the run. Hence, ΔD must be of the order of .25% of the final residue left in the still.

i.e. $\sigma = \dfrac{\Delta D}{(F - D)}$ where σ should not exceed .25%.

$R_4 = 11.5$	$FR^*_4 = 38.33$	$X_4 = 7.40\%$
$R_3 = 11.08$	$FR^*_3 = 26.83$	$X_3 = 10.32\%$
$R_2 = 8.5$	$FR^*_2 = 15.75$	$X_2 = 14.09\%$
$R_1 = 7.24$		$X_1 = 17.22\%$

TABLE 6.1 'a' Size of Distillate Increment (D) 0.1

$R_4 = 11.4$	$FR^*_4 = 38.61$	$X_4 = 7.40\%$
$R_3 = 11.19$	$FR^*_3 = 27.21$	$X_3 = 10.14\%$
$R_2 = 8.71$	$FR^*_2 = 16.02$	$X_2 = 13.88\%$
$R_1 = 7.31$		$X_1 = 17.18\%$

TABLE 6.1 'b' Size of Distillate Increment (D) 0.3

$R_4 = 10.9$	$FR^*_4 = 39.82$	$X_4 = 7.4\%$
$R_3 = 11.26$	$FR^*_3 = 28.92$	$X_3 = 10.05\%$
$R_2 = 9.75$	$FR^*_2 = 17.66$	$X_2 = 13.9\%$
$R_1 = 7.91$		$X_1 = 17.02\%$

TABLE 6.1 'c' Size of Distillate Increment (D) 0.7

TABLE 6.1 RESULTS FROM THE ANALYSIS INTO THE EFFECT OF ALTERING THE SIZE OF THE DISTILLATE INCREMENT.

(Charge to system 254 Moles of mixture containing 20% M.C.H. Removal of 50.83 Moles of Distillate to leave a residual still composition of 7.4% M.C.H.)

A similar analysis was carried out in order to determine the magnitude of the increment by which the distillate composition was reduced during the trial and error search. It is apparent that because of the large hold-up in the still, the still composition from the mass balance analysis is considerably less sensitive to changes in the distillate composition than that from the McCabe-Thiele Analysis. (Refer to the Programme Flow Diagram - Figure A8). Providing that use was made of this fact when determining the resultant composition, use of increments of the order of 0.5% resulted in negligible error. (Where 0.5% represents the reduction in the percentage of more volatile component present in the distillate after each trial and error search). The results from the computational analysis of this increment have been detailed in Table 6.2.

6.5.4 RESULTS FROM THE THEORETICAL ANALYSIS BASED ON THE DYNAMIC PROGRAMMING OPTIMISATION ROUTINE.

For this theoretical analysis, the distillation of a mixture of Methyl Cyclohexane and Toluene was examined. Charges of 28 litres to the still were considered, with compositions of either 20% or 50% Methyl Cyclohexane. The batch distillation unit was considered to have a column equivalent to 24 theoretical plates (for ease of comparison with the experimental batch unit). As has been mentioned previously, the assessment of the separation achieved within the column, was based on a steady-state model, which ignored the effect of hold-up within the column or auxiliary equipment.

The performance of the distillation unit was based on the heat requirement indicated by the reflux ratio vector. The systems

$R_4 = 11.3$	$FR^*_4 = 38.29$	$X_4 = 7.4\%$
$R_3 = 11.1$	$FR^*_3 = 26.99$	$X_3 = 10.1\%$
$R_2 = 8.65$	$FR^*_2 = 15.89$	$X_2 = 13.86\%$
$R_1 = 7.24$		$X_1 = 17.22\%$

TABLE 6.2 'a' Size of Distillate Composition Increment $(\Delta y) = 0.2$

$R_4 = 11.4$	$FR^*_4 = 38.61$	$X_4 = 7.4\%$
$R_3 = 11.19$	$FR^*_3 = 27.21$	$X_3 = 10.14\%$
$R_2 = 8.71$	$FR^*_2 = 16.02$	$X_2 = 13.88\%$
$R_1 = 7.31$		$X_1 = 17.18\%$

TABLE 6.2 'b' Size of Distillate Composition Increment $(\Delta y) = 0.5$

$R_4 = 11.65$	$FR^*_4 = 39.56$	$X_4 = 7.4\%$
$R_3 = 11.4$	$FR^*_3 = 27.91$	$X_3 = 10.05\%$
$R_2 = 8.9$	$FR^*_2 = 16.51$	$X_2 = 13.91\%$
$R_1 = 7.61$	$FR^*_2 = 16.51$	$X_1 = 17.03\%$

TABLE 6.2 'c' Size of Distillate Composition Increment $(\Delta y) = 1.0$

TABLE 6.2 RESULTS FROM THE ANALYSIS INTO THE EFFECT OF ALTERING THE SIZE OF THE DISTILLATE COMPOSITION INCREMENT.

(Charge to system 254 Moles of a mixture containing 20% M.C.H. Removal of 50.83 Moles of Distillate to leave a residual still composition of 7.4% M.C.H.)

examined were intended to indicate both difficult and somewhat easier distillation tasks, when using the two initial mixtures.

6.5.4.1 Distillation of the 20% Methyl Cyclohexane Mixture.

The separation of the 20% methyl cyclohexane mixture was analysed by considering the removal of 5.6 litres of distillate with a variety of compositions. The results of four such analyses are outlined in Tables 6.3, 6.4, 6.5 and 6.6. What one must consider, to assess the benefits of the optimisation routine, is a comparison of these analyses with those results obtained when using other techniques.

The heat requirement based on the vector FR was chosen as the measure of performance, and the results from the other analyses modified such they could be represented in this manner. The results from the various analyses have been presented in Table 6.7

It can be seen that employment of the Dynamic Programming optimisation routine results in savings when compared with the constant reflux and fixed distillate composition techniques. These savings are particularly evident with the more difficult separations, when savings of 7% and 35% respectively may be obtained.

6.5.4.2 Distillation of the 50% Methyl Cyclohexane Mixture.

For this analysis, a charge of 28 litres to the still was considered once again. However, various quantities of distillate, with a range of compositions were distilled from the charge. The result of these analyses have been detailed in Tables 6.8, 6.9, 6.10

$R_4 = 24.5$	$FR_4 = 80.17$	$X_4 = 4.98\%$
$R_3 = 19.71$	$FR_3 = 55.67$	$X_3 = 9.11\%$
$R_2 = 18.15$	$FR_2 = 34.96$	$X_2 = 12.85\%$
$R_1 = 16.81$		$X_1 = 16.15\%$

TABLE 6.3 MIXTURE DISTILLED TO GIVE 45 MOLES OF DISTILLATE WITH A COMPARISON OF 89% M.C.H.

$R_4 = 21.5$	$FR^*_4 = 75.47$	$X_4 = 5.9\%$
$R_3 = 19.5$	$FR^*_3 = 53.97$	$X_3 = 9.8\%$
$R_2 = 18.02$	$FR^*_2 = 34.47$	$X_2 = 13.5\%$
$R_1 = 16.45$		$X_1 = 16.9\%$

TABLE 6.4 MIXTURE DISTILLED TO GIVE 45 MOLES OF DISTILLATE WITH A COMPOSITION OF 86% M.C.H.

$R_4 = 17.5$	$FR^*_4 = 62.25$	$X_4 = 6.65\%$
$R_3 = 16.0$	$FR^*_3 = 44.75$	$X_3 = 9.8\%$
$R_2 = 15.0$	$FR^*_2 = 28.75$	$X_2 = 13.74\%$
$R_1 = 13.75$		$X_1 = 17.0\%$

TABLE 6.5 MIXTURE DISTILLED TO GIVE 45 MOLES OF DISTILLATE WITH A COMPOSITION OF 84% M.C.H.

$R_4 = 14.5$	$FR_4 = 53.0$	$X_4 = 7.273\%$
$R_3 = 13.9$	$FR_3 = 38.5$	$X_3 = 10.31\%$
$R_2 = 12.6$	$FR_2 = 24.6$	$X_2 = 13.74\%$
$R_1 = 12.0$		$X_1 = 16.99\%$

TABLE 6.6 MIXTURE DISTILLED TO GIVE 45 MOLES OF DISTILLATE WITH A COMPOSITION OF 80% M.C.H.

Note: For all these analyses the charge to the system was 254 Moles of a mixture containing 20% M.C.H.

Run No.	Distillate Composition	% Recovery of M.C.H.	RELATIVE TOTAL ENERGY REQUIREMENT		
			Dynamic Programming Analysis	Constant Reflux Analysis	Constant Distillate Composition Analysis
1	91.5% M.C.H.	82%	93.2	104	139.2
2	84% M.C.H.	75%	62.25	72.4	83.2
3	80% M.C.H.	70%	53.0	56.8	60.0

TABLE 6.7

COMPARISON OF ENERGY REQUIREMENTS WHEN USING DIFFERENT TECHNIQUES TO CARRY OUT THE DISTILLATION TASK.

(Initial charge in each case was 254 Moles of a mixture containing 20% M.C.H., from which 45 Moles of Distillate were withdrawn).

$R_4 = 25.0$	$FR^*_4 = 94.6$	$X_4 = 18.0\%$
$R_3 = 23.70$	$FR^*_3 = 69.6$	$X_3 = 29.78\%$
$R_2 = 23.2$	$FR^*_2 = 45.9$	$X_2 = 38.38\%$
$R_1 = 22.7$		$X_1 = 44.91\%$

TABLE 6.8 MIXTURE DISTILLED TO GIVE 112 MOLES OF DISTILLATE WITH A COMPOSITION OF 90% M.C.H.

$R_4 = 22.2$	$FR^*_4 = 77.3$	$X_4 = 21.4\%$
$R_3 = 20.7$	$FR^*_3 = 55.1$	$X_3 = 32.65\%$
$R_2 = 18.2$	$FR^*_2 = 34.4$	$X_2 = 39.85\%$
$R_1 = 16.2$		$X_1 = 46.8\%$

TABLE 6.9 MIXTURE DISTILLED TO GIVE 106 MOLES OF DISTILLATE WITH A COMPOSITION OF 90% M.C.H.

$R_4 = 12.0$	$FR^*_4 = 36.4$	$X_4 = 10.0\%$
$R_3 = 10.8$	$FR^*_3 = 24.4$	$X_3 = 19.1\%$
$R_2 = 8.1$	$FR^*_2 = 13.6$	$X_2 = 29.7\%$
$R_1 = 5.5$		$X_1 = 40.3\%$

TABLE 6.10 MIXTURE DISTILLED TO GIVE 135 MOLES OF DISTILLATE WITH A COMPOSITION OF 85% M.C.H.

Note: For all these analyses the charge to the system was 254 Moles of a mixture containing 50% M.C.H.

whilst Tables 6.11 include details of comparisons between the Dynamic Optimisation routines, and the conventional techniques. Once again, a range of savings as a result of using Dynamic Programming is indicated. It can be seen that the optimisation routine is particularly advantageous when the desired separation is more difficult. (Savings of up to 31% and 12½% are indicated when compared with constant distillate composition and constant reflux ratio respectively).

6.5.4.3 Further Theoretical Analyses Inspired by Experimental Work.

As a further development of these theoretical analyses, experimental work was carried out in order to both compare the techniques experimentally, and to compare the theoretical results with those obtained by theoretical computations. (Section 7).

Because the original computations did not allow for column hold-up, it was decided to investigate the effect of considering total charge to the system. Typical results from this analysis are indicated in Table 6.12. It can be seen that if the computation considers actual total charge, and actual system contents, the solution is quite different from that obtained when neglecting the material held in the column and reflux drum..

6.5.4.4 Inclusion of Hold-Up in the Steady-State Model.

It is apparent that the theoretical computations outlined previously have two key limitations. The first is that they made no allowance for the hold-up within the column, and the second is

Run No.	Distillate Composition	% Recovery of M.C.H.	RELATIVE TOTAL ENERGY REQUIREMENT		
			Dynamic Programming Analysis	Constant Reflux Analysis	Constant Distillate Composition Analysis
1	90% M.C.H.	80%	94.6	100.1	137.1
2	90% M.C.H.	75%	77.3	80.4	82.1
3	85% M.C.H.	90%	36.4	40.0	38.0

TABLE 6.11 COMPARISON OF ENERGY REQUIREMENTS WHEN USING DIFFERENT TECHNIQUES TO CARRY OUT THE DISTILLATION TASK.

(Initial charge in each case was 254 Moles of a mixture containing 50% M.C.H.)

$R_4 = 14.9$	$FR^*_4 = 53.8$	$X_4 = 8.3\%$
$R_3 = 13.9$	$FR^*_3 = 38.9$	$X_3 = 11.6\%$
$R_2 = 12.8$	$FR^*_2 = 25.0$	$X_2 = 14.1\%$
$R_1 = 12.2$		$X_1 = 18.3\%$
		$X_0 = 21.4\%$

TABLE 6.12 'a' Charge of 244 Moles of 21.4% M.C.H. mixture.

$R_4 = 11.6$	$FR^*_4 = 39.3$	$X_4 = 15.6\%$
$R_3 = 10.3$	$FR^*_3 = 27.7$	$X_3 = 17.85\%$
$R_2 = 9.3$	$FR^*_2 = 17.4$	$X_2 = 20.6\%$
$R_1 = 8.1$		$X_1 = 23.2\%$
		$X_0 = 26.2\%$

TABLE 6.12 'b' Charge of 275 Moles of 26.2% M.C.H. mixture

TABLE 6.12 COMPARISON OF DYNAMIC PROGRAMMING ANALYSES, WITH AND WITHOUT CONSIDERATION OF THE HOLD-UP OF MATERIAL IN THE APPARATUS.

Note: The two mixtures were distilled to give 45 Moles of distillate with a composition of 80% M.C.H.

that they made no allowance for the dynamic response of the system.

It has been mentioned previously, that a dynamic model of the experimental distillation unit has been developed by Whalley et al (81). However, this model was somewhat unwieldy for repeated reiteration within the computer routines. For this reason, its inclusion in the theoretical work, particularly the optimisation studies, was considered to be impracticable.

The limitation of zero equipment hold-up was not so difficult to overcome. Infact, the steady-state model was fairly readily extended to include an allowance for the material hold-up within the system. This modification, which allowed for variable material hold-up in the distillation column and reflux drum, was included in the optimisation programme. An analysis of a variety of distillation tasks was then carried out, using the modified optimisation programme. Typical results from this investigation have been detailed in Table 6.13. It can be seen from these results, and by comparison with the simple standard dynamic programming analysis in Table 6.13'a', that theoretically, for the systems examined, the energy requirements to achieve a given separation were always similar, and almost independent of hold-up. Naturally, for this to be true, when one makes a comparison between various distillation tasks, and if the analysis includes the effect of using equipment with differing quantities of material hold-up within the system, extreme care must be taken to ensure that an appropriate basis for comparison has been taken. This may be illustrated by reference to Table 6.13.

6.6 USE OF THE SNATCH TECHNIQUE TO CARRY OUT A BATCH DISTILLATION TASK.

The purpose of this analysis was to determine what advantages could theoretically accrue from this form of non-conventional operation. The main task of the theoretical investigation was to ascertain what separations were possible when using this technique, and also to determine the effects of altering some of the process variables.

The results from this investigation were then compared with those obtained when using the other techniques.

However, a direct comparison of the results would not have been realistic, since the theoretical investigation did not assess the energy requirements of the technique.

The purpose of the theoretical analysis was merely to give an insight to the effects of operating a batch distillation unit according to the snatch technique. To do this, programmes based on both packed and plate columns were developed. These programmes, which always considered that steady-state conditions existed prior to the removal of a snatch of distillate, could be used to investigate the effects of manipulating a number of process variables.

The variables that had to be considered were hold-up in the reflux drum and in the distillation column, and the size of the snatches of distillate. To examine the effects of changing these variables,

$R_4 = 14.9$	$FR_4^* = 53.8$	$X_{s_4} = 8.3\%$
$R_3 = 13.9$	$FR_3^* = 38.9$	$X_{s_3} = 11.6\%$
$R_2 = 12.8$	$FR_2^* = 25.0$	$X_{s_2} = 14.1\%$
$R_1 = 12.2$		$X_{s_1} = 18.3\%$
		$X_{s_0} = 21.4\%$

TABLE 6.13 'a' Charge to System 244 Moles of a 21.4% M.C.H. mixture (no allowance for Hold-Up)

$R_4 = 16.0$	$FR_4^* = 53.5$	$X_{s_4} = 8.3\%$
$R_3 = 13.5$	$FR_3^* = 37.5$	$X_{s_3} = 12.22\%$
$R_2 = 12.4$	$FR_2^* = 24$	$X_{s_2} = 15.54\%$
$R_1 = 11.6$		$X_{s_1} = 18.6\%$
		$X_{s_0} = 21.4\%$

TABLE 6.13 'b' Charge to System 264 Moles of a 24.3% M.C.H. mixture (Hold-Up of 21 Moles in the Reflux Drum)

$R_4 = 15.8$	$FR_4^* = 53.0$	$X_{s_4} = 8.3\%$
$R_3 = 13.6$	$FR_3^* = 38.1$	$X_{s_3} = 12.35\%$
$R_2 = 12.65$	$FR_2^* = 24.5$	$X_{s_2} = 15.6\%$
$R_1 = 11.85$		$X_{s_1} = 18.8\%$
		$X_{s_0} = 21.4\%$

TABLE 6.13 'c' Charge to System 275 Moles of a 25% M.C.H. mixture (Hold-Up of 21 Moles in the Reflux Drum and 11 Moles in the Column)

TABLE 6.13 COMPARISON OF THREE DYNAMIC PROGRAMMING ANALYSES, IN EACH OF WHICH, 45 MOLES OF DISTILLATE WERE REMOVED FROM IDENTIAL INITIAL STILL CONTENTS.

it was decided to consider a constant total charge to the process and the removal of a given amount of distillate. For the theoretical analysis, with a steady-state conditions between snatches, the effects of altering these variables would be indicated by the distillate composition. However, for a real process, the further variable of total run time must be considered. If a constant run time were allowed for all the analyses, the size of the distillate snatch would actually be related to the frequency with which samples were taken, and hence, the time allowed for the column contents to return to steady-state. However, a final observation which must be made, is that the size of the snatch is directly related to the magnitude of the disturbance it causes to the column contents. Therefore, with smaller snatches, less time is required to return to steady-state.

Obviously, for a real distillation task, in a real batch distillation unit, there is an optimum set of these variables. It will depend on the material charged into the still, the requisite amount of distillate and its composition, and the physical characteristics of the distillation unit.

6.6.1 SNATCH OPERATION APPLIED TO A DISTILLATION UNIT WHICH INCLUDED A REFLUX DRUM WITH A SIGNIFICANT HOLD-UP.

A programme was developed to carry out the mass balance analysis of a batch distillation operation in a 24 plate distillation unit. As has been mentioned previously, for the purpose of the theoretical analysis, the contents of the distillation unit were considered to be at steady-state before a snatch of distillate was removed.

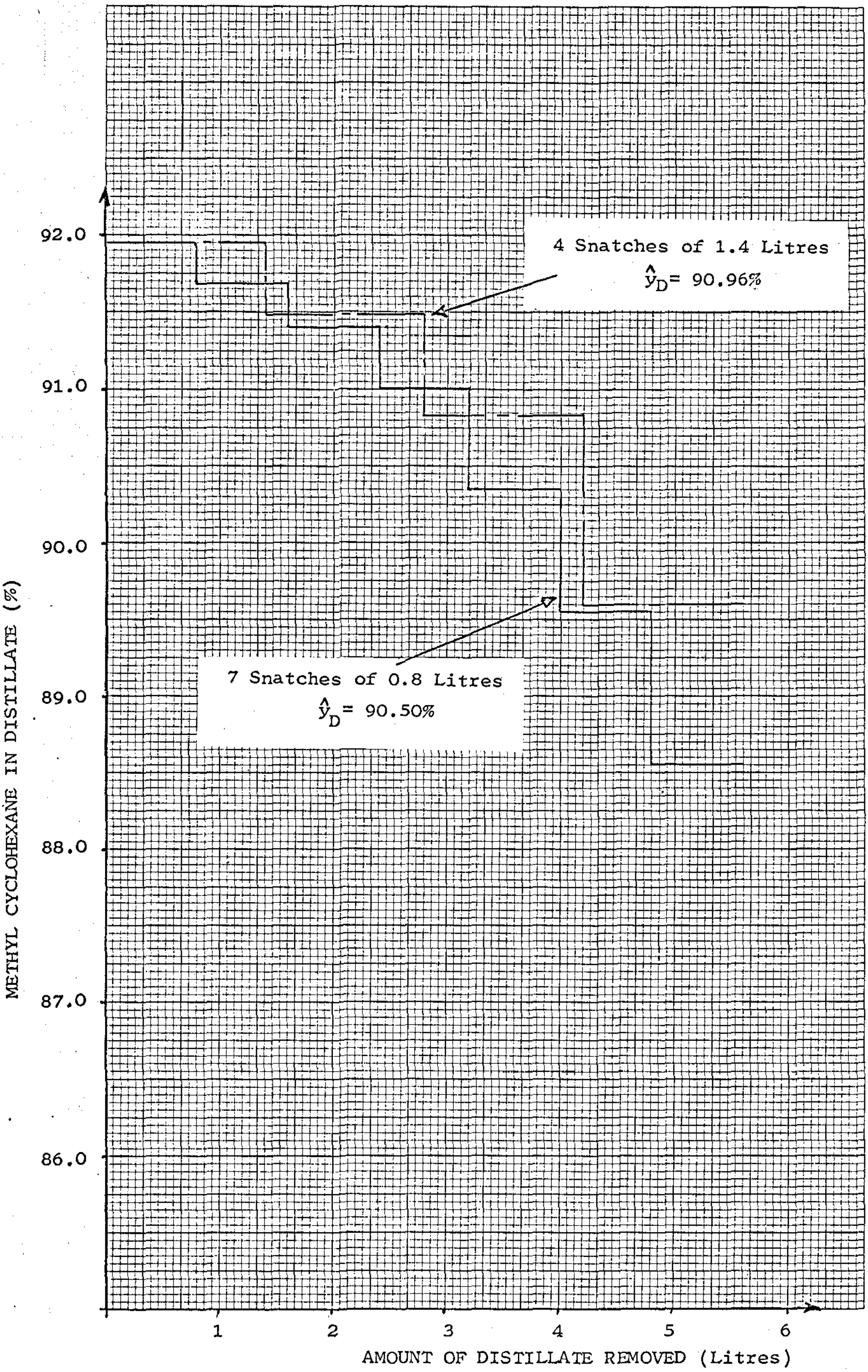
It was decided to investigate the effects of altering three variables in the programme. These variables were:-

1. Size of Snatch
2. Hold-Up in the Reflux Drum
3. Composition of Charge to the Unit

For the purpose of the investigation, the collection of 5.6 litres of distillate from a 31 litre sample was considered. For the general analysis of the problem, a sample with a composition of 24.3% M.C.H./75.7% Toluene was considered. This corresponded to a charge in the experimental rig which resulted in a still composition of 20% Methyl Cyclohexane at total reflux, and was the charge which was employed during a number of the experimental investigations into batch distillation.

The first variable that was investigated, was the effect of altering the amount of distillate removed at each snatch. If no account were made of the dynamics of the system, from intuitive reasoning, one would expect that provided snatches smaller than the hold-up in the reflux drum were always taken, the best separation would be achieved by larger snatches.

As is demonstrated in Graph 6.1, the theoretical analysis confirmed these conclusions. However, it can be seen that with a significant hold-up in the reflux drum and column (5% in the reflux-



GRAPH 6.1 SNATCH COMPOSITION PROFILES FROM THEORETICAL ANALYSIS

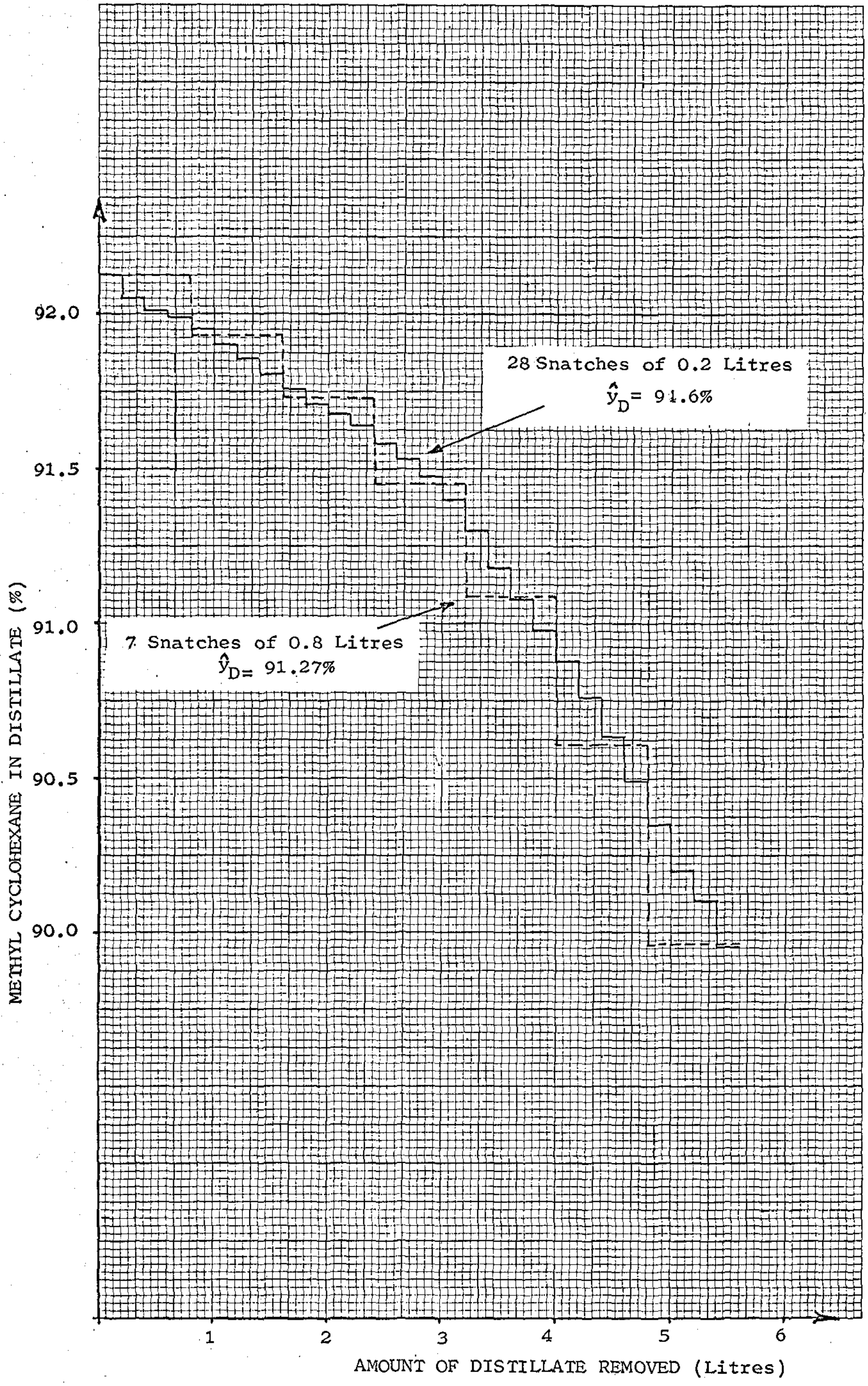
drum, 8% in the column, totalling 13% in all), the effects of varying the snatch size is not particularly significant. However, a further analysis where the hold-up in the reflux drum was smaller (2.5%), resulted in a more significant difference (Graph 6.2). These differences are best illustrated by examining the effects of altering the size of the reflux drum.

It is obvious, that for a given charge, and using a given snatch size, the separation that may be achieved is directly related to the hold-up in the column and the reflux drum. This is confirmed by reference to Graph 6.3

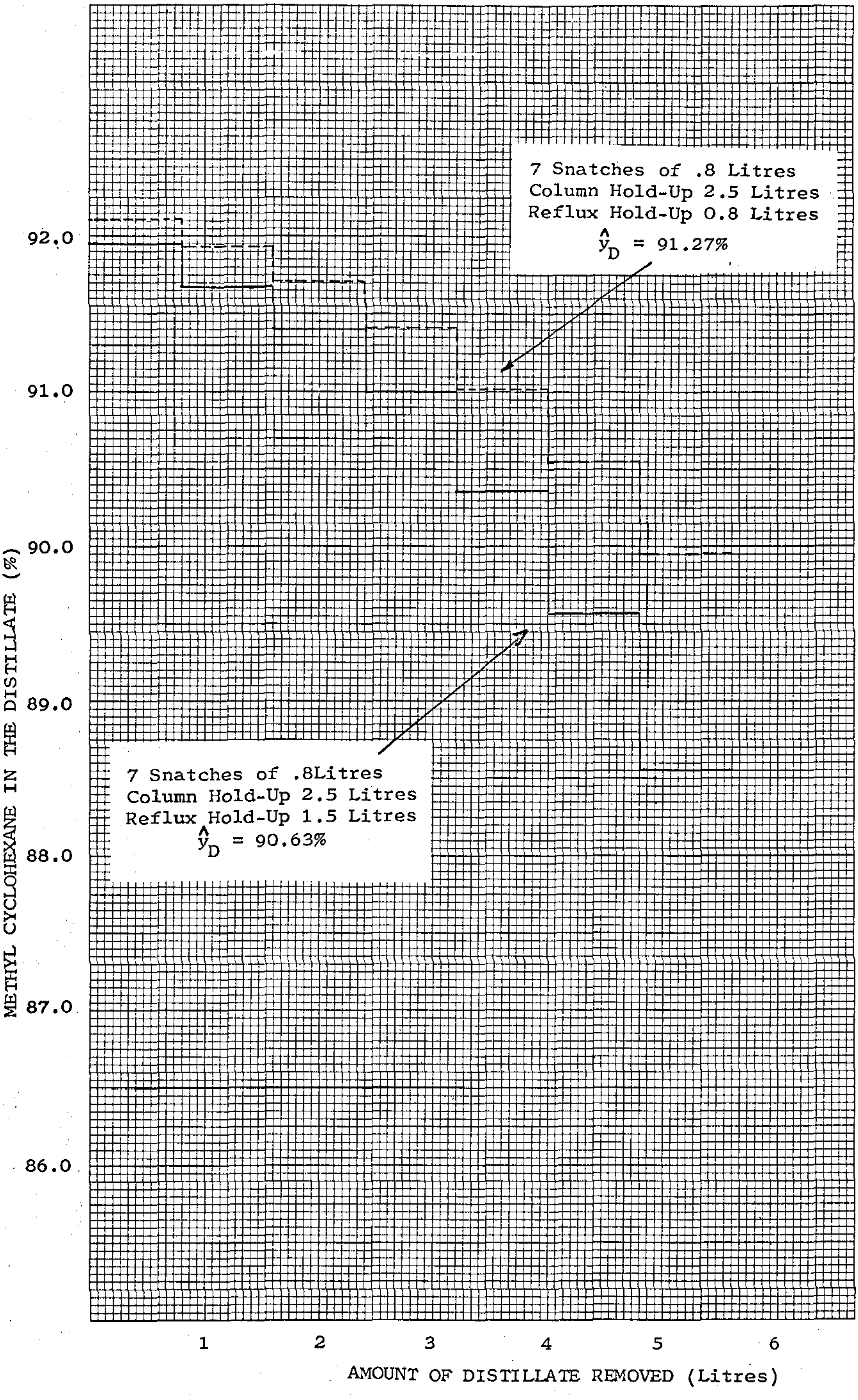
Finally, the previous investigations were repeated using a different charge to the distillation unit. The purpose of these theoretical runs was to investigate whether the composition of the original sample had a significant effect on the results achieved by altering the snatch size and the size of the reflux drum.

This theoretical analysis was carried out by repeating the previous computations but with the composition of the charge into the distillation unit altered from 24.3% M.C.H. to 50% M.C.H.

The investigation did not indicate any significant changes to the effects of altering the other variables. However, in practice, the change in composition could result in significant differences in the equilibrium characteristics of the system in question. This in turn could be an important factor in choosing the appropriate processing characteristics.



GRAPH 6.2 SNATCH COMPOSITION PROFILES FROM THEORETICAL ANALYSIS



GRAPH 6.3 EFFECT OF REFLUX DRUM HOLD-UP ON DISTILLATE COMPOSITION ANALYSIS

6.6.2 ASSESSMENT OF RESULTS FROM THEORETICAL SNATCH OPERATION AND COMPARISON WITH REAL PROCESS.

As has been stressed previously, all these results made no allowance for the dynamics of the system, but instead considered that steady-state conditions prevailed before a snatch of distillate was removed from the unit.

Purely from an analysis of the theoretical results, it would appear that the ideal conditions would be to have a small hold-up in the reflux drum, and to snatch distillate such that the reflux drum was emptied by each snatch.

By having a smaller reflux drum, one would gain increased separation in the distillation unit (Graphs 6.2 and 6.3), and the system would tend to take less time to reach steady-state. (In so much as the non well-mixed reflux drum had only a small hold-up). The effects of removing the total contents of the reflux drum would be two-fold. The first is that as much as possible of the Methyl Cyclohexane rich product would have been removed at each step, thus utilising fully the energy that has been expended to achieve this. However, the second is that because of poor mixing in the reflux drum, one would expect the size of the disturbance to be proportional to the time required for it to return to steady-state.

It is apparent that it would be desirable for each snatch to empty the reflux drum. The remaining theoretical variable of reflux drum size would be governed by the practical arrangements of actual average distillate composition required, the ease with which the components of the mixture may be separated, and the frequency with which snatches are removed.

To optimise on size of reflux drum for a given distillation task, one would require a dynamic model of the system. This would be a model of a batch distillation unit which could represent the system endeavouring to reach steady-state under total reflux conditions, but being subjected to the periodic disturbances by the removal of snatches of distillate. Naturally, the most significant changes in the top-product composition would take place immediately after a snatch of distillate had been removed. The top product composition would then tend towards its steady-state value, but the composition profile would take the form of an exponential decay.

6.6.3 SNATCH OPERATION APPLIED TO A DISTILLATION UNIT IN WHICH THERE WAS SIGNIFICANT HOLD-UP ONLY IN ITS COLUMN.

A programme was developed to carry out the mass balance analysis of a batch distillation operation in a 24 Plate distillation unit. The programme was similar to that mentioned previously (Section 6.6.1), except in that each snatch of distillate emptied liquid from the column plates, instead of from a reflux drum.

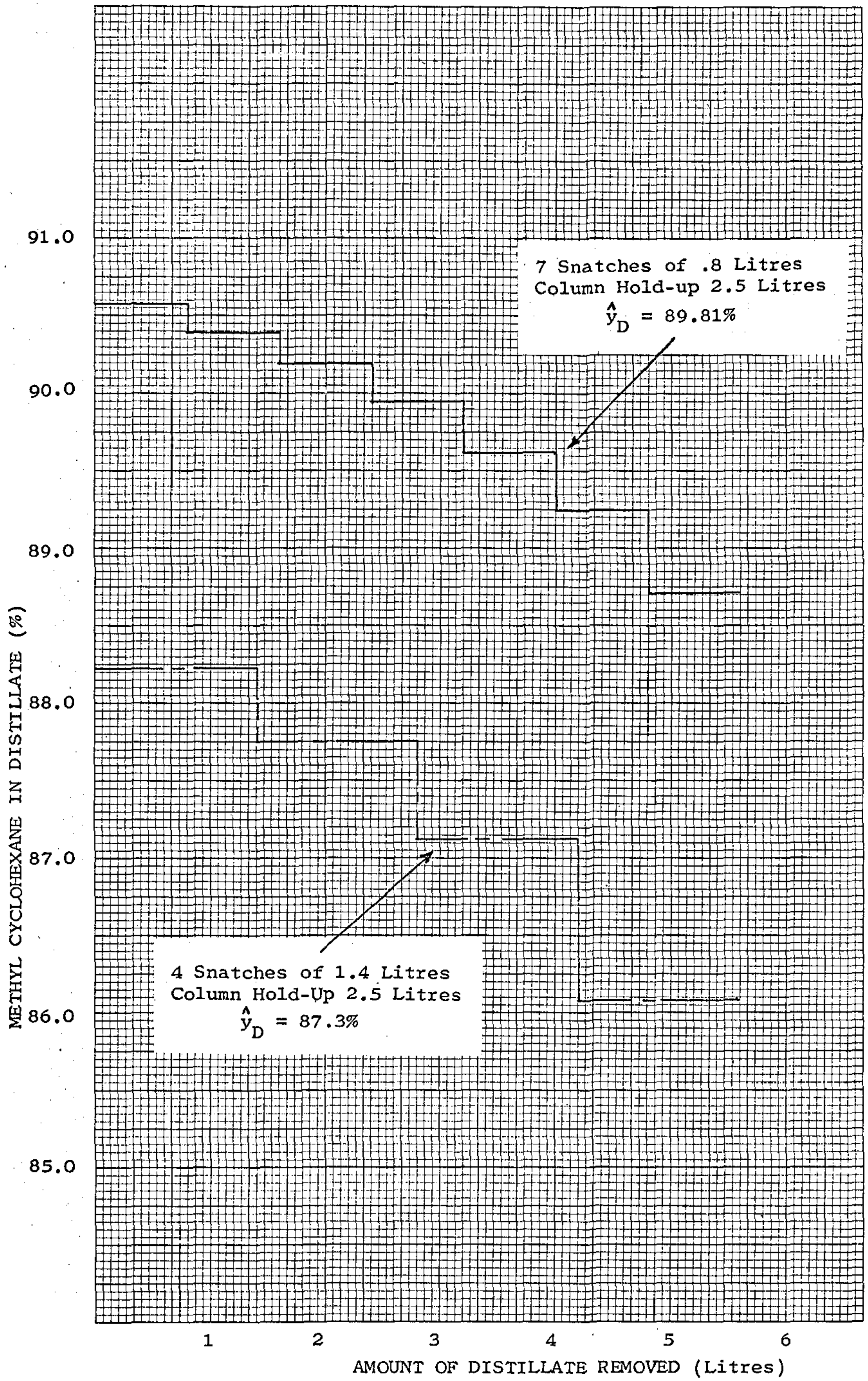
The main object of this analysis was to investigate the effects of varying the snatch size, and to do this a distillation task similar to that mentioned previously (Section 6.6.1) was once again investigated.

Intuitively, it could be assessed that larger 'snatch' sizes, would result in worse separation. This intuitive reasoning was confirmed conclusively by the theoretical analysis (Graph 6.4). Infact, when larger snatches of distillate were removed, the separation achieved was quite poor.

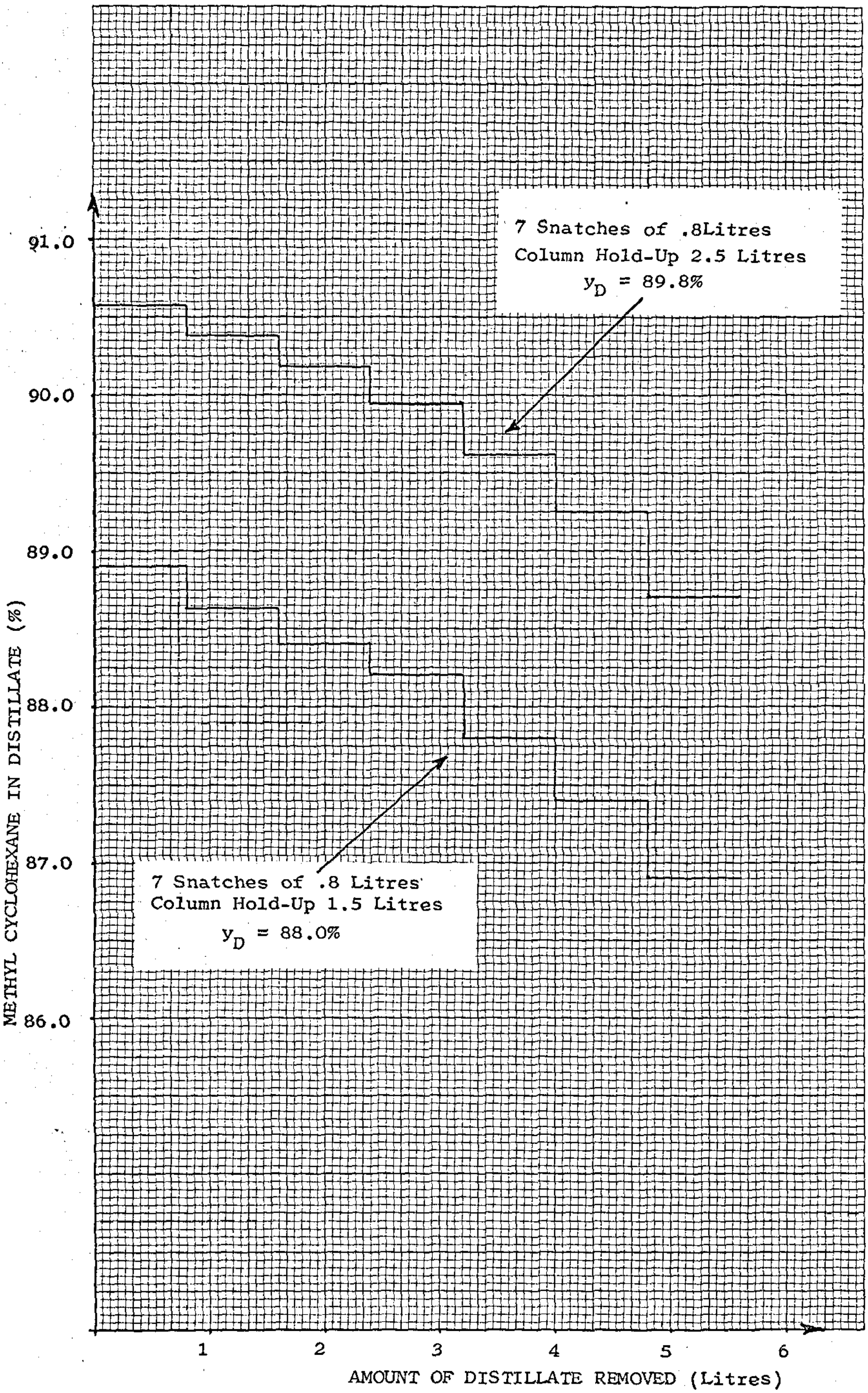
A further variable that was investigated was the effect of altering the magnitude of the column hold-up. Once again the results from the theoretical analysis confirmed those obtained by intuitive reasoning. For a given snatch size, the quality of the separation was directly related to the column hold-up. (Graph 6.5).

Finally, the process was examined for any significant effects resulting from a change in the charge composition. For the purpose of this theoretical analysis, the charge composition was altered from 24.3% M.C.H. to 50% M.C.H. By comparing the results of the previous analysis into snatch size (Graph 6.4) with the analysis when starting with a still composition of 50% Methyl Cyclohexane (Graph 6.6), it can be seen that different composition profiles result. It is apparent from these results, that if one wished to consider the effect of altering the charge composition thoroughly, one would have to consider the composition profile of the column contents throughout the batch distillation operation.

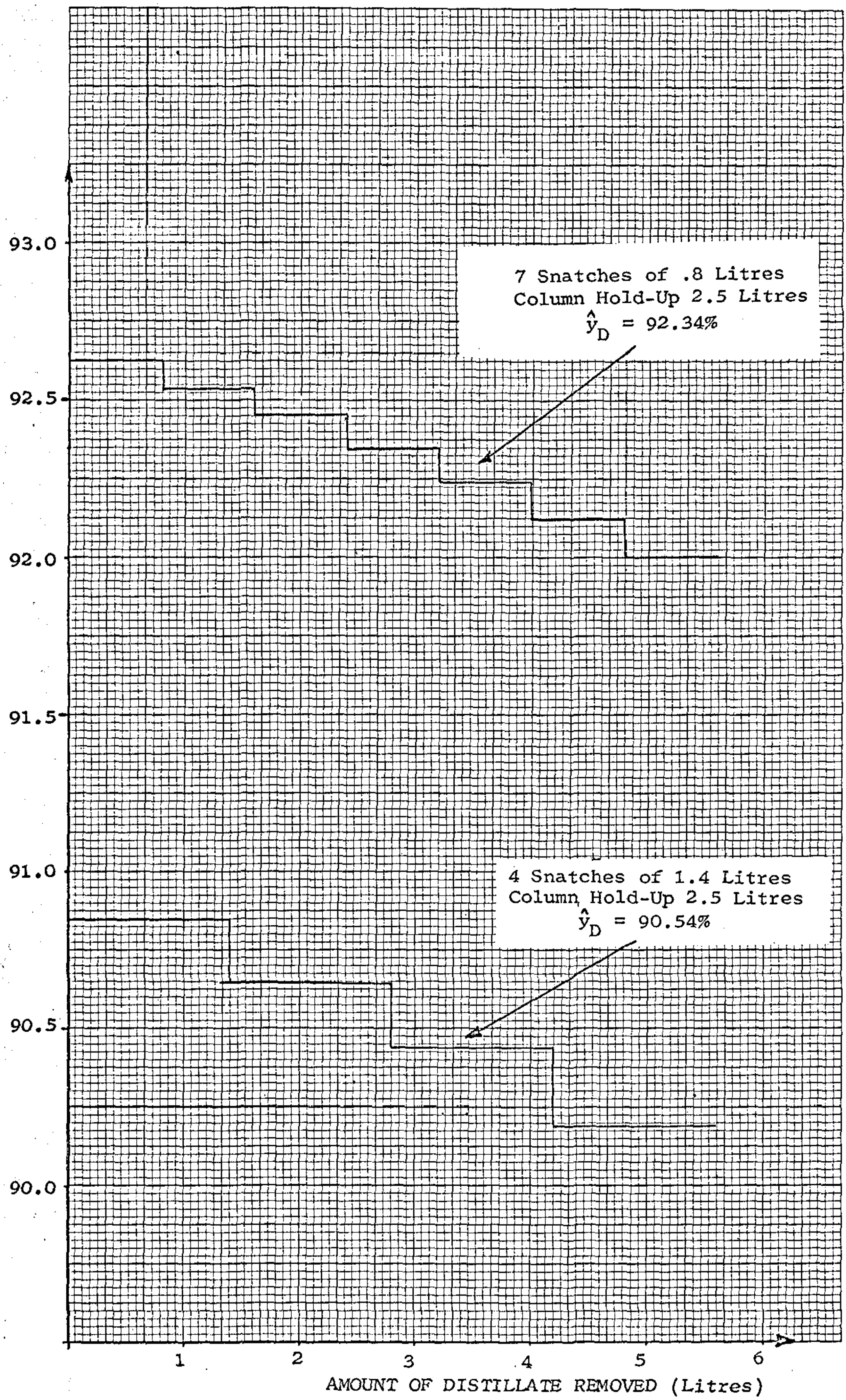
The characteristics of the mixture could be such that over a number of upper plates there is little change in composition when one composition is distilled, but when a second composition is employed these plates hold liquids of widely varying composition.



GRAPH 6.4 IDEAL SNATCH COMPOSITION PROFILE WITH ZERO REFLUX DRUM HOLD-UP



GRAPH 6.5 EFFECT OF COLUMN HOLD-UP ON DISTILLATE COMPOSITION PROFILE



GRAPH 6.6 EFFECT OF COLUMN HOLD-UP ON DISTILLATE COMPOSITION PROFILE

6.6.4 ASSESSMENT OF RESULTS FROM THE THEORETICAL SNATCH OPERATION AND COMPARISON WITH A REAL PROCESS.

With a real system, one would have to consider the time required for the unit to reach steady-state. Naturally, units with small hold-ups would tend to require a shorter time.

The use of small snatches is the most suitable, whether considering dynamic or steady-state behaviour.

However, generally, when employing the snatch technique, it would be advantageous to arrange for an appropriate hold-up in the reflux drum from which snatches of distillate could be removed.

Although, the top product composition would drop, a high overall distillate composition could be maintained with a reduction in the requisite energy required. (The system would have been allowed less time to reach, and be further from, steady-state).

EXPERIMENTAL INVESTIGATION INTO OVERALL COLUMN OPERATION STRATEGIES

SECTION 7:- EXPERIMENTAL INVESTIGATION INTO OVERALL COLUMN OPERATION STRATEGIES.

CONTENTS

EXPERIMENTAL INVESTIGATION INTO OVERALL COLUMN OPERATION STRATEGIES.

7. INTRODUCTION

The experimental analysis of batch distillation was carried out in the distillation unit specified earlier (Section 4). The mixture employed was Methyl Cyclohexane and Toluene.

Generally, the equipment was charged with material such that at total reflux the still contents were 28 litres of 20% Methyl Cyclohexane mixture.

In as much as the column hold-up was approximately 1.5 litres, and that of the auxiliary equipment 1.5 litres, the total charge to the system was 31 litres. The composition profile within the column resulted in the fact that the feed to the unit had an actual composition of 24% Methyl Cyclohexane.

Throughout the experimental work, the boil-up rate was fixed at a constant level of 750 cc/min., which was a little below the column's flooding point. It was controlled at this level by the use of either the analogue of digital controllers. The reading from the submerged orifice meter in the condensate line was used as the measured process variable in this control loop. However, to obtain the actual boil-up rate this reading had to be corrected to allow for the vapour condensed by the sub-cooled reflux. (The temperature of the liquid in the reflux return line was maintained at 40°C. by manual control of the cooling water flow-rate).

The reflux return flow rate was controlled by the valve in the distillate product line. Two techniques were used to control the reflux ratio. The first used the reflux return flow rate, and the second used measurement of the product collection rate. The inadequacy of merely relying on the reflux return flow rate measurement has been detailed elsewhere (Section 4.7). However, the reading may be used to control the system approximately, whilst precise determination of the reflux ratio is dependent on measurement of the distillate collection rate (Appendix 2).

The on-line chromatograph (Section 4.3) was used to measure the composition profile of the distillate and still contents as the experimental run progressed. The separation achieved during each experimental run could be determined by examination of these profiles. The performance index of the distillation technique employed was judged by this 'separation' profile and the duration of the experimental run.

Since the purpose of the experimental work was to compare the various distillation techniques, as well as to obtain a comparison between actual and theoretical results, various techniques were all employed carrying out similar separation tasks.

7.1 EXPERIMENTAL INVESTIGATIONS USING THE CONSTANT REFLUX RATIO TECHNIQUE

The initial experimental work was to determine the separation that could be achieved within the column when using the conventional distillation techniques.

For the purpose of the investigation into the fixed reflux ratio technique, the work took the form of running the batch distillation unit (described in Section 4) at a fixed reflux ratio, until the appropriate amount of distillate had been removed.

Since the endeavour of the experimental work was to compare the theoretical results with those from the practical investigation, during the initial runs, the reflux ratio employed was that which had been predicted by the off-line computation (Section 6.1). It was therefore merely necessary to specify the boil-up rate, and fix the reflux flow return at the appropriate rate. (Naturally, consideration must be made here of the effect of returning sub-cooled reflux to the top of the column). Since calibration of the condensate flowmeter could be carried out more precisely, it was best to check the reflux ratio by using this reading and that of the distillate collection rate. Appropriate correction could then be made to the reflux return loop if necessary. This arrangement resulted in adequate control of the reflux ratio as the experimental observations will indicate.

A typical experimental investigation was the separation of a 20% Methyl Cyclohexane/80% Toluene mixture, to give 5.6 litres of distillate with a predicted average composition of 91.5% M.C.H. (The results from the theoretical computation have been detailed in Section 6.5.4.1. - Table 6.7).

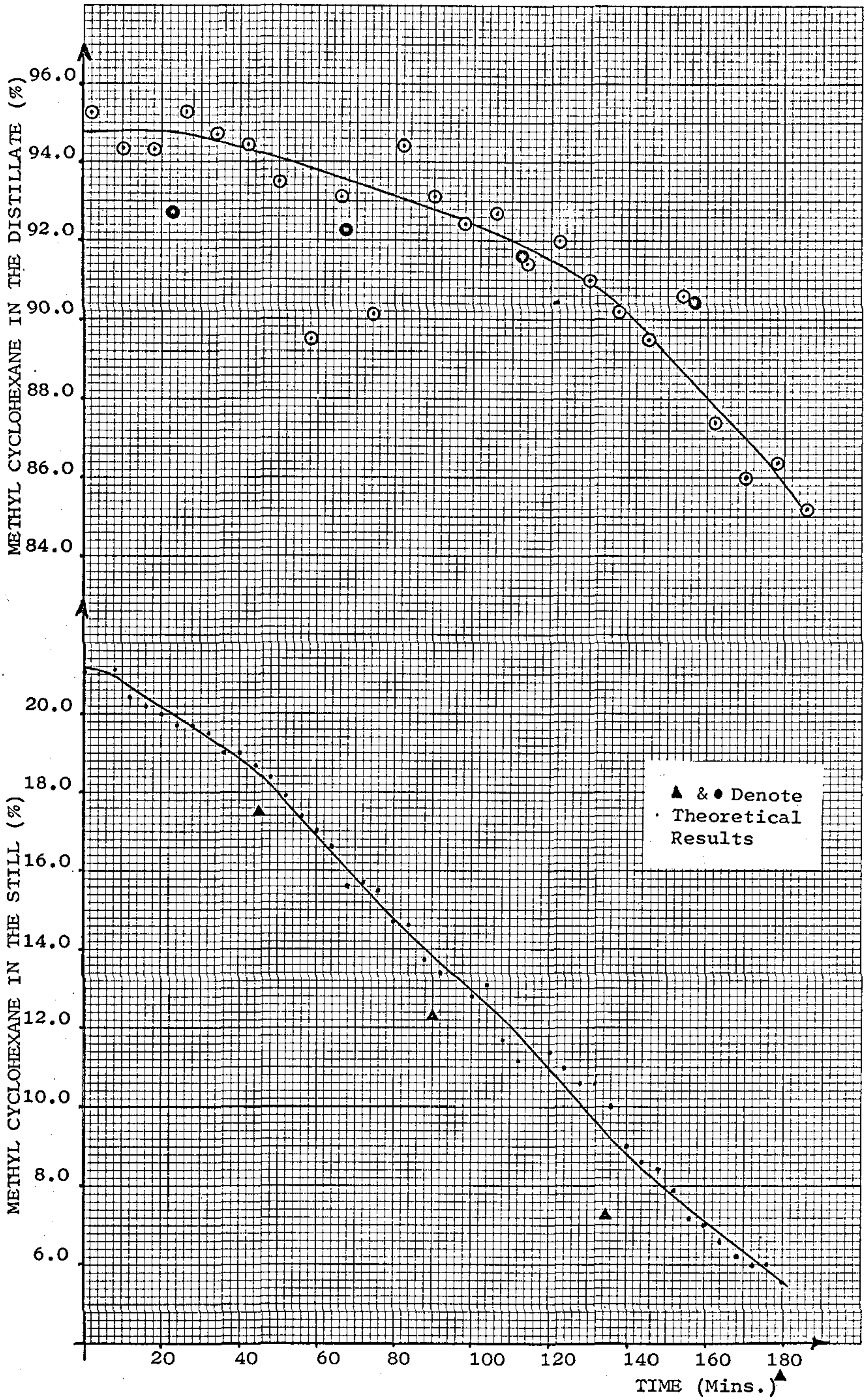
The apparatus was run in the manner detailed above, and the performance of the separation monitored by continuously measuring the composition of the still contents and the product from the top of the column. This was achieved by using the on-line chromatograph with its automatic sampling device. In addition, at the end of the run, the average distillate composition was also determined.

The results from this work were analysed by plotting the theoretical and actual composition profiles on the same graph (Graph 7.1).

By referring to these results, it may be seen that the theoretical separation is somewhat better than that achieved by the experimental work. This fact is made particularly apparent by considering in full, the information from the still composition profile. There are two factors which must be considered. The first is that as the run progresses the theoretically 'inert' material hold-up has a lower requirement of Methyl Cyclohexane. This excess of Methyl Cyclohexane is feed-stock for the still, which, in conjunction with the effects of the dynamic behaviour of the distillation unit, results in an enriched still composition. The final stage of the theoretical distillation task is therefore never undertaken during the practical studies. (This point is underlined by some of the results from the studies of Snatch Distillate, where a complete change in the column profile occurs towards the end of certain runs. - Section 7.4).

7.2. EXPERIMENTAL INVESTIGATION USING THE CONSTANT DISTILLATE COMPOSITION TECHNIQUE

Once again the purpose of the experimental work was to compare



GRAPH 7.1 THEORETICAL & ACTUAL COMPOSITION PROFILES FOR CONSTANT REFLUX

the experimental results with those which had been obtained from the theoretical computations.

For the purpose of this comparison, the separation detailed in Table 6.7 to produce distillate with a composition of 91.5% Methyl Cyclohexane has been considered.

The off-line computation predicted the reflux ratio profile required to achieve the requisite separation, and it was this profile which was employed to compare the actual and theoretical results.

To carry out the experimental work, the still was first charged with the appropriate mixture (outlined in the previous sections). The technique for controlling the boil-up rate has also been explained in the preceeding sections. Control of the reflux ratio was somewhat more involved.

Initially the reflux ratio was fixed at the value indicated by the off-line computation, but as the distillate was collected, and the distillate composition dropped, the reflux return flow controller was trimmed to maintain the appropriate distillate composition. This was straight forward, since the composition of the distillate was monitored by the on-line chromatograph, and its results analysed by the computer which printed out the compositions of the samples as they were analysed.

The boil-up rate had been held constant throughout the experimental run, and the rate at which the distillate was collected was monitored continuously. It was therefore possible to determine the reflux ratio that had been required to maintain the desired top product composition.

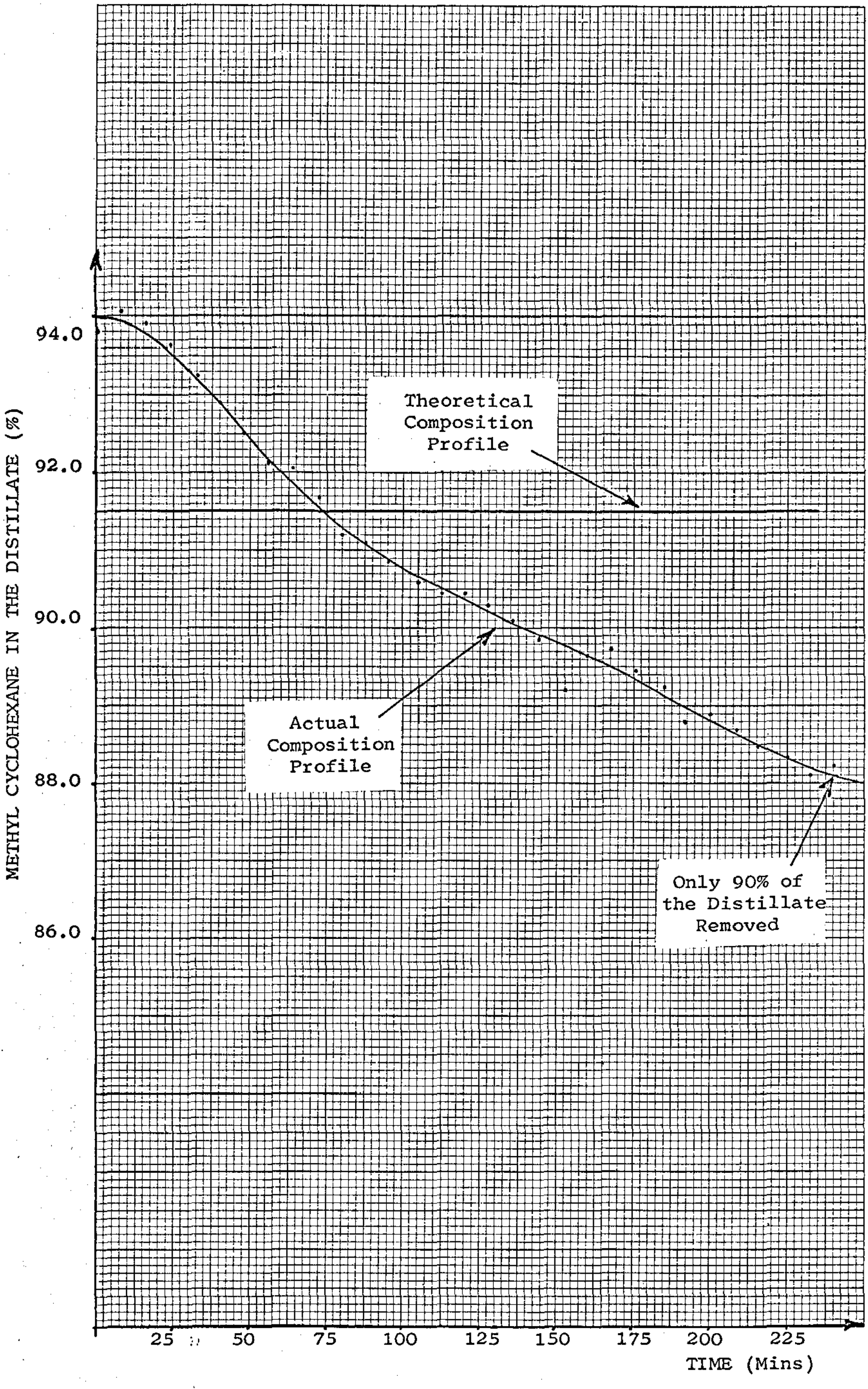
It may be seen from the comparison of the actual and predicted reflux ratio profiles (Graph 7.2), that once again the effects of the dynamics of the system result in a poorer experimental separation. In fact, when this technique was used, the performance of the distillation unit was considerably worse than when the constant reflux ratio technique was employed.

7.3 EXPERIMENTAL INVESTIGATIONS USING THE DYNAMIC PROGRAMMING TECHNIQUE

This was the final direct comparison between experimental and theoretical separations. For this work the optimal reflux strategies, according to the off-line dynamic programming based analysis, were employed on actual distillation tasks. The composition profiles at the column head and in the still were plotted once again, and compared with the theoretical computations.

There were two techniques which could be employed in the comparison of the theoretical computations with actual results. They were based on changing the reflux ratio either after the appropriate amount of distillate had been removed, or when the still contents had reached the predicted composition. The experimental work that was carried out illustrated the use of either as a basis. In each case, the other variable was used as the medium for comparison between the theoretical and experimental results.

A detailed report of the experimental procedure, with typical



GRAPH 7.2 DISTILLATE COMPOSITION USING CONSTANT TOP PRODUCT TECHNIQUE

results, have been outlined in Appendix 10. The results may be summarised by saying that once again the actual separation was somewhat worse than that predicted by the theoretical computations (Typical response curves are indicated in Graph 7.3).

A further development of the dynamic programming analysis, was to up-date the predictions made during the initial off-line computation. This was done by re-predicting the optimum reflux strategy at cetain intervals during the experimental run.

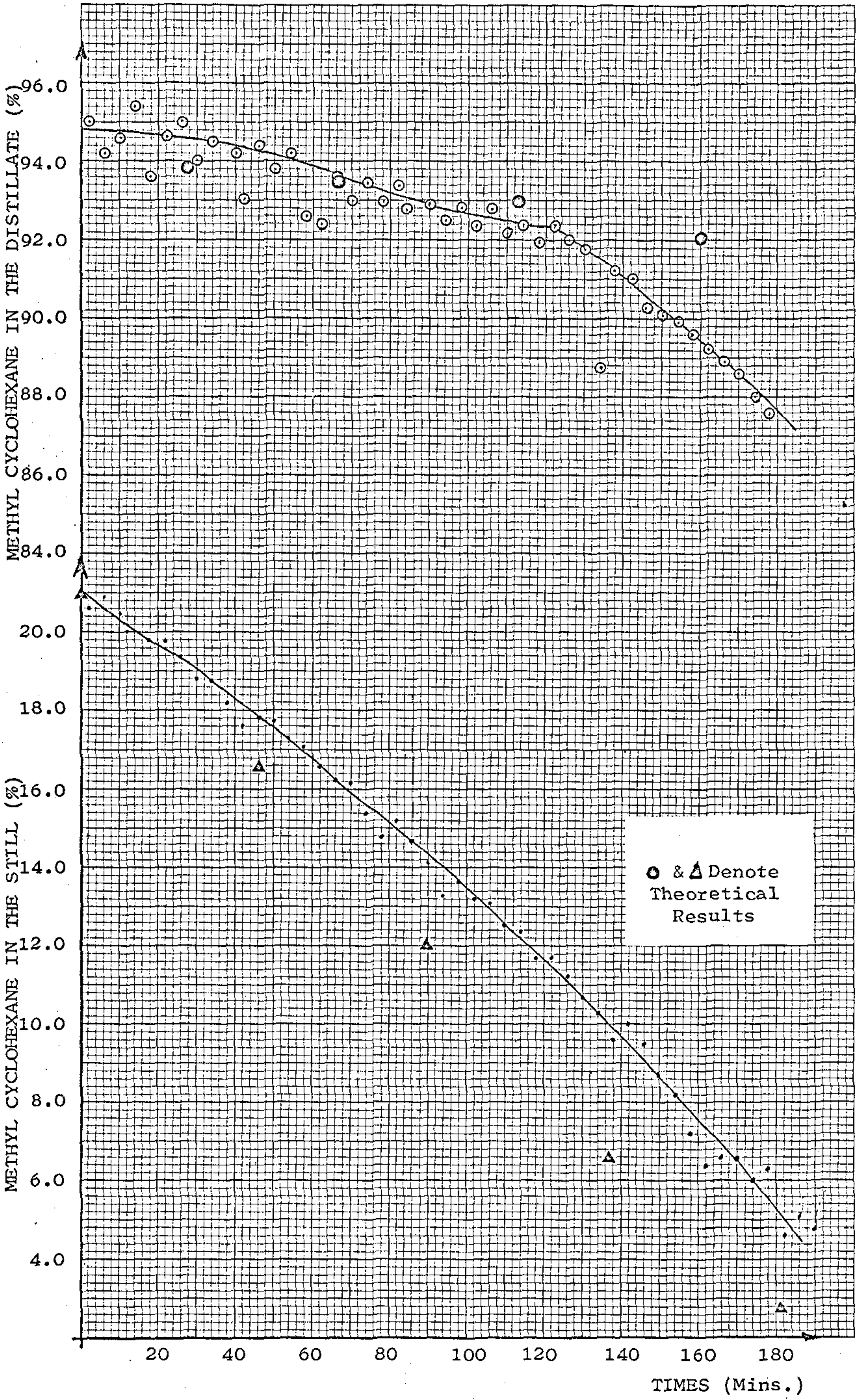
To achieve this, the values of R_m, X_m, X_{m-1} obtained during the off-line computation were all stored. In addition, the predicted profiles of X_{still} with time, and X_{n-1} with R_n, were also stored. The computer was then programmed to analyse the still composition at four minute intervals, and to monitor continuously the amount of distillate collected.

After the removal of a specified amount of distillate had been recorded, the actual still composition ($X_{\text{still exp.}}$) was compared with the stored predicted value of the still composition ($X_{\text{still pred.}}$). If the experimental and theoretical still compositions were found to differ significantly, then re-assessment of the optimum reflux vector was carried out.

i.e. $X_{B_D} - X_{B_E} = e$

if $|e| >$ Acceptable Error, re-calculate FR^*_n.

The re-calculation of FR^*_m was carried out using the present state of the still, according to experimental information, as the starting point.



GRAPH 7.3 THEORETICAL AND ACTUAL COMPOSITION PROFILES

Using the results calculated off-line, a new optimum vector FR_m^* may be calculated rapidly, since the computation time for this step was of the order of five seconds when using the on-line Ferranti Argus 108 computer.

This procedure may of course be repeated as often as was found to be necessary.

A further advantage of employing this up-dating procedure, was that it ensured that, during the last stage, the composition in the still was brought towards the desired final composition. As a result, the errors for each stage were not compounded. In fact, with respect to the composition variable, the errors were corrected for at the end of each stage.

This technique was employed on one of the distillation tasks illustrated in Appendix 10. For the purpose of the illustration, it was decided to use the up-dating procedure at the end of each stage of the distillation (prior to the change in the reflux ratio). The computer was programmed to introduce the sub-routine after the appropriate amount of distillate had been collected. Hence, at the end of the first stage of distillate collection, the still composition was measured, and then used as the basis for determining the new optimal reflux strategy. The flow diagram of this sub-routine has been included to illustrate its mode of operation, and position relative to the main analysis programme (Figure 7.1).

The effect of employing the sub-routine is best illustrated by comparing the results of experimental runs with and with-out the use of the sub-routine (Graph 7.4). It can be seen that the

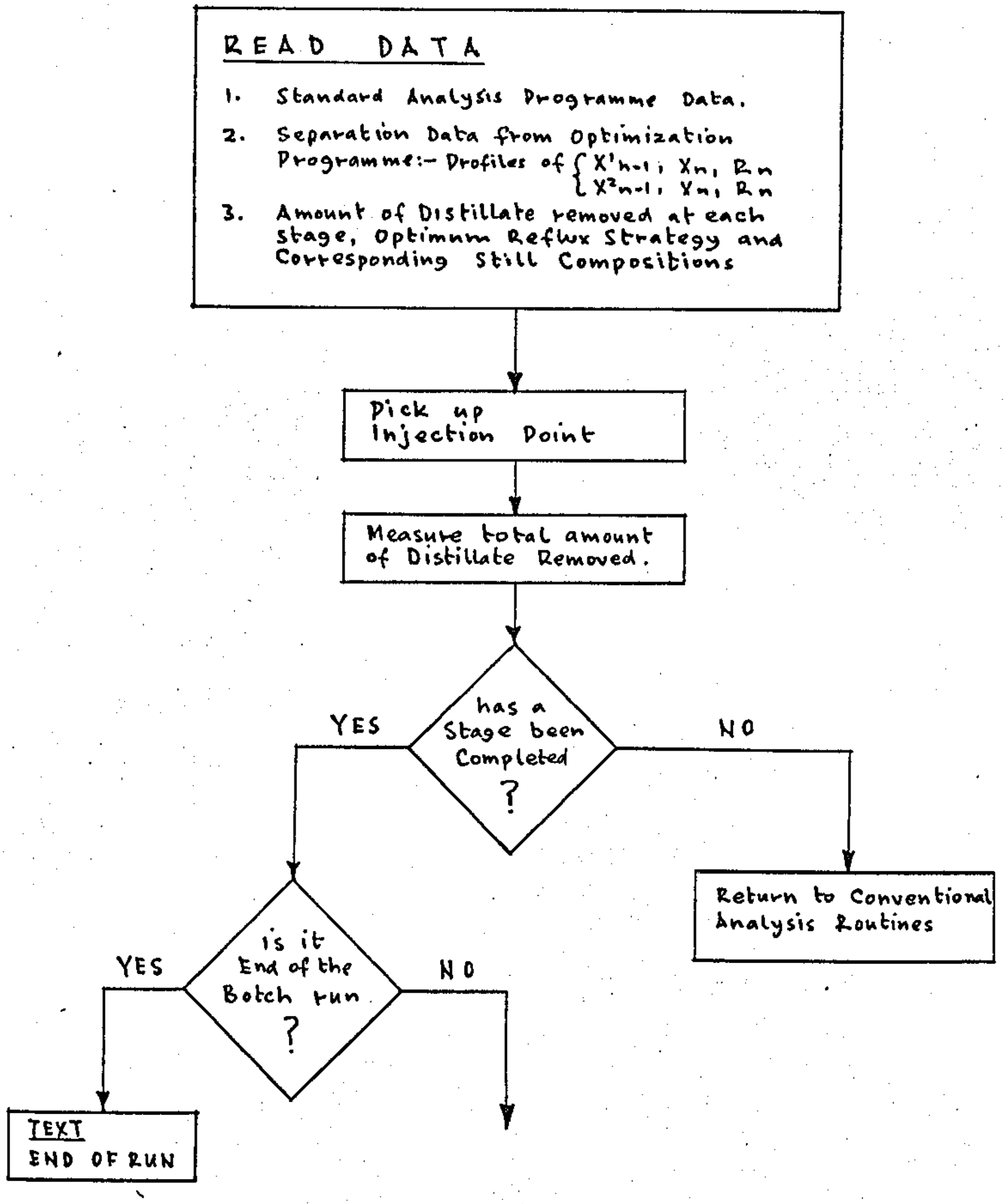


FIGURE 7.1

FLOW DIAGRAM OF ROUTINE TO
UP-DATE OPTIMUM REFLUX VECTOR ON-LINE - PART 1

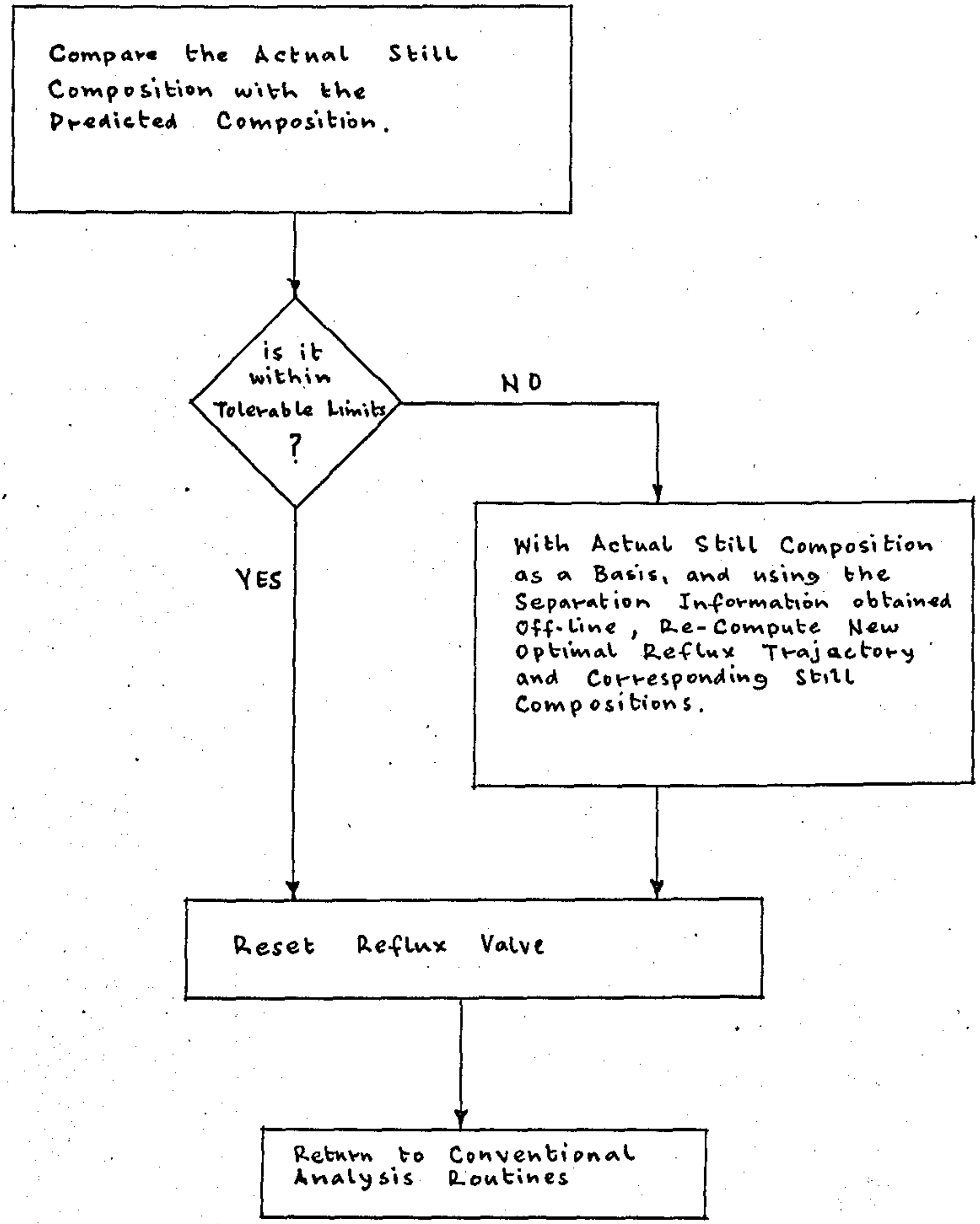
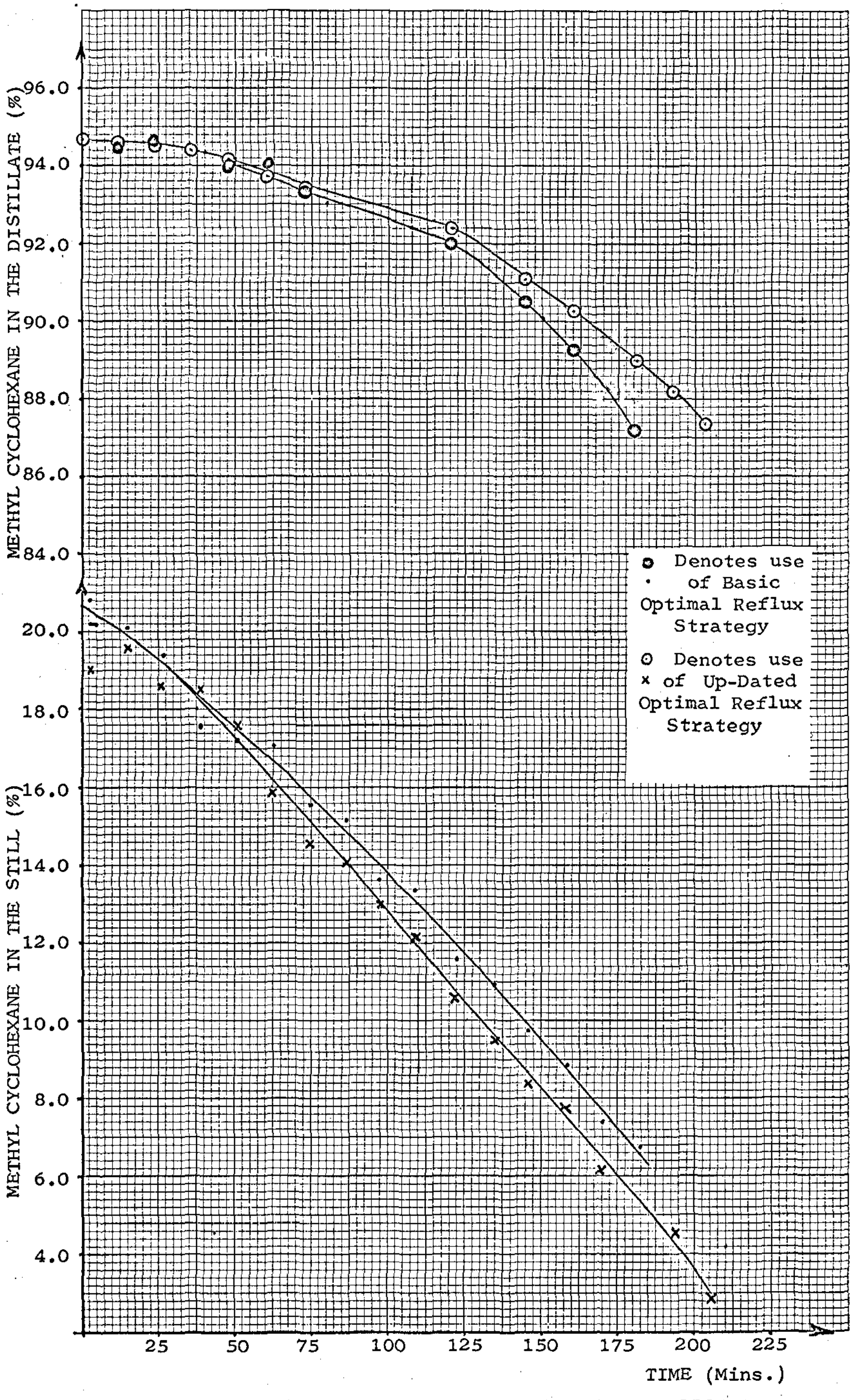


FIGURE 7.1

FLOW DIAGRAM OF ROUTINE TO
UP-DATE OPTIMUM REFLUX VECTOR ON-LINE - PART 2



GRAPH 7.4 EFFECT OF UP-DATING ROUTINE ON COMPOSITION PROFILES

inclusion of the sub-routine results in significant changes in the reflux strategy. These changes in turn produce a distillate nearer the requisite composition.

7.4 EXPERIMENTAL INVESTIGATION INTO USE OF THE SNATCH TECHNIQUE

The purpose of this work was to ascertain what benefits would accrue from operating the batch distillation unit according to this technique. This took the form of both comparing its performance with that of other techniques, and of investigating the practical effects of using various different snatching rates.

For the initial work, distillation tasks identical to those which had been previously investigated were undertaken. This allowed a direct comparison to be made of the benefits of operating the unit according to the various techniques. The basis of the comparisons was that the same run time, and hence it was assumed, the same heat input, was used for the snatch experimental runs, as had been used with the other experimental runs (This was of course dependant on the fact that the same boil-up rate had been used throughout the experimental work).

This initial analysis included work to determine what would be the best arrangement for snatch size and frequency with which the snatches were removed. Since the overall run time was fixed, the two variables were of course inter-dependant. For this reason, once the magnitude of a snatch had been determined, the number and frequency of the snatches were also fixed .

Control of the two main loops during the experimental work was straight forward. The boil-up rate was controlled in the manner outlined in the previous sections, whilst the control of the reflux loop was a simple manual arrangement. Normally the column was maintained at total reflux, but by manipulating a manual valve on the distillate off-take line, snatches of distillate could be easily withdrawn by emptying the reflux flow-meter (Figure 7.2). This arrangement was extremely efficient, and infact, the time required to remove even the largest snatches was of the order of five seconds.

Whilst a detailed description of the experimental procedure and results from a typical analysis have been included in Appendix A11, the results have been summarised below (Graphs 7.5 and 7.6).

Since the distillation unit was charged with the same material for all these investigations, these results illustrate the effect of altering the snatching rates.

The first set of graphs (Graph 7.5), indicates the separation achieved when using the same run time as had been employed in the previous experimental investigation, and the second set of graphs (Graph 7.6), were obtained when the experimental run time was cut by a third. (This is also of course, a reduction of the heat input by one third).

The separation achieved during each of these experimental analyses, was assessed by determining the average composition of the distillate collected during each run. By considering these results (Graph 7.7), it can be seen that there is a distinct

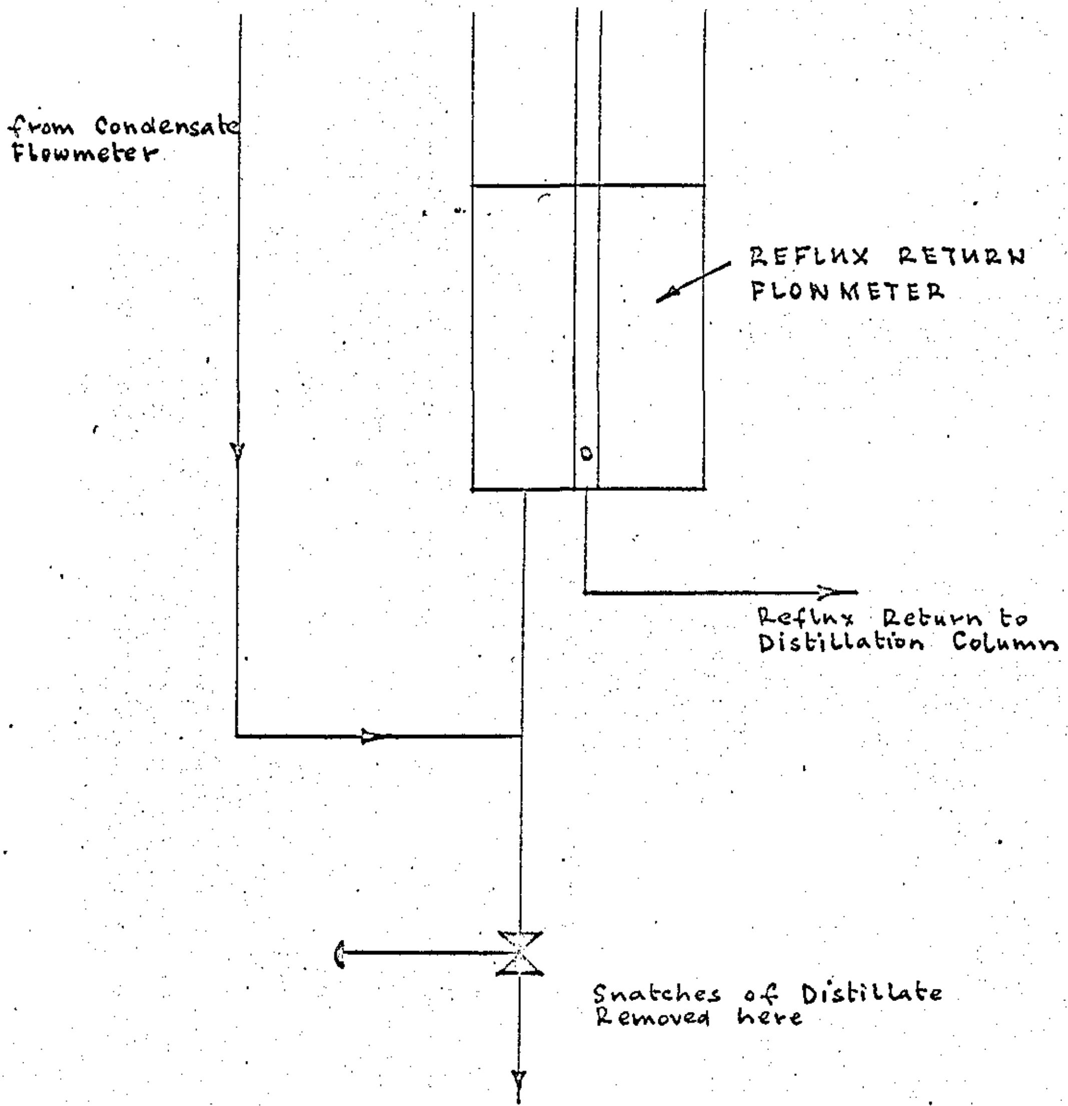
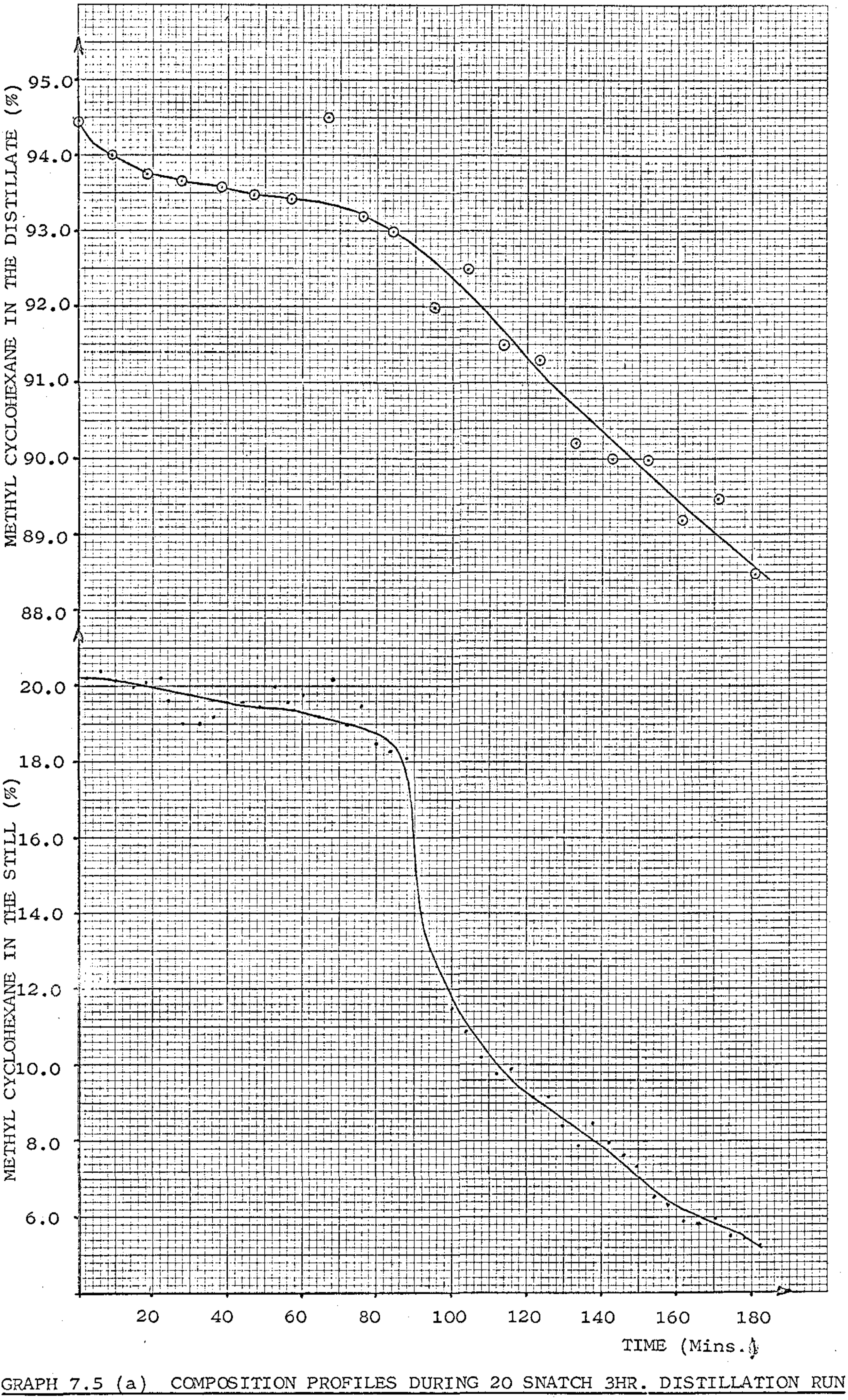
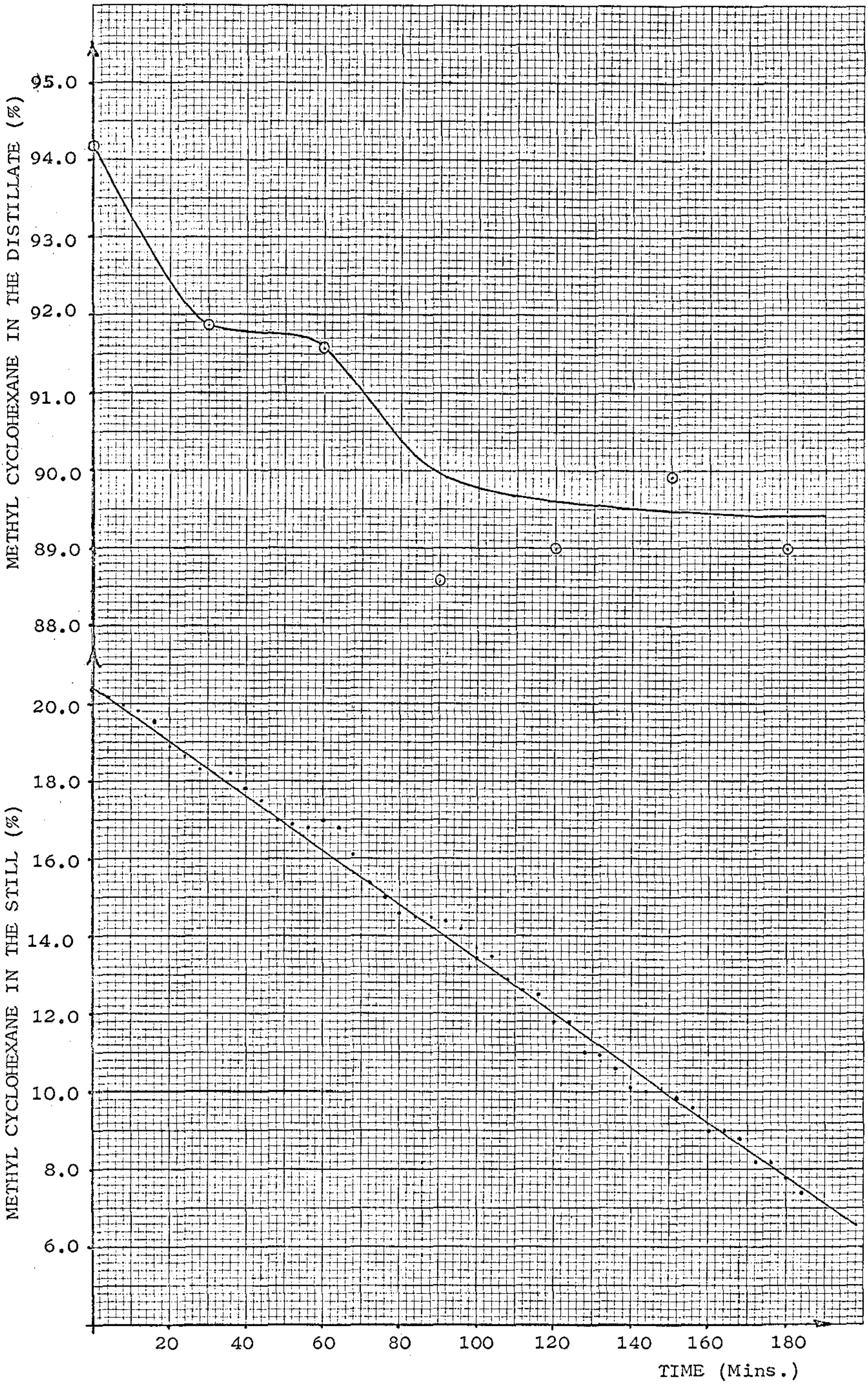


FIGURE 7.2

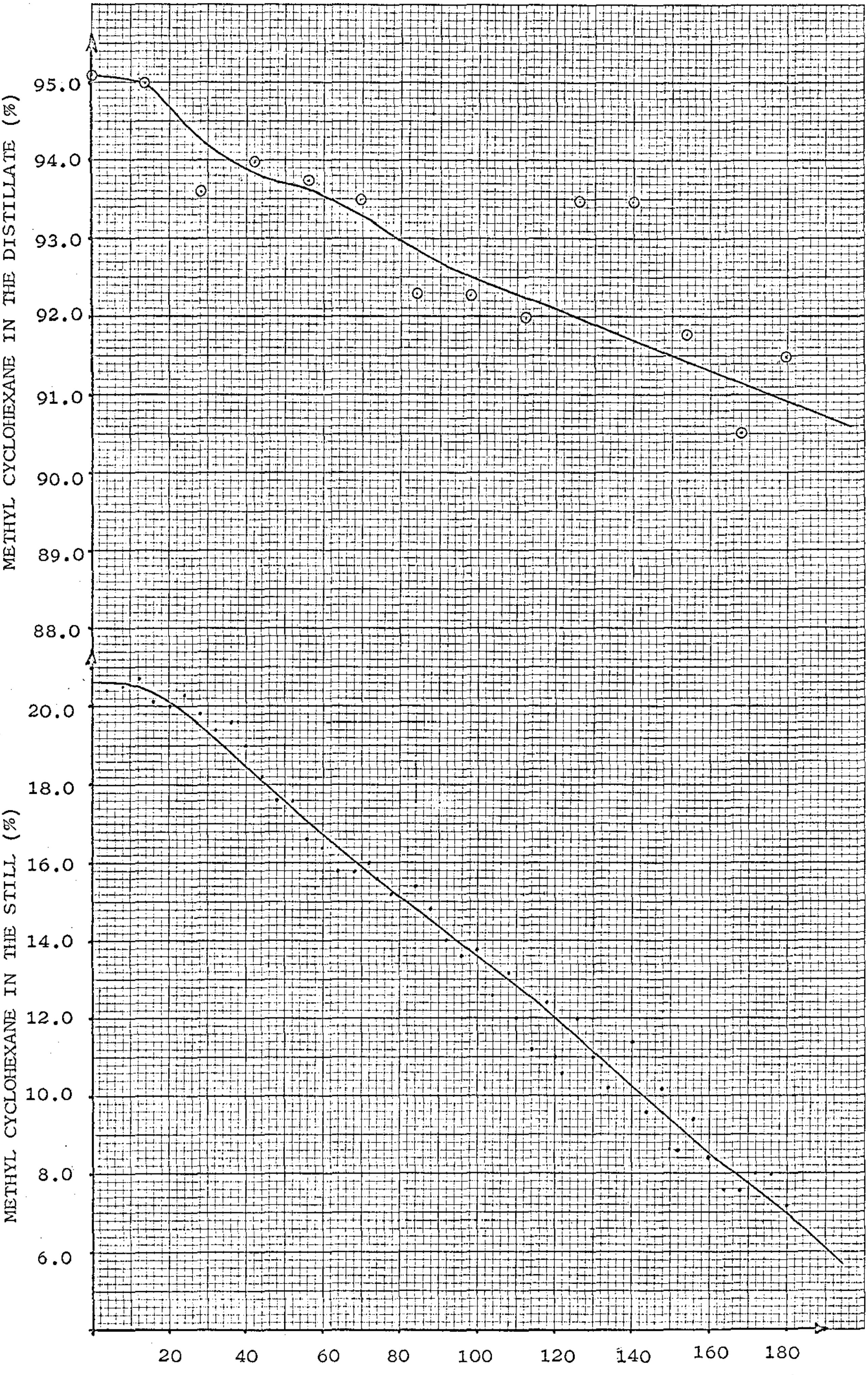
ARRANGEMENT FOR THE REMOVAL OF SNATCHES OF DISTILLATE FROM THE REFLUX RETURN FLOWMETER.



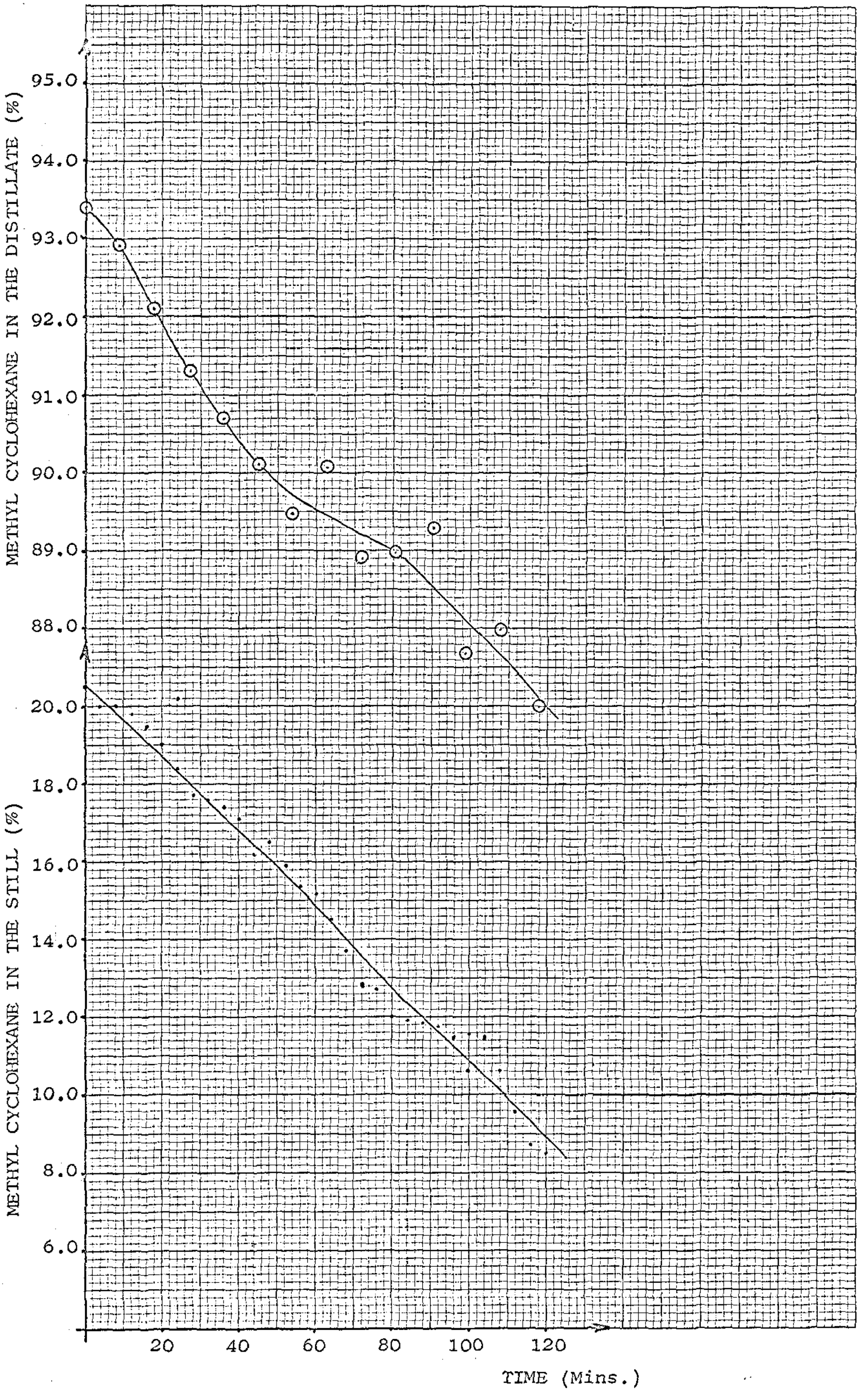
GRAPH 7.5 (a) COMPOSITION PROFILES DURING 20 SNATCH 3HR. DISTILLATION RUN



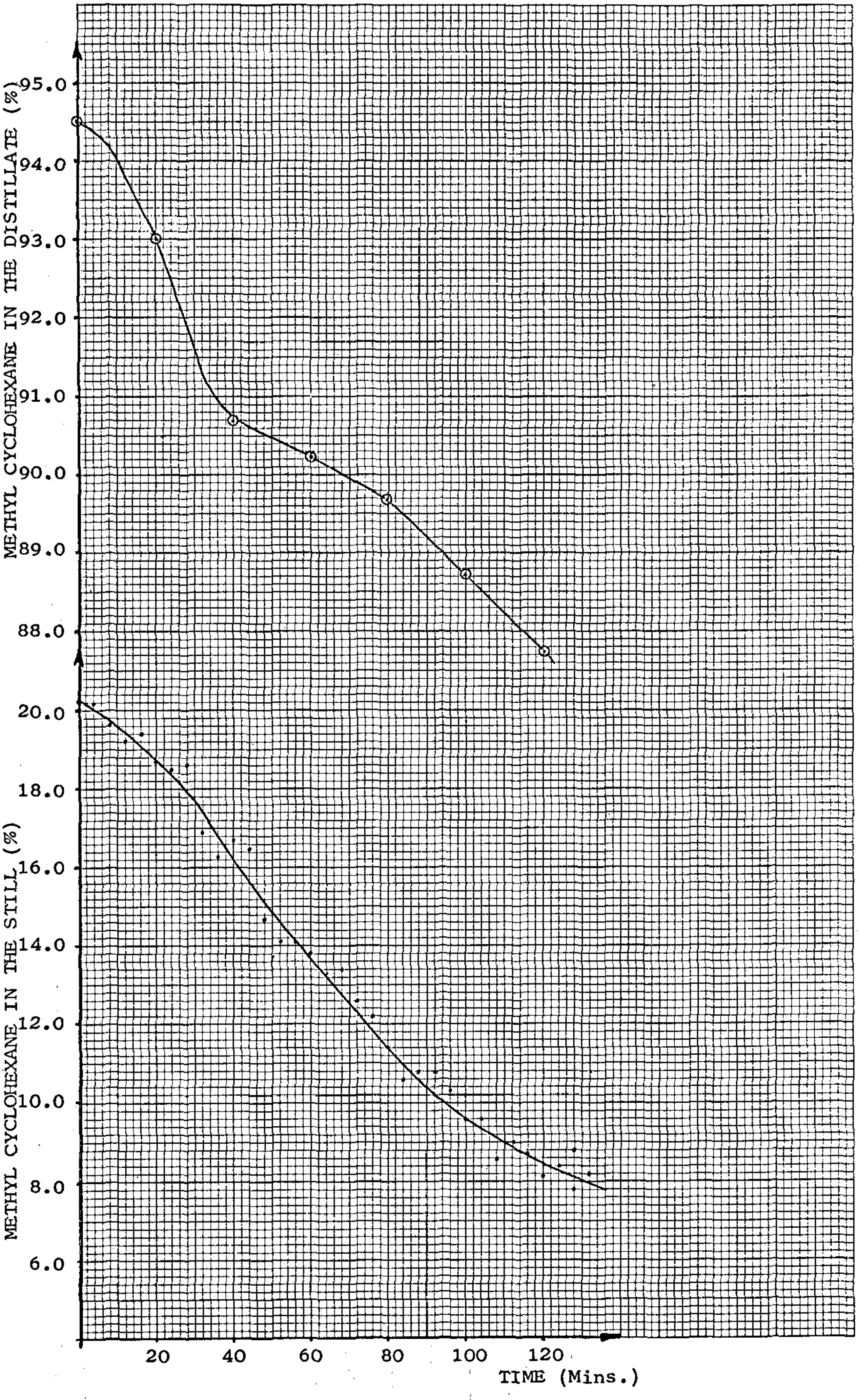
GRAPH 7.5 (b) COMPOSITION PROFILES DURING 7 SNATCH 3HR. DISTILLATION RUN



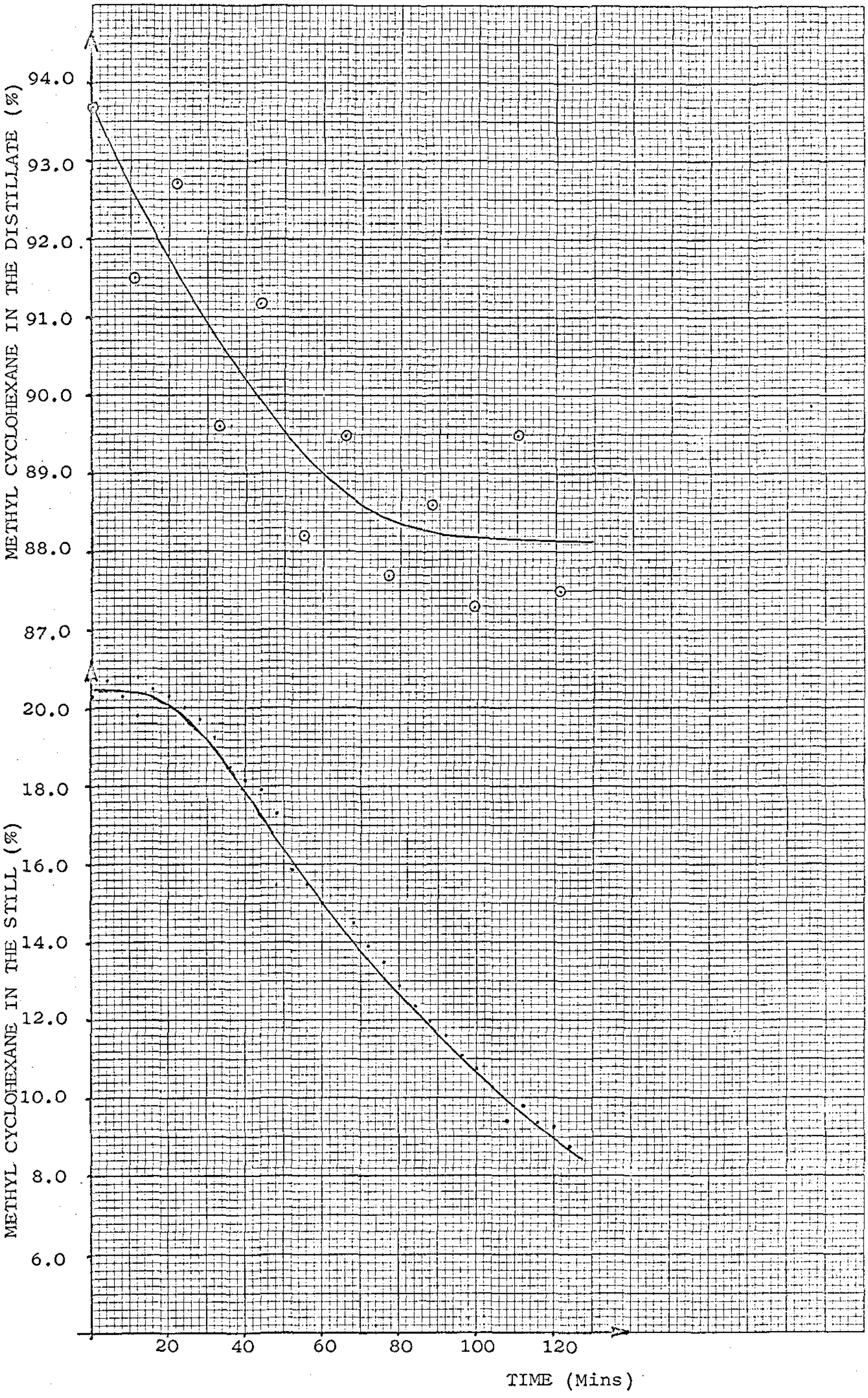
GRAPH 7.5 (c) COMPOSITION PROFILES DURING 14 SNATCH 3HR. DISTILLATION RUN



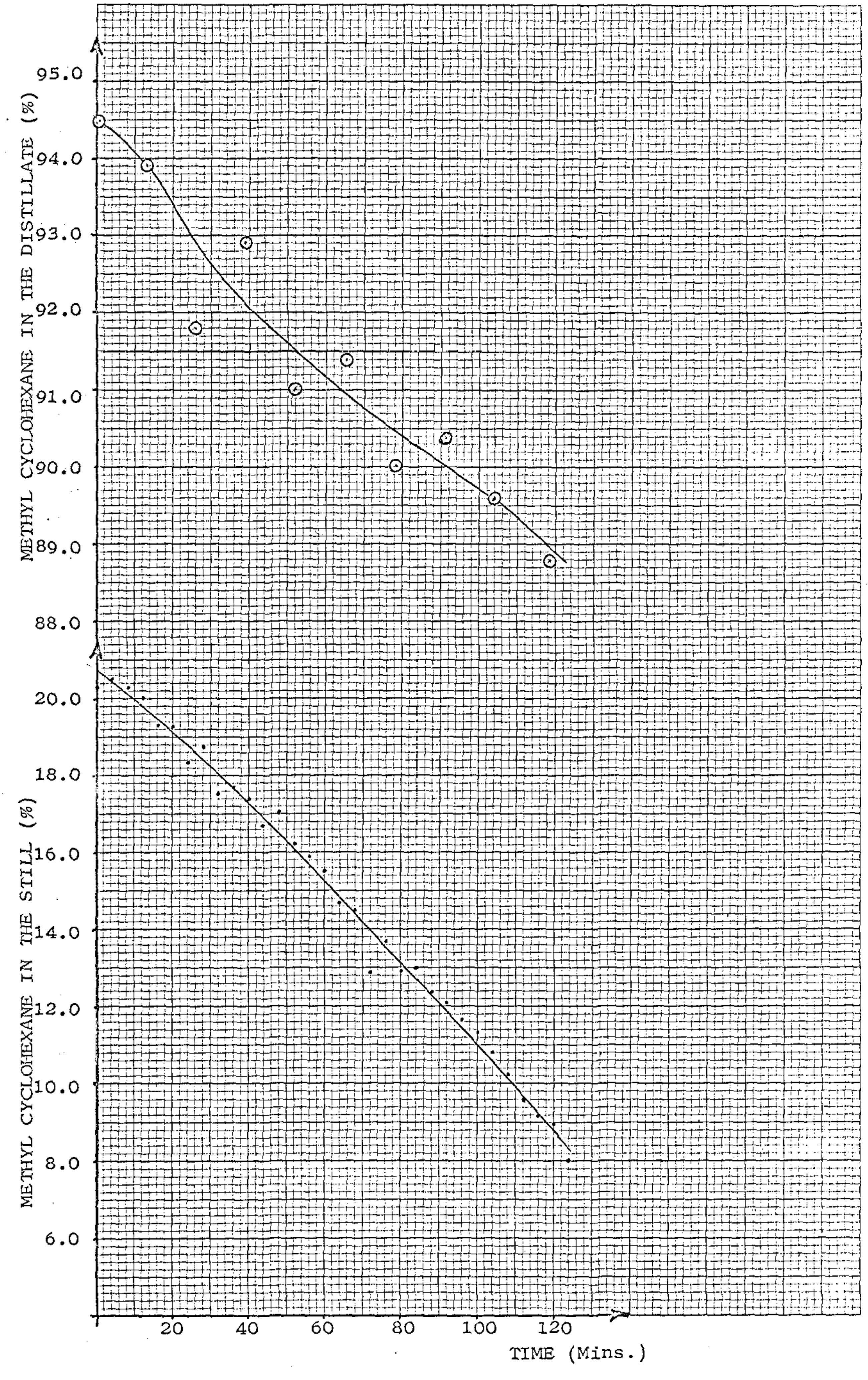
GRAPH 7.6 (a) COMPOSITION PROFILES DURING 14 SNATCH 2HR. DISTILLATION RUN



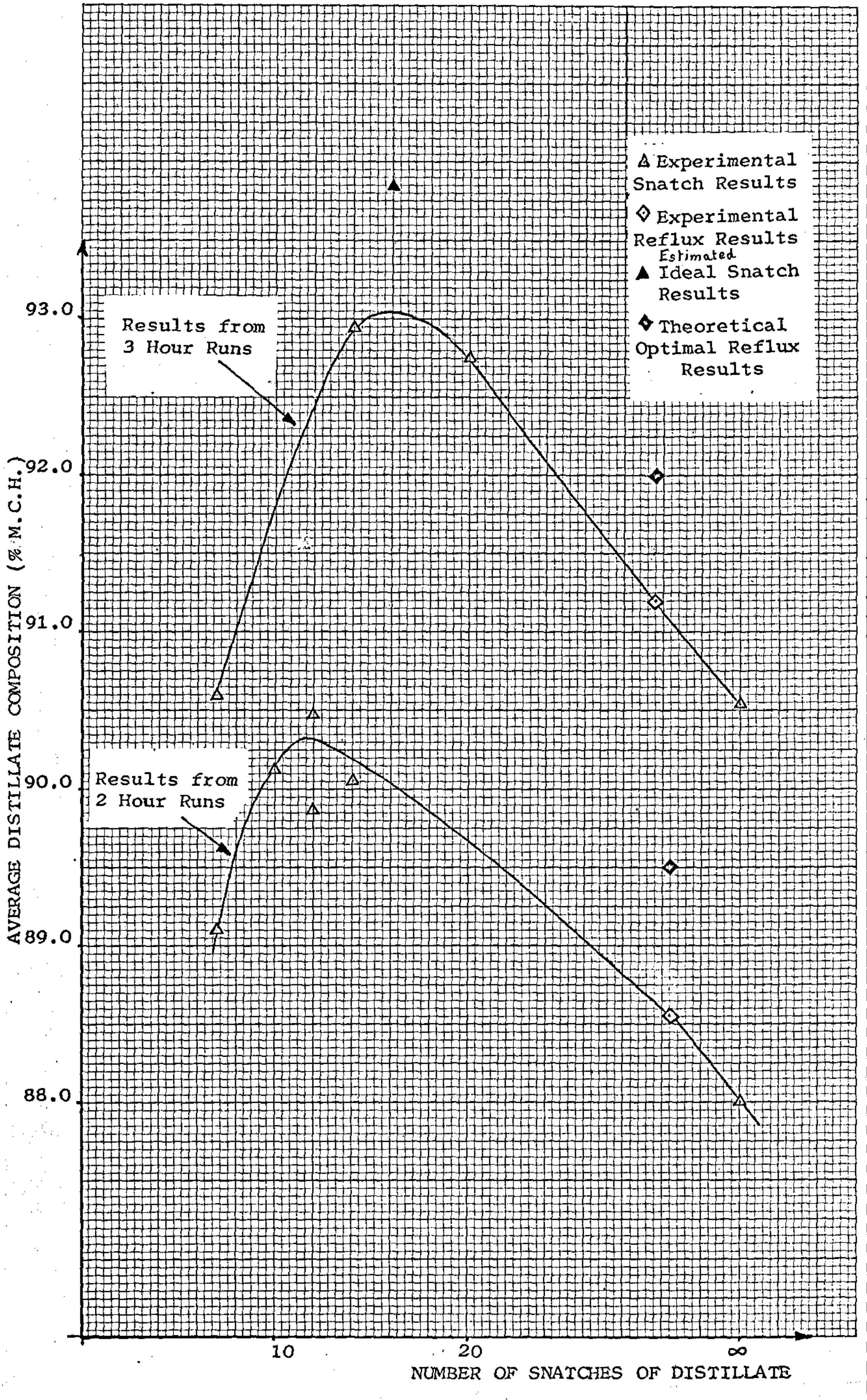
GRAPH 7.6 (b) COMPOSITION PROFILES DURING 7 SNATCH 2HR. DISTILLATION RUN



GRAPH 7.6 (c) COMPOSITION PROFILES DURING 12 SNATCH 2HR. DISTILLATION RUN



GRAPH 7.6 (d) COMPOSITION PROFILES DURING 10 SNATCH 2HR. DISTILLATION RUN



GRAPH 7.7 DISTILLATE COMPOSITION PROFILES FROM 2 & 3 HOUR RUNS

optimum snatching rate for a given distillation task.

Naturally, it must be realised that the snatching policies indicated by these results are dependent on the apparatus employed and the nature of the distillation task under investigation. For this reason, the optimal strategies indicated in the results must only be considered in the context in which they were obtained.

The importance of this work was of course the possible economic advantages when applying it to a batch distillation task~~s~~. Consideration of this will be made in some detail in the proceeding section (Section 7.5). However, the possible saving in heat requirement is well illustrated when the technique is compared to the constant reflux technique (Graph 7.7). By considering these results it can be seen that possible savings in heat requirement of the order of 30% are indicated.

7.5 COMPARISON OF THE VARIOUS DISTILLATION TECHNIQUES.

The various distillation techniques had all been analysed by considering their employment in similar distillation tasks. It was therefore possible to compare the different techniques by considering their ability to separate the two components.

The particular task examined in detail was the separation of a 20% Methyl Cyclohexane/80% Toluene mixture, and the basis of the comparison was to use the same experimental run time for all the techniques. (The boil-up rate was of course also fixed throughout the experimental work).

Because of the computational task involved, the initial run time chosen was that which had been specified by an analysis based on the off-line Dynamic Programming optimisation routine. This routine specified the optimum reflux ratio strategy required in order to carry out the distillation task when using four off-take stages. If one then specifies the boil-up rate within the distillation unit, the total run time is fixed. The details of the theoretical analysis have been outlined in Section 6 (Table 6.7).

With this information, the distillation task was undertaken using Dynamic Programming based optimisation, Constant Reflux Ratio, and the Snatch techniques. The performance of all these techniques were compared by considering the average composition of the distillate obtained from the distillation unit.

A similar analysis was also undertaken for the second distillation task, for which the experimental run time was two thirds of that employed previously, and the results from using the various techniques compared once again.

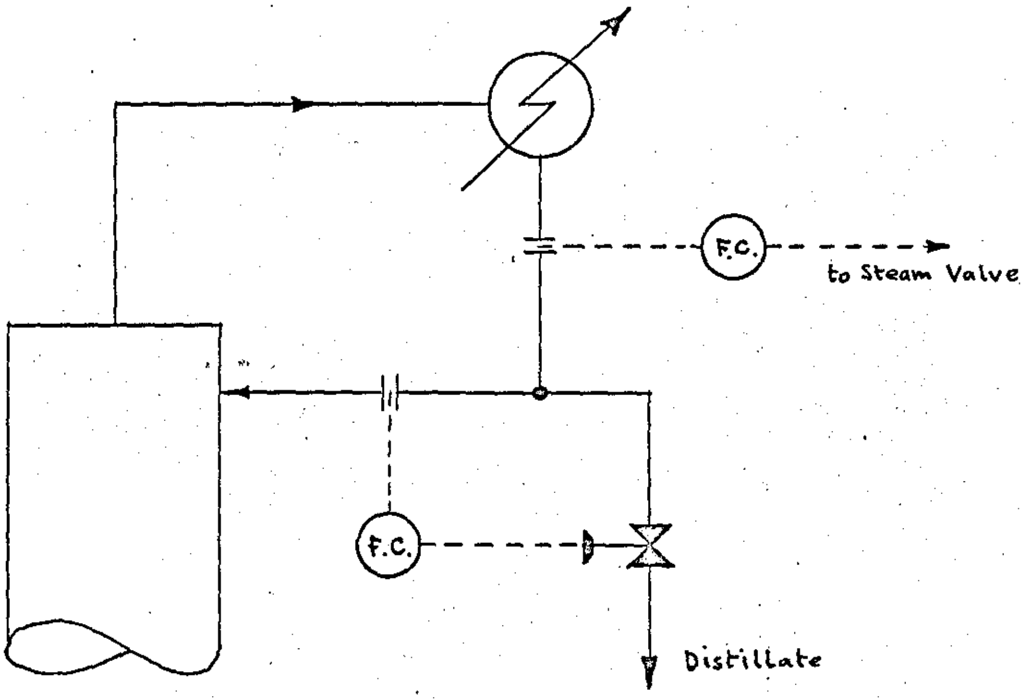
It can be seen from these results, (Graph 7.7), that the use of the Dynamic Programming technique results in an improvement when compared with the Constant Reflux technique. However, this improvement is somewhat marginal, and to obtain it, a not inconsiderable amount of computer effort is required. For this reason, its application would be limited to particularly suitable distillation tasks. (Typical tasks worth considering would be those which are undertaken repeatedly, and which are required to give a high yield of the more volatile component).

However, this work is somewhat overshadowed by the results from the analysis of the snatch technique which indicate that it is considerably superior to the other modes of operation.

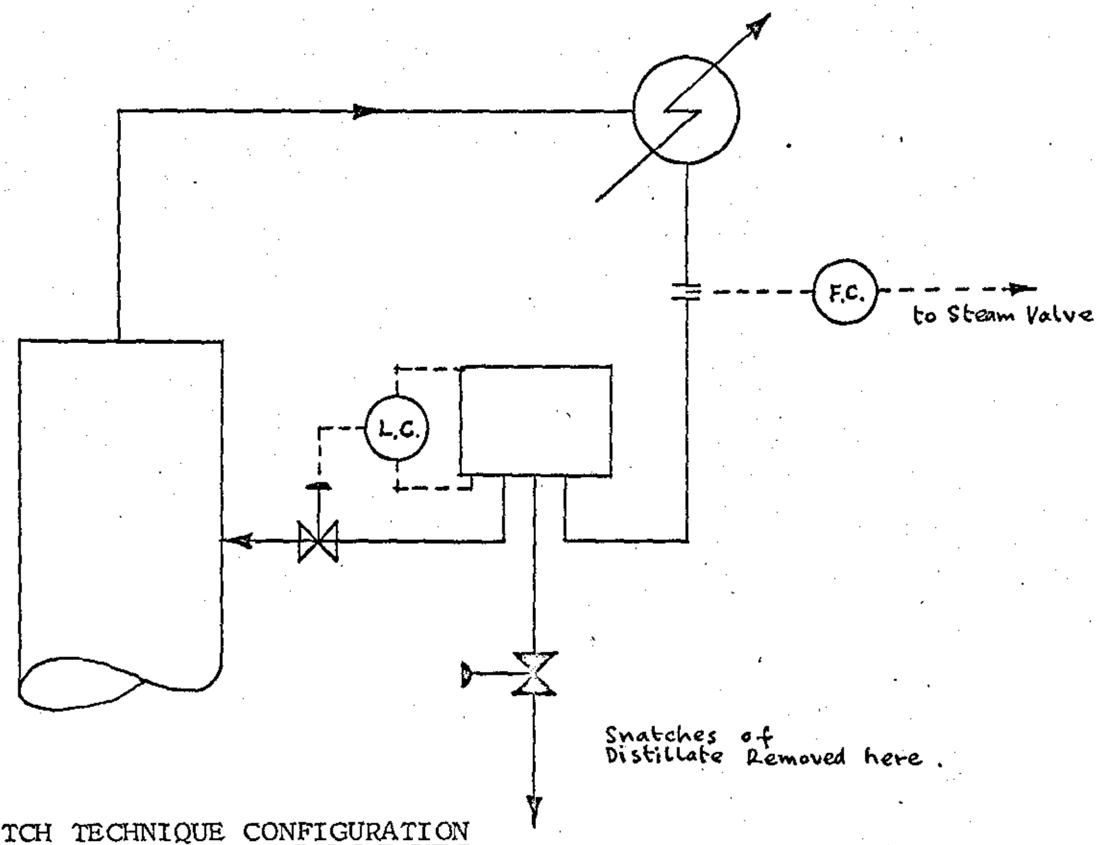
In fact, by comparing the results from the two sets of experimental analyses, it can be seen that by using the snatch technique, savings of the order of 30% in total heat requirement are possible.

Perhaps more significant than the reduction in energy requirements, is the fact that its application also increases the possible throughput of a given unit by a factor of the order of 30%. This is because the reduction in energy requirement is not accompanied by a reduction in the rate at which the energy can be utilised.

A further facit of the Snatch technique is that, unlike other non-conventional modes of operation (e.g. cyclic and use of optimum reflux strategies), there are no extra difficulties in its implementation, when compared to the conventional distillation techniques. In fact, it is merely necessary to replace the reflux return control loop with a level controller on the reflux drum (Figure 7.3). This control loop is required to ensure that the desired amount of liquid is always held in the reflux drum.



ORIGINAL CONFIGURATION



SNATCH TECHNIQUE CONFIGURATION

FIGURE 7.3

MODIFICATIONS TO THE REFLUX RETURN LOOP FOR SNATCH TECHNIQUE IMPLEMENTATIONS

Although the results from the experimental work indicate extremely useful savings when employing the snatch technique, the column could have been operated more efficiently. Infact, there were two areas in which improved operating techniques could have been introduced.

The first area of improvement concerns inadequacies of the equipment. There were two points here, both of which concern the hold-up in the reflux drum. Because the apparatus had only been modified from conventional operation, as distinct from being specifically designed for Snatch operation, the snatches of distillate were always removed from a constant hold-up of 800 cc.

The second fault with the experimental apparatus was that the hold-up from which the snatches were removed was a poorly mixed drum. For this reason, the separation achieved in this drum during the total reflux period was substantially less than ideal.

Infact, the optimal snatch strategies indicated by the experimental work were dependent on these inadequacies of the apparatus as well as on the fundamental dynamic response of the system. The experimental work indicated an optimal snatch size within the range 375 cc. → 450 cc., whilst the hold-up in the drum was 800 cc. However, it is probable that improved separation could have been achieved with a smaller hold-up in the reflux drum.

The second area of possible improvement was the use of a snatch strategy which was time dependent. This would allow material to be removed faster initially when the separtion was somewhat easier, and allow more time for the material to separate towards the end of the run. This could be achieved by using either a variable snatch size with a fixed snatching frequency, or by fixing the snatch size and allowing the frequency to vary. To determine this optimal snatching strategy, an optimisation routine similar to that carried out to optimise the reflux ratio strategy could be carried out.

DISCUSSION ON THE RESEARCH WORK CARRIED OUT AND SUGGESTED FURTHER DEVELOPMENTS

SECTION 8:- DISCUSSION ON THE RESEARCH WORK CARRIED OUT AND SUGGESTED FURTHER DEVELOPMENTS.

CONTENTS Page

DISCUSSION ON THE RESEARCH WORK CARRIED OUT AND SUGGESTED FURTHER DEVELOPMENTS.

8.1 INTRODUCTION

The purpose of the research work was to investigate the role of the digital computer in the control of distillation units.

The investigation considered features which may be grouped into two distinct aspects. The first role to be considered was the control of individual loops, and the second, the overall operation of distillation units (in particular batch distillation units).

We will consider now the control of the individual loops. Here, the special features which result from using the digital computer are two-fold. Namely, they are that non-linear control algorithms may be employed, and that a wider range of measured variables may be used in the control loops.

The second role was the investigation of overall column operation. This analysis was carried out mainly off-line, and the results obtained were available for use in feed-forward routines.

8.2 OBSERVATIONS FROM THE INVESTIGATION OF THE FEED-BACK LOOPS.

The control of distillation units must satisfy two requirements, viz. control of column hydraulics and control of mass transfer. For continuous columns, the aim of the control strategy is to ensure that the system is maintained at the desired static state as efficiently as possible. Although one might think that this would be relatively straight forward, it is often complicated by the complex design of sophisticated continuous distillation trains. On the other hand, with batch distillation units, although their operation is of a necessity 'dynamic', their design and behaviour may be considered to be more straight forward.

The design of the unit considered for the experimental work was extremely conventional and straight forward (Section 4). It was operated at atmospheric pressure and the cooling water outlet temperature could easily be controlled manually. For these reasons, it was found that only the boil-up rate, the reflux return, and the top-product composition needed control systems to ensure satisfactory operation.

In fact, for the unit used in the experimental investigation, the hydraulics could be controlled completely by controlling the boil-up rate and the reflux return rate. By employing measurements from the automatic chromatograph, the mass transfer within the unit could also be controlled.

8.2.1 CONSIDERATION OF THE HYDRAULIC LOOPS.

It was found from the initial experimental work that reasonable control of these loops was possible when using analogue controllers. However, precise control of these loops was not possible without using more sophisticated controllers. This is best illustrated by considering the control of the boil-up loop. It was found that for satisfactory control of the loop near its flooding point, it was necessary to employ a digital cascade control system. This digital controller used both the pressure drop across the column, and the top product flow rate. In addition, it included a linear regression filtering routine to process the pressure signal and rate restraints on the outputs from the two stages of the controller. It is of course with controllers of this form where the significant benefits accrue from 'standard' Direct Digital Control installations.

8.2.2 CONSIDERATION OF THE MASS TRANSFER LOOP.

This control loop was of primary interest to the schemes which endeavoured to analyse the overall operation of the distillation unit. In particular, to those which considered optimal or sub-optimal operation. However, it is an excellent illustration of the increased flexibility with which the measured variables for various control loops may be chosen.

The chromatograph automatically took and processed samples from the distillation unit, and the readings from the chromatograph were in turn processed by the computer to obtain the composition of the sample. This composition was then utilised in a digital cascade controller which controlled the reflux ratio.

8.2.3 CONCLUSIONS FROM THE FEED-BACK LOOP ANALYSIS.

The predominant fact which emerged from the analysis of the feed-back loops was that the digital computer was a tool with which powerful controllers could be generated to control the more difficult loops. Conventional controllers are linear in their operation, and limited in their employment of measured variable.

The digital controller has a much greater flexibility, and it is this flexibility which must be fully utilised in order to gain the maximum advantage from Direct Digital Control.

Finally, one must consider the small non-conventional control installations. In the recent past, the conventional role of Direct Digital Control has been in the control of several hundred loops (e.g. oil refineries etc.). This rôle is now being extended to smaller applications with the advent of small control computers, and sophisticated controllers.

The manufacturers of conventional controllers are now producing 'tailor' made controllers, which are able to control satisfactorily because they have been especially designed for a given task. These may take the form of a piece of digital logic, or a sophisticated pneumatic arrangement. They may utilise either a standard signal from a conventional measured variable, or include a specific interface section which processes a signal from a non-conventional source.

The latest popular trend in digital control computers has been the development of small computers with typically a minimum of 1K of core store and one working accumulator. These computers are cheap, and may be employed to advantage on a whole range of purposes.* On larger installations they may be employed in banks, overall control of which may be governed by a larger computer. Alternatively, where direct digital control is considered to be of

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*The basic Ferranti Argus 600 costs £1,700.

use in only a limited area, a single computer may be employed.

If we consider first, the 'banking' system with the larger computer installations. Typical of the problems which have impeded the spread of Direct Digital Control has been the fear of catastrophic failure if the computer malfunctions. In addition, there has been the difficulty of ensuring efficient use of the computer and of justifying the total cost of its installation. By employing the small computers, these problems may be overcome. The cheap, small, control computers are fully utilised controlling their given area of plant, and their overall operation is governed by a larger computer, which may also be employed in carrying out modifications to the control programme, and for monitoring the plant. In addition, in the event of sub-computer failure, it may step in and replace the malfunctioning computer. Finally, the use of a large powerful computer may be utilised for complex optimisation studies.

This computer would normally be employed on a host of other tasks, and only brought in on a time shared basis. A scheme of this nature fully utilises the power of the computer, and results in increased reliability by its installation. Finally, the reduction in stand-by computer power means that the computer cost barrier may be broken more easily.

We may consider now the application of the single small computer installation, since most of the benefits from its use are also true for the multi-computer installations. Here its role is that of a powerful flexible controller. It has the advantage over the hardware controllers that it may be easily modified in the light of operating experience, or because of a change in use.

In addition, once the computer has been installed on the plant, its possible use on a host of other tasks may be investigated. These other tasks may include optimisation, alarm scanning, and data logging. The experience from these extra investigations may result in the desire for a more powerful machine which may often be obtained by modifications to the existing system.

Hence, it is felt, that with the advent of these latest generation of small computers, we are on the thresh-hold of a further surge forward in the application of digital computers in the control field. There is, however, a further problem which has already been realised by other authors (31), and was also illustrated by this research work. This is the problem of efficient, accurate, reliable measuring elements.

It is apparent that a highly efficient controller is of little use if its measured variable cannot be read accurately. Although these controllers may include filtering routines for processing the signal, very often this may not be sufficient. There were two examples of difficult measured variables in the experimental work.

The first was the measurement of the distillate off-take. The basic problem here was of measuring a small flow rate by differencing two much larger flow rates. This illustrated the problem of compounded errors, which together gave an extremely large composite error, and also the problem of finding an alternative means of reading relatively small fluctuations immediately, automatically and accurately.

The second measured variable of special interest was the mass transfer analysis, using the chromatograph with its automatic sampling device. Here we have a sophisticated measurement technique, but once again, extreme care must be taken when it is employed. For the experimental work under consideration, the two component mixture was amenable to an investigation using a gas-liquid chromatograph. Initially, extreme care had to be employed to ensure that it was calibrated accurately, and subsequently, it was checked from time to time for any drifts. Infact, during the course of the experimental work, periodically it had to be completely re-calibrated.

Hence, in conclusion, it may be said that the new generation of small digital computers will probably result in a substantial upsurge in the use of direct digital control.

This upsurge will almost certainly be coupled with the deployment of an increased proportion of sophisticated control algorithms. Finally, a key factor which will tend to govern the rate at which this explansion accelerates, will be the development of new and better sensing mediums, with which to automatically measure an increased range of process variables.

8.3 OBSERVATIONS FROM THE INVESTIGATION OF THE FEED-FORWARD SYSTEMS.

The investigation into the feed-forward control of the distillation unit may be sub-divided into two sections. They are the prediction of the mass transfer behaviour of the unit off-line, and the utilisation of this information on-line.

The feed-forward analysis that was carried out was based on the behaviour of a batch distillation unit (described in Section 4), and has mainly been based on the fact that the optimum trajectory for a batch distillation task is determined solely by the reflux strategy used (23).

The initial work was to predict the mass transfer when using the conventional batch distillation techniques of constant reflux ratio and constant distillation composition.

This was followed by an analysis into the merits of optimal operation of the batch distillation unit. It was decided to employ Dynamic Programming as the optimising technique.

For all these predictive investigations, allowance was made for the dynamic behaviour of the reboiler only. As a result, (mainly because the column dynamics gave a dominant mass transfer time constant of the same order of magnitude as the still time constant), it was not possible to obtain the predicted optimum trajectory.

Finally, an analysis of two non-conventional modes of operation was carried out. These were the 'cyclic' technique and the 'snatch' technique.

8.3.1 THE ANALYSIS OF CONVENTIONAL OPERATION OF THE BATCH DISTILLATION UNIT.

The programmes which were developed to predict the mass transfer within the unit did not include any allowance for the dynamic behaviour of the system. Infact they also did not include any allowances for the material held up in the column or reflux drum. For this reason, comparisons between experimental and theoretical results were somewhat limited.

8.3.2 THE ANALYSIS OF THE OPTIMISATION BASED ON DYNAMIC PROGRAMMING.

The dynamic programming technique, which was used to determine the optimal operating programme for the batch distillation unit, has both advantage and limitations when compared with other optimising techniques.

An important advantage of the technique is that the required data may be specified as either empirical or theoretical information, or a combination of both. In addition, it may be expressed in almost any form.

Moreover, other linear or non-linear programming methods generally require that all cost and operating data be put in the form of rather restricted types of equations. A further problem is that they often result in optimising equations which are difficult to solve.

When compared with trial and error techniques dynamic programming is considerably more efficient. In addition, the number of trial and error calculations increases exponentially with the number of stages considered, whilst the number of dynamic-programming calculations increases only linearly.

However, the major limitation of the dynamic programming techniques, is its restriction on the dimensionality of the composition vectors.

Mixtures of three or more components present great difficulties, because the computation effort required increases exponentially with the number of components. Unfortunately, this difficulty is significant with almost every method of solving an optimisation problem.

Once again, the model of the system did not allow for the dynamic behaviour of the system, and for this reason, the value of the investigation is somewhat restricted.

However, the theoretical analyses indicated appreciable benefits when the process was operated in an optimal manner, compared with the conventional operation. Although the experimental investigations did not compare directly with the theoretical work, the benefits of operating the column according to an optimal strategy were once again evident.

A particularly useful extension of the initial work, was the inclusion of the facility for on-line up-dating of the optimal trajectory. Since the inclusion of a perfect model in the optimisation routine is unlikely, and also because of the possibility of random disturbances on real plant, perfect off-line prediction is impossible. As a result, there were two benefits which accrued from the inclusion of the routine. The first is that the process was repeatedly returned to its optimum trajectory. The second is that the errors during the separation were not allowed to compound, but, on the contrary, were corrected for repeatedly during the distillation run. Hence, before the final stage of the separation, the contents of the still were checked, as was the total amount of distillate that had been removed to-date. From these facts, and the information stored in the computer, the final reflux ratio required to ensure the appropriate separation could be determined.

8.3.3 THE SPECIAL TECHNIQUES.

The 'cyclic' operation of distillation units that has been cited in the literature (83, 84, 85), appears to have certain advantages. Unfortunately, it is apparent that its practical application has some very real problems. This is best illustrated in the paper by V.N. Schrodt (85). It is felt however, that these problems will be overcome before too long.

As a consequence of the experimental work mentioned in the previous sections, the concept of the 'snatch' technique was initiated

The theoretical analysis was extremely limited, since it did not consider the dynamic behaviour of the system. This is of course an extreme limitation when considering this technique, since one's computations are then predicting the maximum separation possible in the unit. The purpose of these computations were merely to indicate the characteristic of the technique which they did quite adequately.

An experimental investigation of the technique was also carried out. The purpose of this investigation was to repeat the separation tasks which had already been undertaken using the other techniques. It became apparent from this that substantial advantages may accrue from operating distillation units in this manner. Infact, for the separations considered, a saving in heat requirement of up to 30% was indicated.

8.3.4: SUMMING-UP THE ANALYSIS OF FEED-FORWARD CONTROL

The work in this section may be sub-divided into two facets - theoretical computations and experimental investigations.

As a result of the experimental investigation, it was felt that if the theoretical computations did not include a dynamic model of the process, their usefulness was somewhat limited.

A dynamic model of the batch distillation unit has been proposed by Whalley (86). Unfortunately, the problem of its application in the feed-forward predictive routines, particularly the optimisation routines, has been the computational load of repeated reiteration when using the model. Infact, because of the 'curse' of dimensionality, at present the solution to the complete optimisation problem does not appear to be practicable.

As a consequence of the work that has already been carried out, and the points that have been outlined previously, investigations are now proceeding with the development of simplified dynamic models. The purpose of these models will be two-fold. Firstly, they will be employed in realistic off-line predictive computations, and secondly, in the eventual development of models suitable for inclusion in on-line control programmes.

The experimental feed-forward work was important for two reasons. Firstly, it gave an insight into the practical problems of operating the distillation unit when using the different techniques, and secondly, because of the inadequacies of the theoretical computations, it was the most suitable method for comparing the different operating techniques.

The practical problems that arose from this investigation will be considered first. The main problem was in obtaining a suitable means with which to measure the process variables. This has been mentioned previously, but it must be remembered that it is of particular importance when one is considering optimal or sub-optimal operation. This is because it is imperative that during these analyses that the process be defined precisely. Although the 'cyclic' mode of operating the distillation unit was not considered experimentally, it is apparent that it is here that the most serious practical problems would arise. However, this has been detailed elsewhere.

Consideration of the results from the experimental investigations, and the information which may be learned from them, will be made now. One may generalize by saying that of the conventional techniques, for the systems examined, the constant reflux technique was the most efficient. It was also apparent that savings were possible by use of the dynamic programming technique. The comparisons between these techniques had a compatability with the comparisons detailed by the off-line computations. Hence, the information from the two sources may be utilised together.

Finally, one must consider the experimental analysis of the 'snatch' technique. The experimental analysis illustrated that the snatch technique was an extremely useful tool for batch distillation. The basis of the experimental work was to carry out once again the same separations that had been used in the aforementioned analyses, and to compare the results from this work with those obtained previously. It was found that for the separations examined experimentally, the snatch technique was the most efficient method.

8.4 OVERALL SUMMING-UP.

The role of the research work has been an endeavour to consider the practical application of a number of modern control trends based on the use of digital computers.

A number of control algorithms were developed, the requisite interface equipment installed on the apparatus, and the control loops commissioned. From this work, it became apparent that significant advantages could be obtained by the use of non-linear controllers. In addition, it was apparent, that by using digital controllers, it was relatively easy to generate control algorithms designed to control given loops efficiently. Finally, this work also included an analysis of the problem of multi-level programming where, during the execution of the programme, the priority level of each subsection had to be considered.

The feed-forward off-line analysis that was carried out was not intended to give detailed predictive information about the behaviour of the column, but, on the contrary, to give an insight into the relative merits of a number of operating techniques.

The practical investigations were sufficient to both illustrate the problems of introducing feed-forward control strategies, and to give an insight into the relative merits of the various modes of operation.

As a direct result of this experimental work, investigations are now proceeding into the merits of using the snatch technique to carry out a batch distillation task. This work will take the form of a theoretical analysis, using a dynamic model of the response of a batch distillation unit when subjected to periodic disturbances. Once the model has been developed, a comprehensive analysis will be undertaken. This will include an investigation into the effect of altering the main process variables of equipment hold-up, the snatch removal frequency, and the magnitude of the snatches. In addition, a range of different distillation tasks will also be examined, using columns with a variety of theorectical plates.

APPENDICES

APPENDICES.

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APPENDIX 1

THE ANALYSIS PROGRAMME

This programme was written in a combination of Ferranti Argus Autocode and Machine Code. Its main purpose was to process signals from the chromatographic unit, and hence to provide an on-line analysis of the samples under consideration.

In addition to determining the compositions of the samples analysed by the chromatograph, the programme was extended to include certain other features which will be considered elsewhere (measurement of the distillate off-take rate etc.).

The organisation and operation of the programme have been illustrated by its flow diagram (Figure A.1.1.) and in a detailed explaination of its machine code sub-routines (outlined on Pages 22[redacted]5)

By referring to the flow diagram the various stages in the analysis programme are clearly demonstrated. The first task is to fix a provisional baseline for the graphical integration. Once this has been done the computer searches for the injection point. This is done by taking readings from the chromatographic unit as frequently as possible, and detecting the 'kick' as a sample is taken (Figure A.1.1., Parts 1 and 2, and Figure A.1.2.).

Once the injection point has been found, there is an initial delay before the first component of the sample, Methyl Cyclohexane, emerges. This delay is of a specific duration. Hence, the time interval is stated as an item of data, and the section timed by use

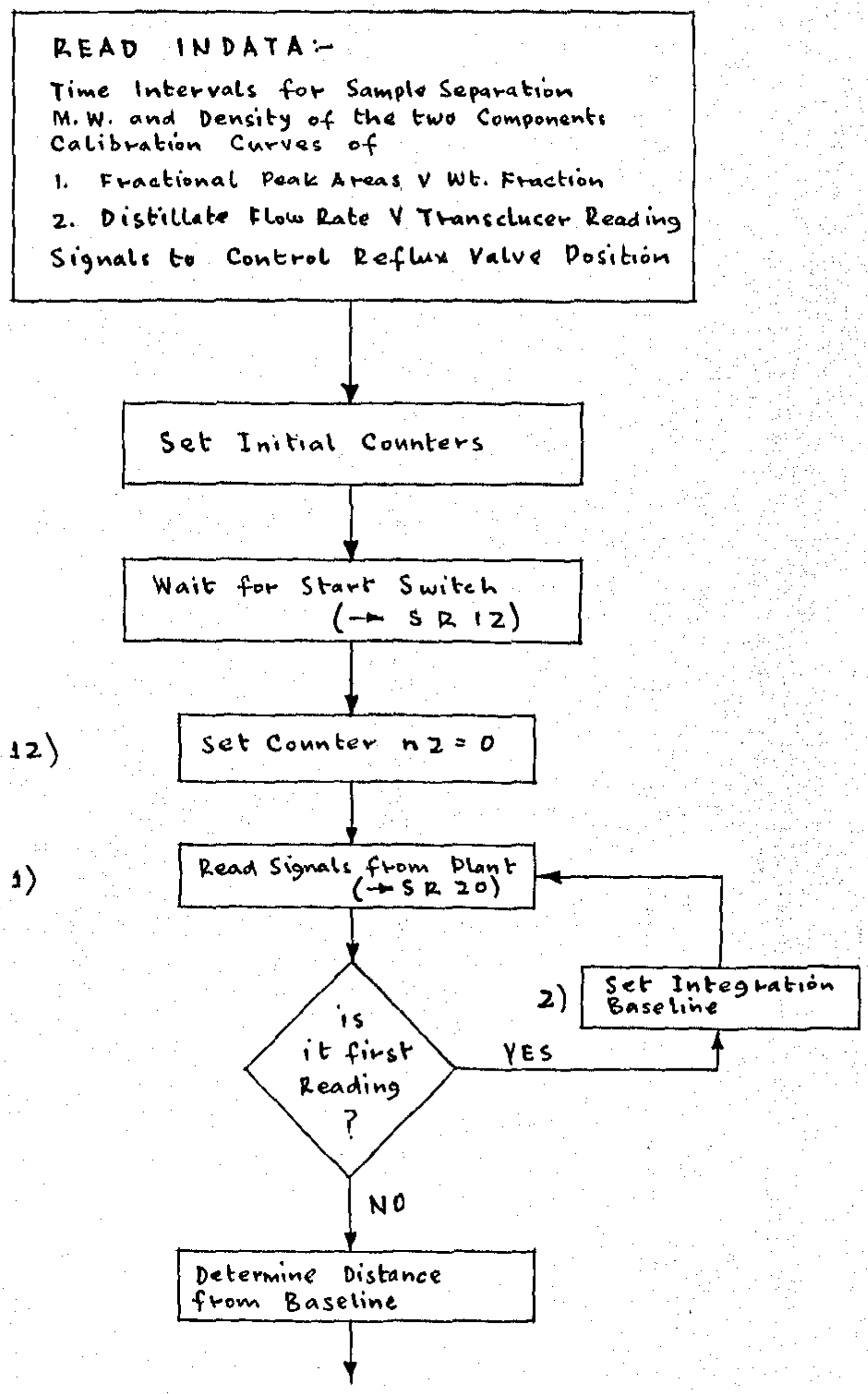


FIGURE A.1.1

FLOW DIAGRAM OF THE ANALYSIS PROGRAMME - PART 1.

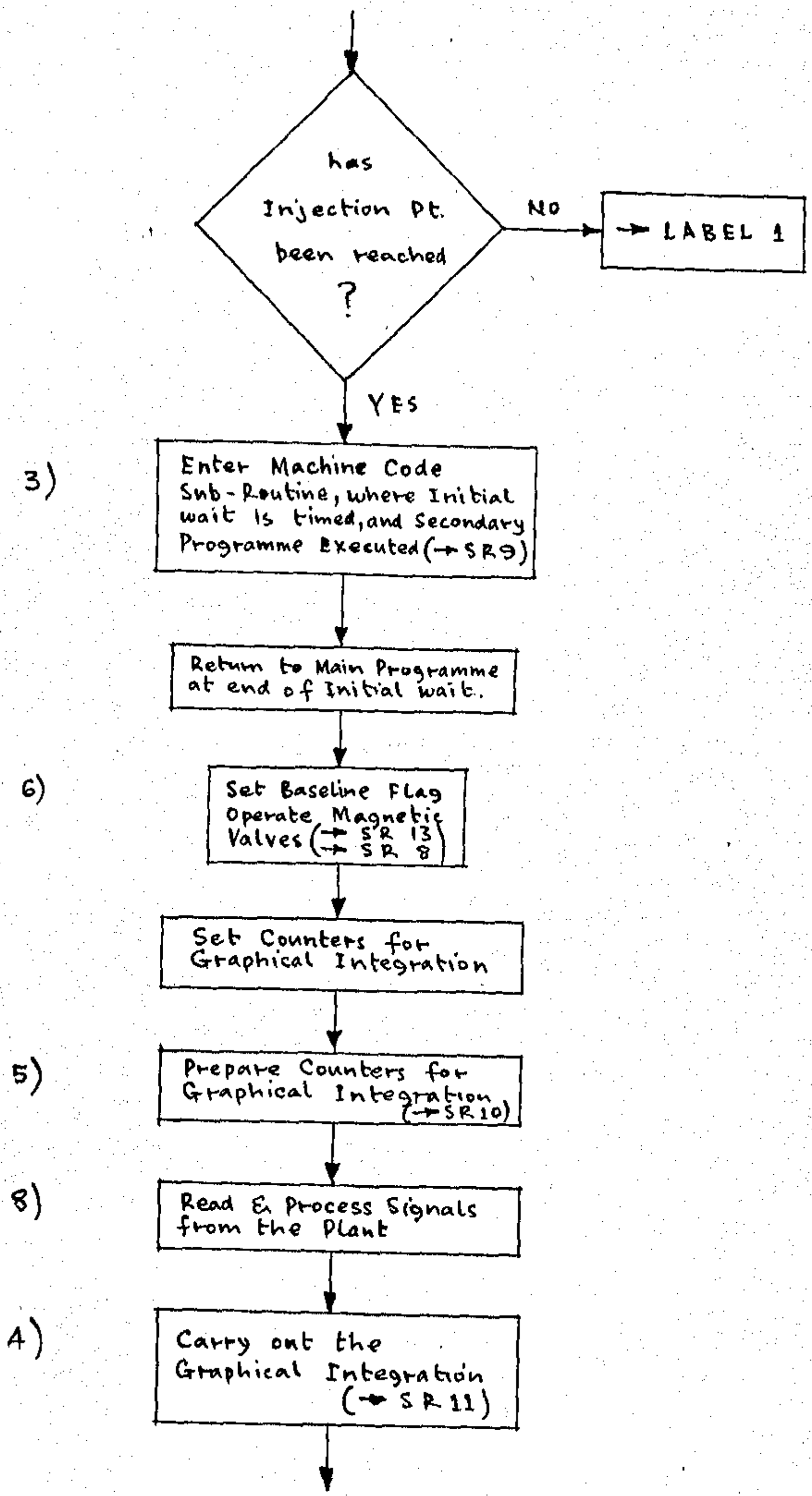


FIGURE A.1.1

FLOW DIAGRAM OF THE ANALYSIS PROGRAMME - PART 2.

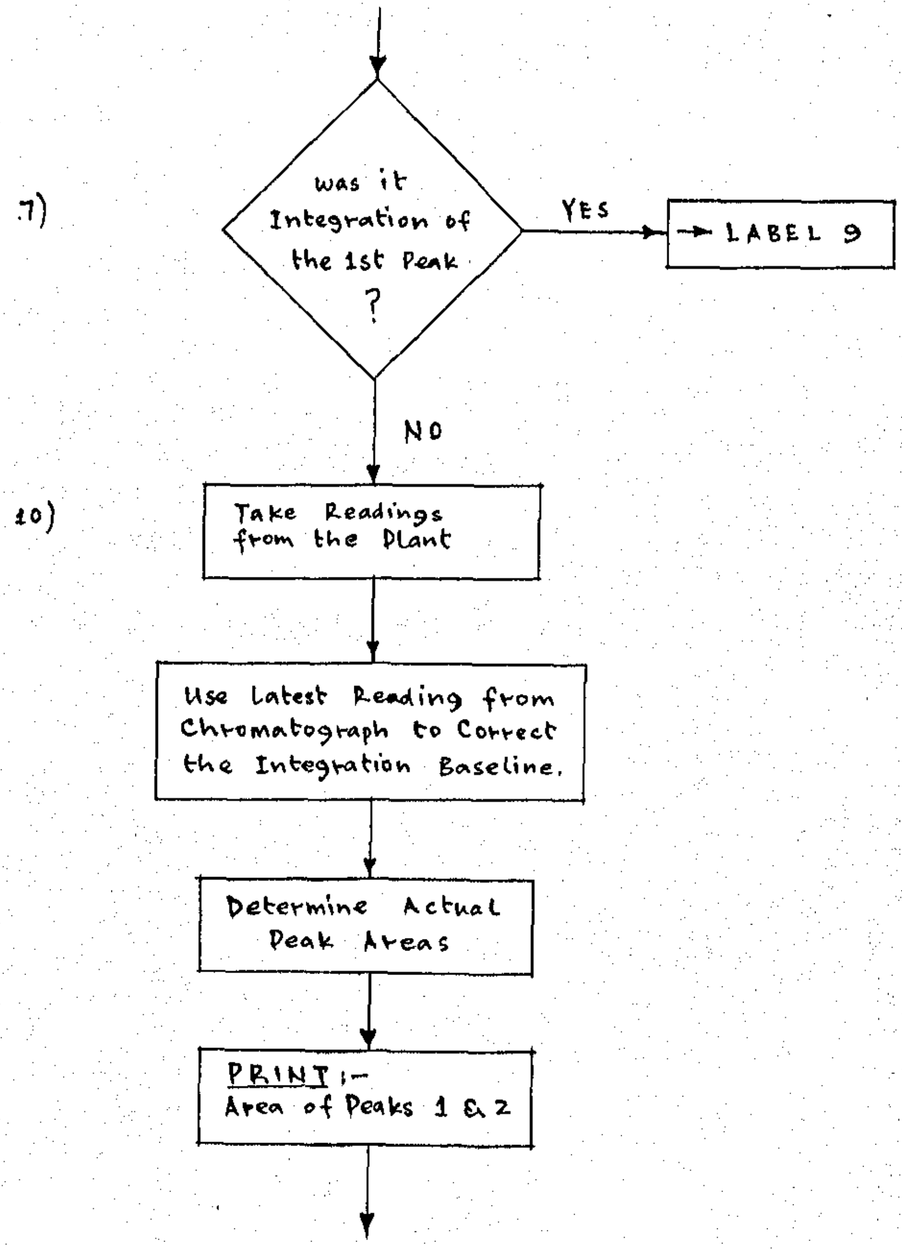


FIGURE A.1.1

FLOW DIAGRAM OF THE ANALYSIS PROGRAMME - PART 3.

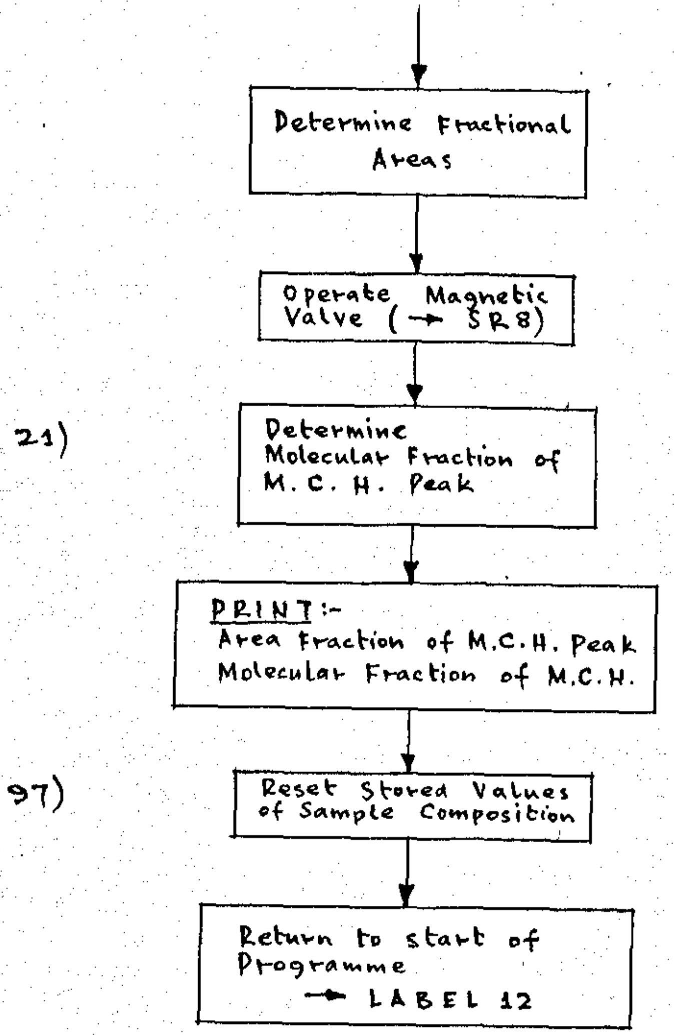
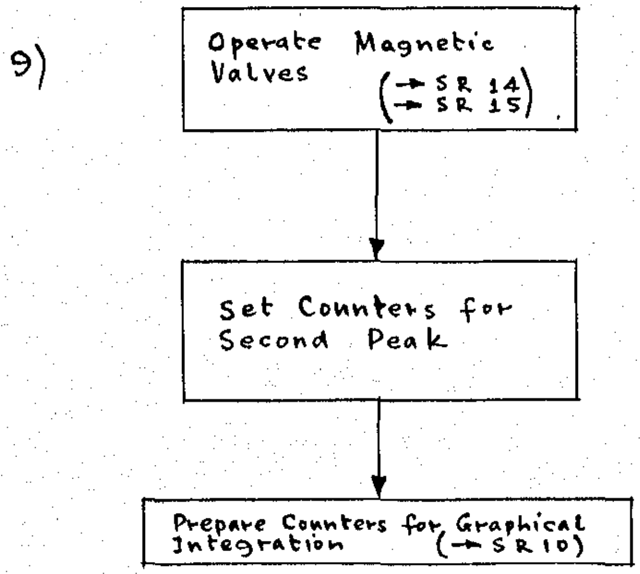
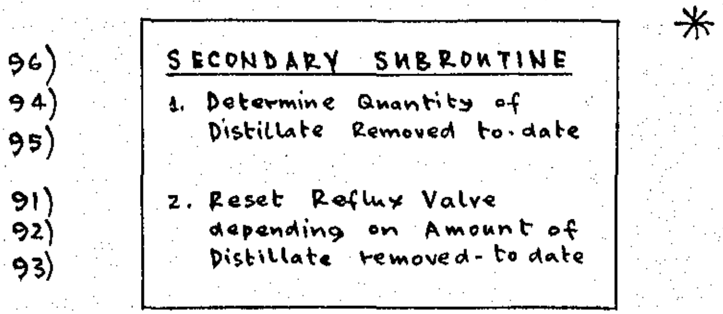


FIGURE A.1.1

FLOW DIAGRAM OF THE ANALYSIS PROGRAMME - PART 4.





* Execution of these Routines is Optional and depends upon the Actual Purpose for which the Programme is being used.

FIGURE A.1.1

FLOW DIAGRAM OF THE ANALYSIS PROGRAMME - PART 5

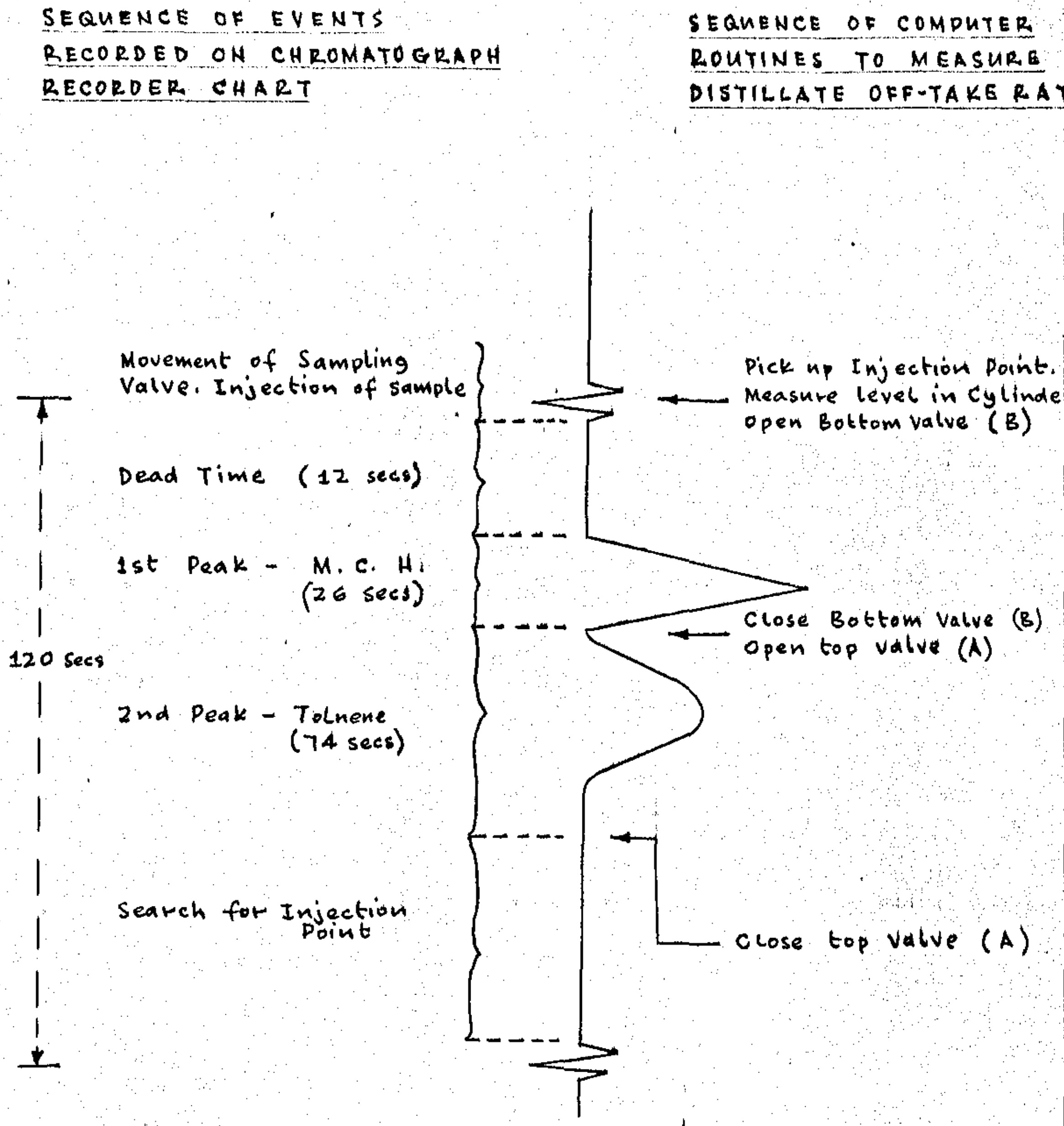


FIGURE A.1.2

TYPICAL CHROMATOGRAPH RECORDER OUTPUT, AND ILLUSTRATION OF THE SEQUENCE OF THE MAIN EVENTS of the interrupt routine. In addition, it is during this section of the programme, that the computer may carry out extra tasks on a priority basis (Section 4.7).

At the end of the initial delay, a graphical integration is carried out to determine the area swept by the Methyl Cyclohexane peak. The duration of this integration is fixed by data fed to the computer, and timed by use of the interrupt routine. (Figure A.1.1., Part 2).

Once the first peak has emerged, the counters are reset, and the same integration routine employed to determine the area swept out by the Toluene peak.

After the areas of the two peaks have been determined, the baseline of the signal from the chromatographic unit is read. This actual baseline is used to correct the two areas previously determined, and is necessary because of differences which may occur when using the two sampling parts in the chromatograph. These actual peak areas are then printed out by the computer (Figure A.1.1., Part 3).

Finally, the fraction of the total area from the Methyl Cyclohexane peak is computed, and by use of a calibration stored in the computer, the mole fraction of Methyl Cyclohexane is also determined. These answers are also printed out by the computer, and a typical printout has been presented (Figure A.1.3.). It can be seen that generally samples are taken in turn from both the distillate line, and the still contents. However, it would of course be possible to sample continuously from either source.

```
        Area of Peak 1                          Area of Peak 2
  Area of Fraction of Peak 1          Mol. Fraction of Component 1


          222.910                              046.400

          000.8277                             000.8989


          098.135                               248.286

          000.2833                              000.3231
```

FIGURE A.1.3

PRINT-OUT FROM BASIC ANALYSIS PROGRAMME

```
N
TWO COMPONENT DIRECT ANALYSIS.
ACTUAL ORGANIC BASELINE.            11/4/67.

V9=2000V4
V18=*20V9
V16=*23V18
V17=*15V16
V20=3V17
V10=*21V20
V11=7V10
V12=*13V11
V21=5V12
V13=*110V21

V14=3V13
V15=3V14
V8=3V15

V=V13
0000 *1420
0000 *1410
0200 *10V7

V=V14
0000 *1400
0000 *1410
0200 *10V7

V=V15
0000 *1421
0000 *1410
0200 *10V7

V=V8
0000 *1401
0000 *1410
0200 *10V7

V=V9
1000 22V3
1300 1


1100 X11
1100 V3
1040 358
1100 25V3

1040 V16
1100 X14
1040 96
0000 *11
0200 *126V7
0000 *25
0000 *11
0200 V
```

```
V=V21

7000 6V3
7300 1
3040 X12
2040 2V
0200 *24V
1040 80
0200 *126V7


+10
+100
+0
+0
+999
+1000
+*11462000
+.999


4000 -1V
7140 X4
7030 X4


7360 -6V
7020 1
0200 4V


4000 -*11V
7140 X4
7030 X4


7220 2V
7000 0
7100 -*21V
7300 23
7370 -*24V
7000 2
7100 X5
5310 18

7100 X4
7310 2
7020 X4
7310 1
7100 X4
7310 2
7020 X4
7310 1

6000 -*37V
6030 X7
6100 -*40V
```

```
6300 23
6370 -*45V
6000 2
6100 X4
4310 14

4120 X5
6100 X4
6310 2
6020 X4
6310 1
7000 -*53V
7030 X6
7310 10

7120 X5
7040 *1777
7153 0
7020 X5
7103 0
0000 *1403
0000 *1403
0000 *1410

0270 X2

V=V18
1100 V17
1000 X0
1100 1V17
1000 X10
1100 2V17
1040 *377
0000 *25
1150 X0

1070 *200
1210 2V
0270 X14
1000 2V17
1070 *17V19
1200 *15V19
1000 1V17
1100 X0

1000 V17
0000 *11
0270 2V17

V=V16
1000 X11
1070 1
1210 3V
1040 6
0200 *126V7
1100 X11
1000 V3
1200 *15V9
```

```
1000 1V17
1100 X0
1000 V17
0000 *11
0270 2V17

V=V17
+0
+0
+0

V=V20
1000 1V3
3000
1070 2
6043 *4112
6310 10
6100 1V
+0
6050 *20000

6250 V
6160 X0
6360 X33
6020 1
6310 1
6103 5V3
3060 1
1210 2V20

0200 *10V7

V=X20
0200 V18

V=V10
1000 23V3
1300 1
1100 X11
1040 8
0200 *126V7

V=V11
1040 3V
1100 X14
0200 *13V9
1000 X11
1070 1
1210 3V
1040 7
0200 *126V7

1100 X11
1040 8
0200 *126V7

V=V12
```

```
1040 *10
0000 *1001
1150 X0
1210 -3V
0200 *10V7

JV1
STOP
N22=TAPE3
V107=TAPE14
V160=TAPE36
V900=TAPE8
N21=115
N27=0
V881=0
V889=.30
V888=.30
V306=0
V307=0
V198=4.5
V199=3.5
V50=N23

V51=N24

N26=N23
→SR12
12)N2=0
1)N1=3
→SR20
V1=N5/100

V3 17=N7/100

V3 07=V317+V307
V3 00=V300+1.0
→2,N2=0
V2=V101-V1

→3,V2>0.2
→1
2)V101=V1
N2=1
→1
3)→SR9
6)N4=101

→SR13
→SR8

N3=2
VN3=0

N23=N26
V0=0
5)→SR10
8)N1=3
→SR20
```

```
V1=n5/100

V317=n7/100
V307=V307+V317
V300=V300+1.0
4)V1=V1-Vn4
V102=V1-V0
V102=V102/2
V102=V0+V102
Vn3=Vn3+V102
V0=V1
→SR11
7)→9,n23=n26
10)n1=3
→SR20
V1=n5/100
V317=n7/100

V307=V307+V317
V300=V300+1.0
V102=V101-V1
V53=V50XV102
V2=V2+V53
V54=V51XV102
V3=V3+V54
V4=V2+V3
PRINTV2,3065
PRINTV3,4065
V2=V2/V4
V3=V3/V4
→SR8

21)n21=n21+1
→21,V2≥Vn21
V103=V2-V(-1+n21)
V104=V(-5+n21)-V(-6+n21)
V105=Vn21-V(-1+n21)
V103=V103XV104
V103=V103/V105
V103=V(-6+n21)+V103
V104=1.0-V103
V105=V103/V109
V106=V104/V110
V106=V105+V106
V103=V105/V106
PRINT V2,3025
PRINT V103,4025
n21=115
→97,n27=1
V889=V103
n27=1
→12
97)V888=V103
n27=0
→12

9)→SR14
→SR15
```

```
n3=3
n23=n24
Vn3=0
V0=0
→SR10
96)V198=V307/V300
V199=n6/100
→96

V300=0
V307=0
V306=0
94)n25=n25+1
→94,V199>Vn25
Vn25=Vn25
V156=Vn25-V(-1+n25)
V157=Vn25-V199

V158=V(7+n25)-V(8+n25)
V157=V158XV157
V156=V157/V156
V156=V(8+n25)+V156
→SR13
n25=159
95)n25=n25+1
→95,V198>Vn25
V159=Vn25-V(-1+n25)
V157=V198-V(-1+n25)
V158=V(8+n25)-V(7+n25)
V157=V158XV157
V159=V157/V159
V159=V159+V(7+n25)


V157=V889/V176
V157=V157XV178
V158=1-V889
V159=V158/V177
V159=V159XV179
V157=V157+V159
V159=V156/V157
V880=V159/2.0
V881=V880+V881
PRINTV881,3045


→93,V881≥V900
n6=V904


→SR21
n0=0
STOP
93)PRINTV889,3025
→92,V881≥V901
n6=V905

→SR21
```

```
n0=0
STOP
92)PRINTV889,3025
→91,V881≥V902
n6=V906


→SR21
n0=0
STOP
91)PRINTV889,3025
→90,V881≥V903
n6=V907


→SR21
n0=0
STOP
90)PRINTV889,3025
TEXT
END
n0=0
STOP
(→0)

115
132
312

.0078
0.00937
98.0
92.0
0.22
0.586
0.69
0.84
0.955
0.18
0.5
0.6
0.76
0.9


2.6
3.1
3.6
4.05
4.5
5.1
5.63
6.2
275.
325.
375.
425.
```

```
475.
525.
575.
625.


.765
.862
98.0
92.0
2.2
3.35
4.9
5.3
6.5
7.8
8.5
9.0
185.
155.
130.
110.
85.
50.
0.
0.

10.0
20.0
30.0
40.0
250.0
280.0
301.0
305.0
X
```

V = V13 ROUTINE TO OPEN BOTTOM VALVE.

```
0000    *1420     Load X(0) with Contents of *1420
0000    *1410     Load X(0) with Contents of *1410
0200    *10V7     Return to next instruction in Main Programme
```

V= V14 ROUTINE TO CLOSE BOTTOM VALVE.

```
0000    *1400     Load X(0) with Contents of *1400
0000    *1410     Load X(0) with Contents of *1410
0200    *10V7     Return to Next Instruction in Main Programme
```

V = V15 ROUTINE TO OPEN TOP VALVE.

```
0000    *1421     Load X(0) with Contents of *1421
0000    *1410     Load X(0) with Contents of *1410
0200    *10V7     Return to Next Instruction in Main Programme
```

V = V8 ROUTINE TO CLOSE TOP VALVE.

```
0000    *1401     Load X(0) with Contents of *1401
0000    *1410     Load X(0) with Contents of *1410
0200    *10V7     Return to Next Instruction in Main Programme
```

V = V9 ORGANISATIONAL ROUTINE FOR INITIAL WAIT AND EXIT TO SECONDARY PROGRAMME.

```
1000    22V3      Load Contents of n22 into X(1)
1300    1         Divide contents of X(1) by 2 ☆

1100    X11       Store Contents of X(1) in X11
1100    V3        Store Contents of X(1) in n0
1040    358       Load 358 into X(1)
1100    25V3      Load Contents of n25 with 358/2 (179) ☆

1040    V16       Load X(1) with V16
1100    X14       Load Contents of X(1) into X14 Link address V16

1040    96        Load 96 into X(1)
0000    11        Clear Lock Out
0200    126 V7    Exit to Autocode Label gien in X(1)

0000    25        Read Interrupt Demand Register
0000    11        Clear Lock-Out
0200    V         Wait at this Address for Interrupt
```

☆ When converting from Autocode to Machine Code, numbers are multiplied by two.

V = V10   RESTORE COUNTER FOR INTEGRATION

```
1000    23V3     Load Contents of n23 into X(1)
1300    1        Divide Contents of X(1) by 2  ☆
1100    X11      Load Contents of X(1) into X11
1040    8        Load X(1) with 8
0200    126V7    Exit to Autocode label given in X(1)
```

V = V11   DECREASE COUNTER - EXIT TO AUTOCODE FIXED BY ITS VALUE

```
1040    3V       Load X(1) with *13V9
1100    X14      Load X14 with contents of X(1)
0200    *13V9    Jump to *13V9
1000    X11      Load X(1) with contents of X(11)

1070    1        Subtract 1 from contents of X(1)
1210    3V       Jump on 3 lines if contents of X(1) not zero
1040    7        Load X(1) with 7
0200    *126V7   Exit to Autocode label given in X(1)

1100    X11      Load X11 with contents of X(1)
1040    8        Load X(1) with 8
0200    *126V7   Exit to Autocode label given in X(1)
```

V = V12   ROUTINE TO ACCEPT SIGNAL FROM START SWITCH

```
1040    *10      Load X(1) with *10
0000    *1001    Load X(0) with contents of *1001
1150    X0       Add Contents of X(0) into X(1)
1210    -3V      Jump Back 3 lines if contents of X(1) are not zero
0200    *10V7    Return to Next Instruction in Main Programme
```

V = X20   INTERRUPT INSTRUCTION

```
0200    V18      Jump to V18
```

V = V17

```
+0
+0
+0
```

☆ When converting from Autocode to Machine Code, numbers are multiplied by two.

V = V18 LINK ROUTINE BETWEEN INTERRUPT AND OTHER ROUTINES.

```
1100   V17     Load V17 with Contents of X(1)
1000   X0      Load X(1) with Contents of X(0)
1100   1V17    Load 1V17 with Contents of X(1)

                                                   Store the Address of
1000   X10     Load X(1) with Contents of X10  )  Next Instruction in
1100   2V17    Load 2V17 with Contents of X(1) )  the main Programme

1040   *377    Load X(1) with *377
0000   *25     Read Interrupt Demand Register
1150   X0      'AND' into X(1), Contents of X(1) and X(0)

1070   *200    Subtract *200 from contents of X(1)
1210   2V      Jump on 2 lines if Contents of X(1) not zero
0270   X14     Jump to Address stored in X14 (link Address)
1000   2V17    Load X(1) with contents of 2V17
1070   *17V19  Subtract *17V19 from Contents of X(1)
1200   *15V19  Jump to *15V19 if contents of X(1) are zero
1000   1V17    Load X(1) with contents of 1V17
1100   X0      Load X(0) with contents of X(1)

1000   V17     Load X(1) with contents of V17
0000   *11     Clear Lock-Out
0270   2V17    Jump to address stored in 2V17
```

V = V16 COUNTER FOR INITIAL WAIT SECTION

```
1000   X11     Load X(1) with contents of X11
1070   1       Subtract 1 from contents of X(1)
1210   3V      Jump on 3 lines if contents of X(1) not zero
1040   6       Load 6 into X(1)
0200   *126V7  Jump to Autocode label given in X(1)

1100   X11     Load X11 with contents of X(1)
10000  V3      Load X(1) with contents of n0
1200   *15V9   Jump to *15V9 if contents of X(1) are zero
1000   1V17    Load X(1) with contents of 1V17

1100   X0      Load X(0) with contents of X(1)
1000   V17     Load X(1) with contents of V17
0000   *11     Clear Lock Out
0270   2V17    Jump to address in 2V17
```

APPENDIX 2

MEASUREMENT OF DISTILLATE COLLECTION RATE

It has been mentioned previously, that a number of techniques for measurement of distillate collection were investigated (Section 4.7).

Basically, the problem was one of measuring the low distillate flow rate accurately and automatically. The method finally developed used a form of level tank device. The measurements from the device were taken and processed by the digital computer, using a sub-routine incorporated in the analysis programme.

This sub-routine was entered on a low priority basis, using a time sharing arrangement. Hence, during free time in the initial wait period of the main programme, it was processed on an interrupted basis (Figure A.2.1.).

The organisation of the sub-routine has been illustrated by its flow diagram (Figure A.2.2.), and a detailed explanation of each line accompanies its print-out (Presented on Page 232). However, to consider in total the techniques for the distillate collection rate measurement, one must consider the whole analysis programme, since routines were added at various stages, to control the arrangements for collecting the distillate.

A typical chart print out from the chromatograph is illustrated in Figure A.1.2, and included on the figure is the sequence of operations used to measure the distillate collection rate.

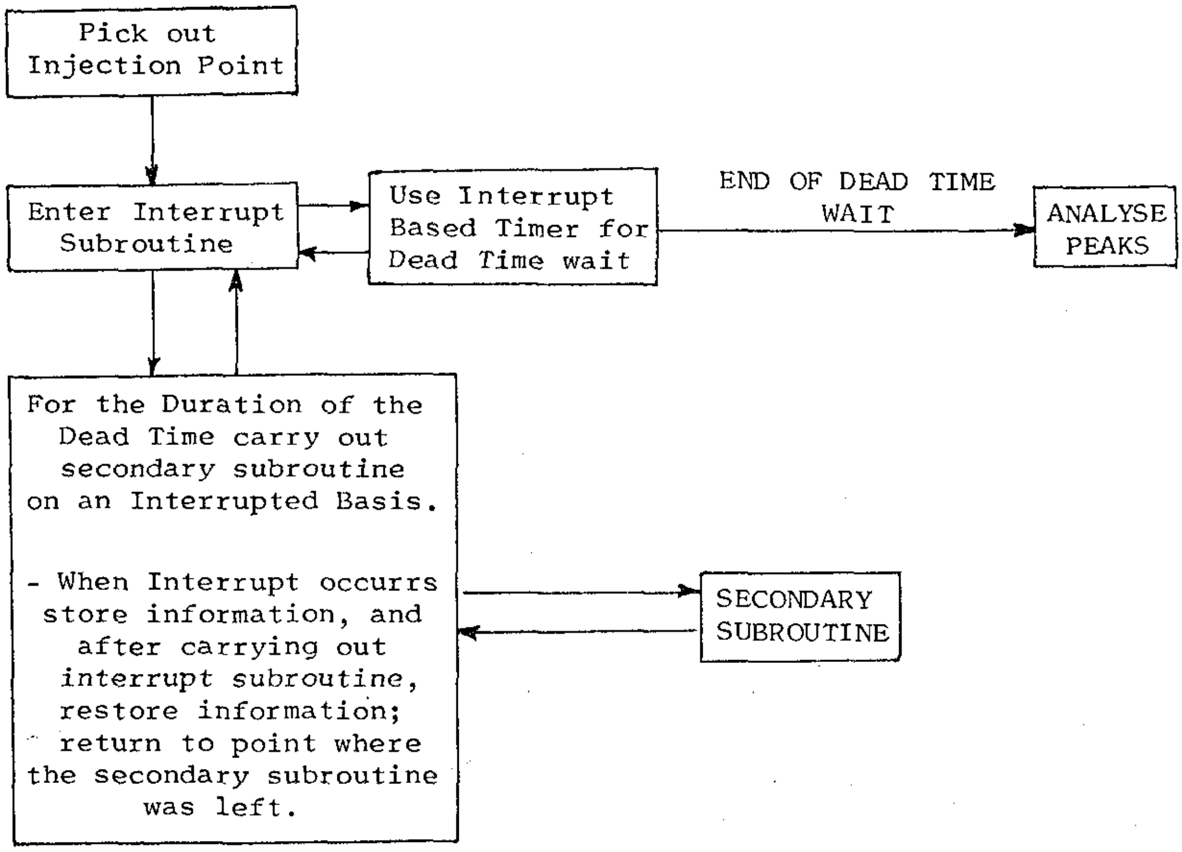


FIGURE A.2.1.

FLOW DIAGRAM INDICATING POSITION IN MAIN PROGRAMME OF THE SUBROUTINE MEASURING THE DISTILLATE COLLECTION RATE.

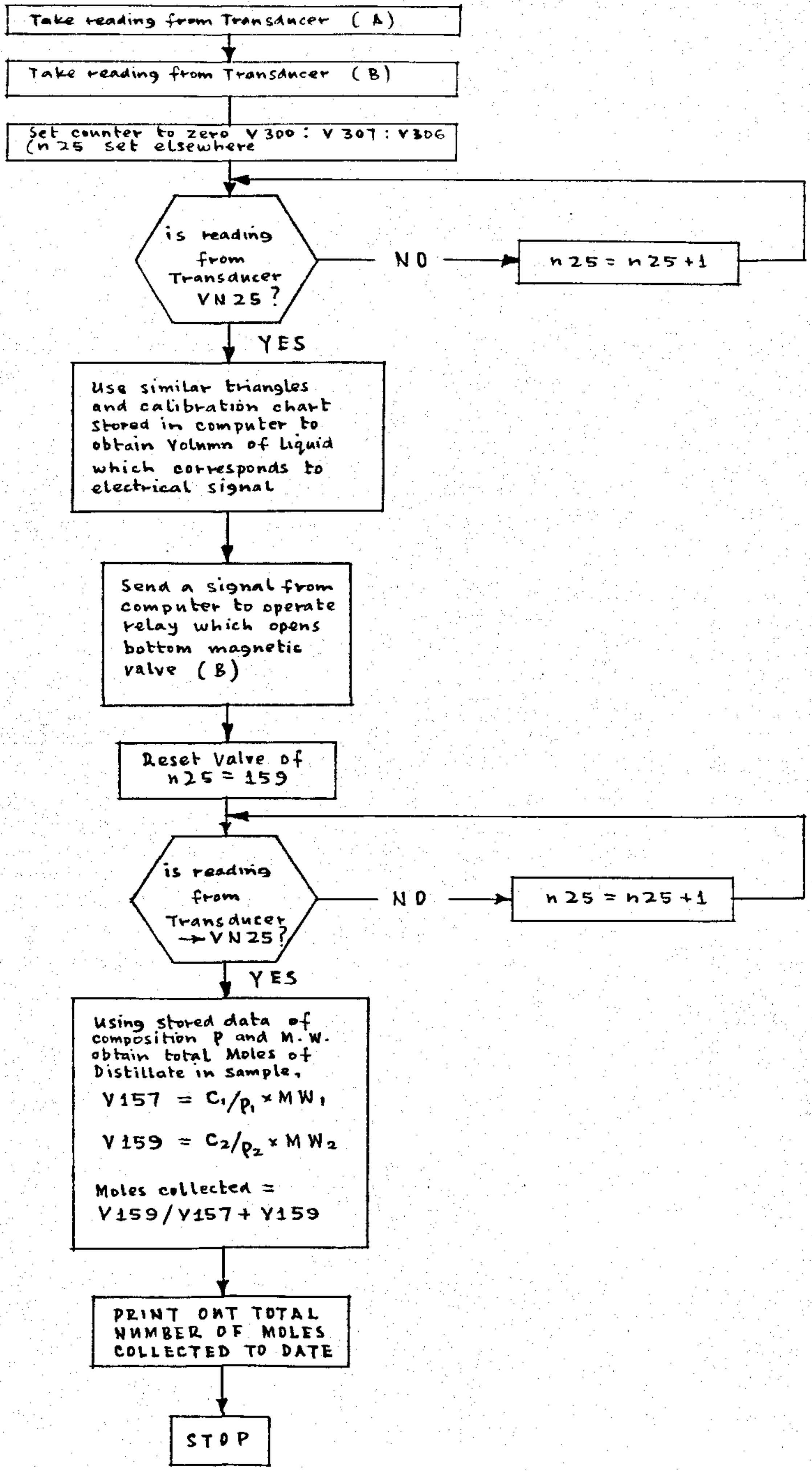


FIGURE A.2.2 FLOW DIAGRAM OF ROUTINE TO MEASURE THE DISTILLATE COLLECTION

Initially, the sub-routine must be fed with start-up data for the first set of readings. However, it then uses those results which it has previously read and processed.

At first, the arrangements for collecting the distillate will be considered in detail.

By referring to Figure A.1.2. it can be seen that after the injection point has been located, there is a dead time before the first peak is transmitted. With the top magnetic valve (A) closed, and while the computer is waiting during this dead time, it reads the signal from the transducer measuring the height of the liquid in the measurement cylinder. After this measurement is taken, the magnetic valve (B), at the foot of the measurement cylinder, is opened by a signal from the computer. This allows the distillate to drain from the cylinder, and collect in the main reservoir.

At the point when the computer decides that the first peak has been transmitted, and the second peak is about to start, it sends out signals to close the bottom magnetic valve (B), and open the top magnetic valve (A). This allows the distillate to collect in the measurement cylinder once more. As soon as the programme reaches the end of the second peak it closes the top valve (A), in order to allow the height in the cylinder to become steady before the cycle is repeated. While the top valve is closed, the distillate is collected in the temporary reservoir above the measurement cylinder (Figure A.2.3.). With this arrangement, approximately 45 seconds are allowed for the cylinder to drain, approximately 75 seconds are

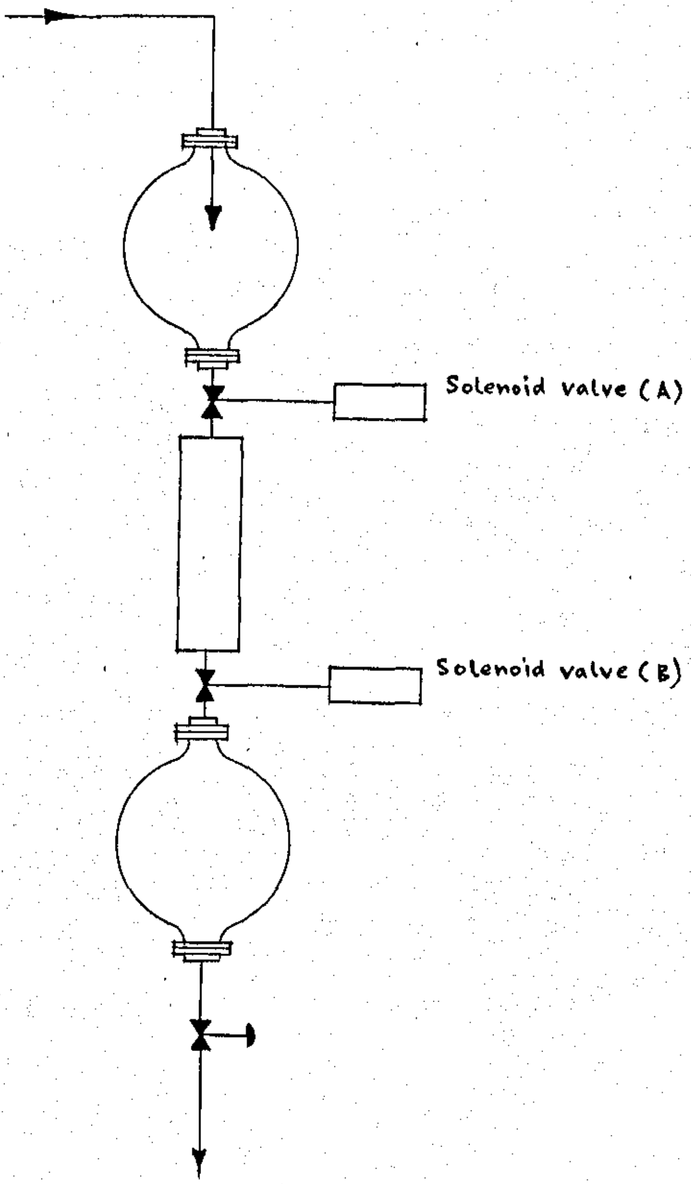


FIGURE A.2.3

SCHEMATIC ARRANGMENT OF THE DISTILLATE COLLECTION APPARATUS allowed for it to fill, and the samples measured at two minute intervals.

Finally, by referring to the sub-routine flow-diagram (Figure A.2.2.), it can be seen that the individual stages in the routine are few. A signal from the transducer indicates the head of liquid in the measurement cylinder, and this signal is converted by the computer into a total volume of liquid. By using data stored in the computer, the routine is able to convert this volume into the rate at which distillate is removed (Moles of Distillate/Minute). This collection rate is printed out by the computer. In addition, there is also a print-out of the total quantity of distillate collected to-date.

```
96)V198=V307/V300              Average of reading from Reflux Transducer.
V199=n6/100                    Reading from Distillate Collection Transducer.
→SR13                          Open bottom magnetic valve (B).

V300=0
V307=0                         Set counters to zero
V306=0

94)n25=n25+1                   Obtain from Store Value  )
→94,V199>Vn25                  of Vn 25  V199 (n25 set  |
                               in main programme).      |  Conversion of
                                                        |  Electrical Signal
Vn25=Vn25                                               |  from Transducer
V156=Vn25-V(-1+n25)            Obtain by Similar Δ's,   }  (A), Volume of
V157=Vn25-V199                 and using calibration    |  Liquid Measure-
V158=V(7+n25)-V(8+n25)         from store, correspond-  |  ment cylinder.
V157=V158XV157                 ing value of Volume of   |
V156=V157/V156                 Liquid in cylinder (=V156))
V156=V(8+n25)+V156

n25=159                        Reset value of counter

95)n25=n25+1                   Obtain from store value  )
→95,V198OVn25                  of Vn 25  V198           |  Conversion of
                                                        |  Electrical
V159=Vn25-V(-1+n25)                                     |  Signal from
V157=V198-V(-1+n25)            Obtain by similar Δ's,   }  Transducer (B),
V158=V(8+n25)-V(7+n25)         and using calibration    |  Meal Value of
V157=V158XV157                 chart from store corres- |  Reflux Flow
V159=V157/V159                 ponding value of Reflux  |  Rate.
V159=V159+V(7+n25)             Return Flow Rate (=V159) )

V157=V889/V176                 Using stored data of
V157=V157XV178                 composition, density
V158=1-V889                    and molecular weight,
V155=V158/V177                 obtain total Moles of
V155=V155XV179                 Distillate in sample
V157=V157+V155                 collected over two
V155=V156/V157                 minute interval.

V880=V155/2.0                  Distillate off-take rate in Moles/Min. (V880)
V881=V880+V881                 Accumulated Total of Distillate Collected
PRINTV881,3045                 PRINT out of Total Number of Moles collected
PRINTV880,4045                 PRINT out Distillate off-take rate

n0=0                           Reset counter for read sub-routine
STOP                           ARREST Programme until interrupt occurs.
```

PRINTOUT AND APPROPRIATE EXPLANATIONS OF DISTILLATE MEASUREMENT SUB-ROUTINE.

APPENDIX 3

DIGITAL 'POSITION' P.I.D. CONTROLLER

A.3 INTRODUCTION

The purpose of the programme is to provide a routine which will implement the sampled-data form of a three term controller. It is written in Astral for use on the Ferranti Argus 108 computer, and may be used as an independent control routine, or included in a larger programme with an appropriate linking routine.

The controller constants and the control algorithm are processed in a manner which allows fast response of the routine. This is achieved by use of fixed point arithmetic, and the 'shift' technique. The shift technique is a method of implementing fast multiplication by a binary number. However, care must be taken to ensure that overflow of the 'word' storage space does not occur.

The input of controller constants is in the form of floating point numbers which are first multiplied by 1,024, and subsequently converted to fixed point integers. These integers are then processed to indicate what shift routines must be employed to give a number equivalent to them.

To carry out the control routine, the error signal is first formed by differencing the set point and the reading from the process variable.

The output signal from the controller positions the control valve, and for this reason must be restricted to an appropriate range.

In addition, to avoid errors like integral saturation, care is taken to ensure that each term is within appropriate limits. Hence, as each term of the control algorithm is formed, a check is carried out to prevent out of range numbers being stored. Each term may have a maximum signal equivalent to an output of + or - 10V, and if these values are exceeded, then the value of the term concerned is brought within these boundaries. Similarly, when the terms are summed the final total is checked to ensure that it is kept within these limits.

The three term controller algorithm may be represented in a sampled-data form as:-

$$V_n = K_1 e_n + K_2 \sum_{r=1}^{r=n} e_r + K_3 \Delta E + V_o$$

where:-

V_n is the valve position after the n th value of the error has been processed.

ΔE is a modified differential of the error function

where:-

$$\Delta E = \tfrac{1}{6} (e_n - e_{n-3} + 3e_{n-1} - 3e_{n-2})$$

e_n is the error at the n th sample interval

The three controller constants are entirely independent of one another, and any of them may be set equal to zero. In addition, the effects of both the integral term and the derivative term are dependent on the sampling frequency, and so these constants must be tuned to suit a particular sampling rate. The relationships with the more conventional constants may be expressed thus:- $K_2 = \frac{T}{T_I}$, $K_3 = \frac{T_D}{T}$

The controller constants all have the following range:-

$K = 0$ or $K = \cdot 001 \rightarrow 15$.

Also included in the programme is a governor to ensure that the maximum allowable step change per sample interval is fixed. This has been included to minimise large disturbances due to changes in controller output.

In addition, the raw process signal may be filtered before use in the controller routine. This is achieved by processing the signal according to the details in Appendix 7.

The controller is timed by an interrupt routine, and is employed at a frequency fixed by one of the controller data items.

Also specified as a data item is the action of the controller. The control routine is first processed, and then an appropriate output routine employed, depending on whether the controller is direct or reverse acting.

A.3.1 SEQUENCE OF OPERATIONS IN THE CONTROLLER ROUTINE.

1. form e
2. form K_1e
3. test K_1e for out of range value
4. if K_1e is outside range then set to maximum permissable value and store
5. obtain previous value of ΣK_2e and update, store ne w value of ΣK_2e and form ΣK_2e

6. test $\Sigma K_2 e$ for excess value, if $\Sigma K_2 e$ is excess then set to maximum and store, otherwise store current value.
7. for differential term, test and limit if necessary
8. Sum the three processed terms of the control algorithm and shift right 10 places (1024)
9. Sum with Vo, test to ensure that sum lies within limits. Modify if necessary.
10. Check action of controller, and output signal to control valve.

A.3.2 ARRANGEMENT OF THE PROGRAMME.

As has been mentioned previously, the controller routine may be employed independently, or incorporated in a larger programme. The arrangement of the controller programme may be seen by reference to its flow diagram (Figure A.3.1). This indicates the employment of a simple single loop controller. *However, this routine may be* modified to include a number of control loops, or for its inclusion in another programme on a time shared basis.

The main data items are as follows:-

1. Action of Controller (0 for direct acting) (1 for reverse acting)
2. Period required for complete traverse of the control valve.
3. Sampling interval, which governs rate at which readings are taken from the process variable.
4. Controller interval, which governs frequency with which control of a loop is updated.
5. Process variable setpoint, this is expressed as a voltage in the range 0-10V.

6. Controller Constants, these are expressed in floating point form.

7. Number of Control loops. For each loop all the previous items of data must be specified.

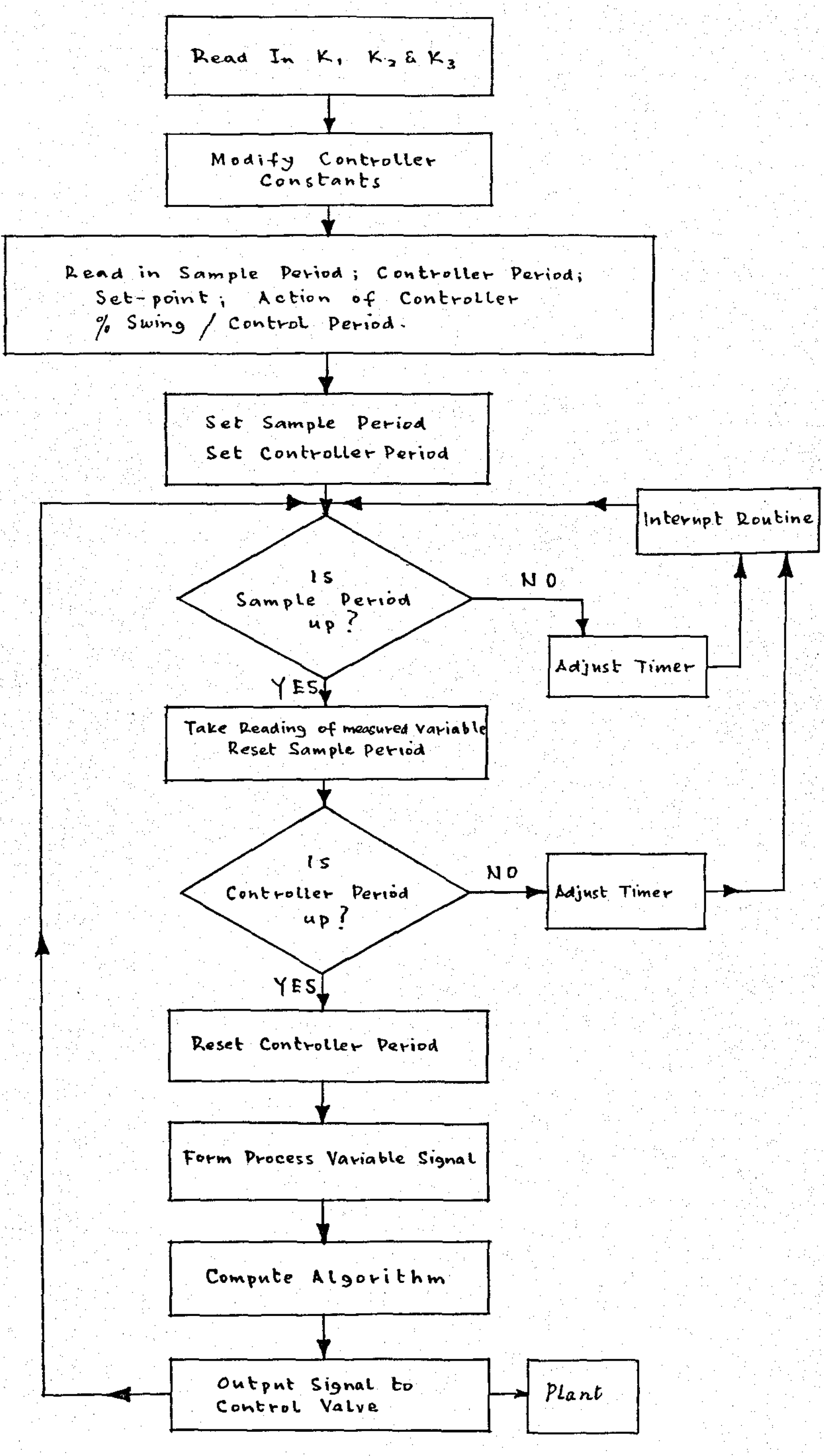


FIGURE A.3.1 FLOW DIAGRAM OF SINGLE LOOP POSITION ALGORITHM CONTROLLER

APPENDIX 4

DIGITAL 'VELOCITY' P.I.D. CONTROLLER.

The velocity algorithm is the basis of another digital controller designed to work with sampled data information. It differs from the position algorithm in that it computes desired changes in valve position, instead of actual valve position.

The basic algorithm is obtained by differencing the position algorithm.

Hence,

$$\emptyset_n = P_n - P_{n-1} \quad \text{-------} \quad (A.4.1)$$

$$\emptyset_n = K_1 (E_n - E_{n-1}) + K_2 E_n + K_3 (\Delta E_n - \Delta E_{n-1}) \text{ --- } (A.4.2)$$

It should be noticed with this algorithm, that although the derivative mode is optional, a small amount of integral action must always be present for stability. This is because the set-point is only present in the integral mode.

This is illustrated by the following equation derived from equation (A.4.2):-

$$\emptyset_n = K_1 (V_n - V_{n-1}) + K_2 (S - V_n) + \frac{K_3}{6} (V_n + 2V_{n-1} - 6V_{n-2} + 2V_{n-3} + V_{n-4}) \text{ --- } (A.4.3)$$

The programme written to implement this algorithm did not include use of the shift technique, but instead, used the conventional multiplication routines.

The controller subroutine was carried out entirely in fixed point arithmetic, since this was faster than working with floating point numbers. However, for convenience, the controller constants were input in the form of floating point numbers, which were first multiplied by 1,024, and then converted to fixed point integers. For this reason, once the output from the controller routine had been calculated, it was scaled down by shifting 10 places to the right (divide by 1,024), before transmission as a modifying signal to the control valve. These scaling arrangements were necessitated by the computation in fixed point arithmetic.

As with the position algorithm, care must be taken to ensure that signals outside the range 0-10V are not generated. The arrangements to check this are somewhat similar to those of the position algorithm. Infact, except for the multiplication routines, and the generation of a movement of control valve position, instead of a totally re-computed valve position, the various stages of the two controllers are comparable.

APPENDIX 5

DIGITAL NON-LINEAR VELOCITY P.I.D. CONTROLLER.

An extension of the velocity algorithm is to use the two readings, e_n and e_{n-1}, to examine the state of the controlled loop.

If one considers the proportional and integral modes of the controller, it can be seen that,

$$\phi_n = K_p (e_n - e_{n-1}) + K_I e_n \qquad \text{-------} \qquad (A.5.1)$$

$$\text{where } \phi_n = P_n - P_{n-1}$$

It is apparent that the proportional and integral terms have the same sign when the controlled variable is moving away from the set point, and the opposite sign when moving towards it. Hence, the output signal may be damped by ensuring that near the set point, the proportional action is always accepted, whereas, at some distance from it, only those proportional terms with the same sign as the integral action are accepted. The control action may be further improved by having a higher proportional gain constant outside the band, and much lower constants within the set-point band.

It may be expressed thus:

$e_n = ADC_{S_n} - ADC_n$ (ADC - Analogue to Digital Convertor)

$$\Delta e = e_n - e_{n-1}$$

Set Point Band = S.B. (say .05 full scale)

If $|e_{n-1}| \leqslant$ S.B. add proportional action into computed change.

If $|e_{n-1}| >$ S.B. do not add proportional action into computed change,

if Δe_n has a different sign to e_n.

If $|e_n| >$ Set Point Band use K_1

If $|e_n| <$ Set Point Band use K_2

By using these arrangements, quite considerable improvements in the control of given loops are possible, particularly, when large overshoots are extremely undesirable.

Once again the control programme was written in Astral for use on the Ferranti Argus 108 computer, and could be used by itself to control a single loop or several loops, or incorporated within a larger programme with an appropriate linking routine. A flow diagram illustrating the logic with which the controller constants are chosen has been presented (Figure A.5.1), and a printout of the basic routines has been included in the ensuing pages.

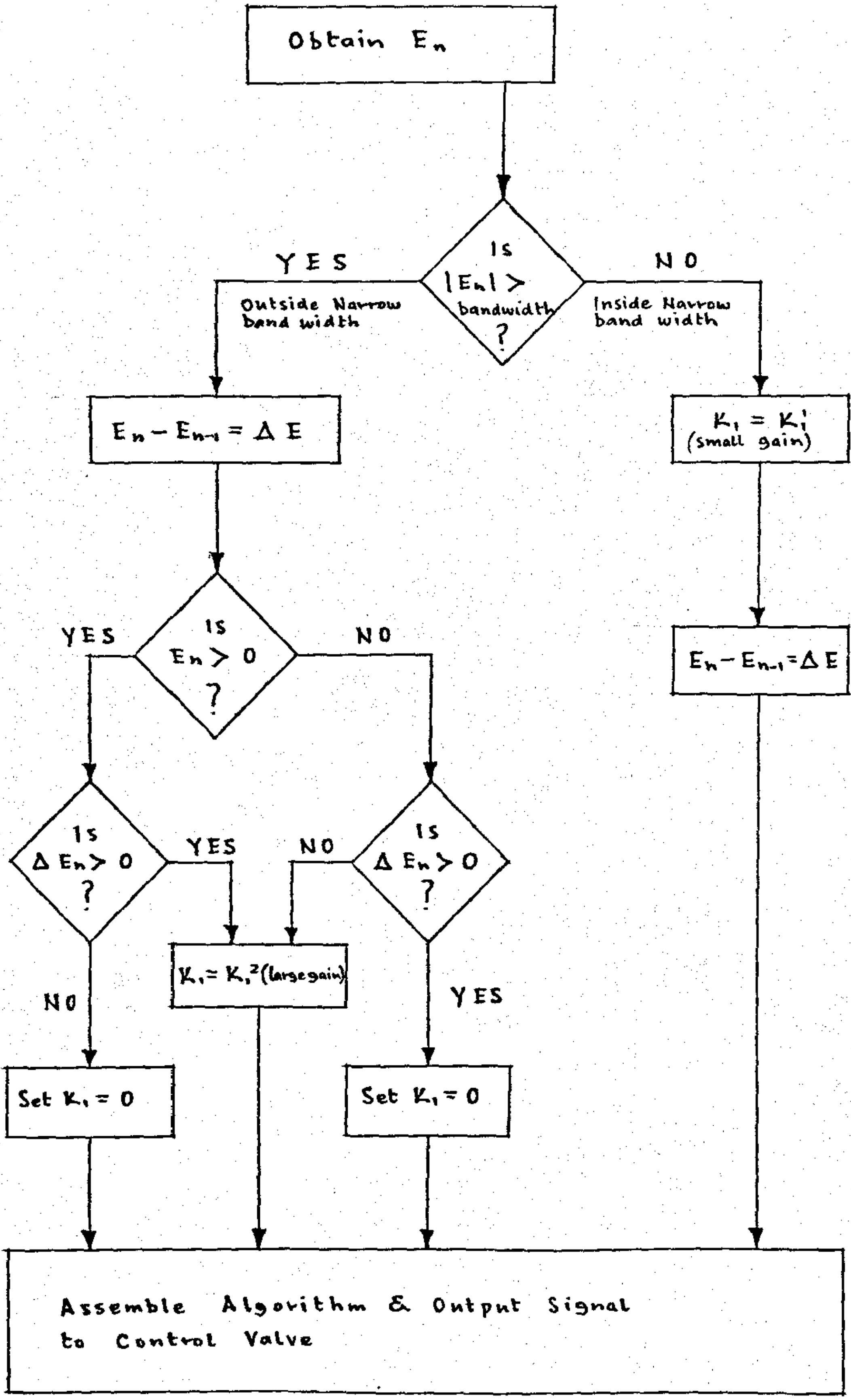


FIGURE A.5.1 FLOW DIAGRAM OF NON-LINEAR VELOCITY CONTROLLER

```
≠TITLE..ΠS.L.VELOCITY WITH BAND FOR PROP CONTROLLERΠ

≠WAIT
≠DATA..SETPOINT
≠DATA..SAMPLE TIMER
≠DATA..PULSE FREQ
≠DATA..PROP CONST
≠DATA..DERIV CONST
≠DATA..INTEGRAL CONST
≠DATA..ESTORE(4)
≠DATA..PLOT DUMP(2)
≠DATA..DERIV DUMP
≠DATA..INTEGRAL DUMP
≠DATA..INTEGRAL STORE

≠DATA..PROPSDUMP
≠DATA..PROPLDUMP

≠DATA..BANDWIDTH
≠DATA..EPRES
≠DATA..OUTPUT
≠DATA..PROPKSMALL
≠DATA..PROPKLARGE

≠DATA..REVERSE

≠LOAD AT..X++14

5 LDX    SAMPLE TIMER

INTERRUPT
0000   *25
0000   *11
0200   V        /JUMP ROUND THIS LINE

1040   *200
0000   *25
1150   XO
1200   INTERRUPT
5  SBC    1
5  JNZ    INTERRUPT     /SAMPLE INTERVAL
0  JZE    READ ROUTINE

≠LOAD AT..*10110
≠ENTER HERE

START
£ PAUSE
£ GO TO..READ DATA
£ CUEREAD..123

INITIAL CONDIT
0   STO    INTEGRAL STORE

0 STO    ESTORE
0 STO     ESTORE+1
0   STO   ESTORE+2
```

```
0 STO    ESTORE+3
5 LDX    SAMPLE TIMER
5 MPI    PULSE FREQ     /DT
6 LDX    INTEGRAL CONST
6 MPI    X5        /KIDT
6 STO    INTEGRAL DUMP
6 LDX    DERIV CONST
5 MPI    6         /6DT
6 DVI    X5        /KD/6DT
6 STO    DERIV DUMP
1 LDX    SETPOINT
1 STO    OUTPUT

ΣLOADFL..PROPKSMALL
ΣMPYFL..⩾128F
ΣFIXFX..23
6 STO   PROPSDUMP

ΣLOADFL..PROPKLARGE
ΣMPYFL..⩾128F
ΣFIXFX..23
6 STO   PROPLDUMP

ΣLOADFL..DERIVCONST
ΣMPYFL..⩾128F


ΣFIXFX..23
6 STO   DERIVDUMP
ΣLOADFL..INTEGRALCONST
ΣMPYFL..⩾128F
ΣFIXFX..23
6 STO   INTEGRALDUMP

RESET
5 LDX   SAMPLE TIMER
Σ GO TO..INTERRUPT

READ ROUTINE
6040 *4010
6310 10
6100 1V
+0
6050 *20000

6250 V
6160 X0
6360 ⩾+1000

6020 1

5 LDX SET POINT
5 SUB X6/SET POINT -SIG(E)
5  STO  EPRES/PRESENT ERROR

DERIV ASSEMBLY
5 JZE   RESET
5 ADD    ESTORE+3     /EN-EN-4
```

```
6 LDX   ESTORE+1       /EN-2
6 ADD   ESTORE+1   /2XEN-2
6 ADD   ESTORE+1    /3XEN-2
4 LDX   ESTORE     /EN-1
4 SUB   X6        /EN-1V3XEN-2

3 LDX   ESTORE+2      /EN-3
4 ADD   X3       /EN-1-3XEN-2+EN-3
4  STO X2
4 ADD   X2        /2EN-1-6EN-2+2EN-3
4 ADD   X5
4 MPI   DERIV DUMP
5 LDX   EPRES
5 MPI   INTEGRAL DUMP

5 ADS   INTEGRAL STORE

PROPORTIONAL
1 LDX  EPRES
2 LDX  X1
1 JGE  2V
1 NLX  EPRES

1 SUB   BANDWIDTH
1 JGE   7V
2 SUB   ESTORE
3 LDX   PROPSDUMP    /SMALL GAIN IN X3
3 MPI   X2
0  JZE   ASSEMBLE

2 JGE   10V      /JUMP IF E +VE
2 SUB   ESTORE
2 JGE   6V            /JUMP IF DE +VE
3 LDX   PROPLDUMP     /LARGE GAIN IN X3
3 MPI   X2
0 JZE   ASSEMBLE

3 LDX   ZERO   /PROP ACTION ZERO
0 JZE   ASSEMBLE

2 SUB   ESTORE
2 JGE   3V     /JUMP IF DE +VE
3 LDX   ZERO   /PROP ACTION ZERO
0 JZE   ASSEMBLE

3 LDX   PROPLDUMP    /LARGE GAIN IN X6
3 MPI    X2

ASSEMBLE
4 ADD   X5
4 ADD   X3
4 JGE  5V
0 SSB  X4
4 SRA  7
0  SSB  X4
0 JZE  2V
4 SRA  7
```

```
4 ADD   OUTPUT
6 LDX   X4

3 LDX   EPRES
3 EXC   ESTORE
3 EXC   ESTORE+1
3 EXC   ESTORE+2
3 EXC   ESTORE+3

SIGNAL OUT

5 LDX  REVERSE

6 JLT  PLOT IS NEG
6 SBC  +999
6 JGE  PLOT TOO BIG
5 JZE  6V
6 STO  X4
4 ADC  +999
4 STO  OUTPUT
0 SSB  X6
0 JZE  B TO BCD
6 ADC  +999
6 STO  OUTPUT



B TO BCD
Σ SETLOOP •• PLOT •• 3 •• 2
6 SRA  23
6 DIV  >10
6 STO••3  PLOT DUMP

6 LDX  Q
Σ REPEAT •• PLOT •• 3

6 SLA  4

6 ADD  PLOT DUMP+1
6 SLA  4
6 ADD PLOT DUMP
6 SLA  10

TRANSMIT

6 STO  *10012
0 LDX  *1403
0 LDX  *1403
0 LDX  *1403


PLOT EXIT
Σ GO TO ••  RESET
```

```
PLOT IS NEG

5 JZE  4V
6 LDC  +999
0 STO  OUTPUT
0 JZE  B TO BCD
6 LDC  0
6 STO  OUTPUT
0 JZE  TRANSMIT


PLOT TOO BIG
5 JZE  5V
6 LDC  0
4 LDC  +999
4 STO  OUTPUT
0 JZE  TRANSMIT
6 LDC  +999
6  STO   OUTPUT
0 JZE  B TO BCD

READ DATA
Σ CLEAR BUFFER
ΣREAD INT..REVERSE


Σ READ INT..SETPOINT
Σ READ INT..SAMPLE TIMER
Σ READ INT..PULSE FREQ
Σ READFL..PROPKSMALL
Σ READFL..PROPKLARGE
Σ READFL..DERIV CONST
Σ READFL..INTEGRAL CONST
Σ READ INT..OUTPUT

Σ READ INT..BANDWIDTH

Σ GO TO..INITIAL CONDIT

≠BREAK
≠END
```

APPENDIX 6

THE SIMPLEX EVOLUTIONERY TECHNIQUE

The technique relies on testing three sets of controller constants. The point which gave the worst control is rejected, and replaced with one whose co-ordinates are a function of the original three. This is repeated until the control problem has been satisfactorily investigated.

Hence, initially, three sets of controller constants chosen using the rules recommended by Kropholler et al (46). The controller performance was determined for each set of constants, by measuring the value of $\int E^2\,dt$ for a step change up and down in the controlled variable. The set which resulted in the worst performance index were then rejected, and new constants chosen by the summation of the respective constants at the best points, and the subtraction of their values at the rejected point.

i.e. with points A_{ny} B_{ny} C_{ny}

$$A_n^1 = B_n + C_n - A_n$$

$$A_y^1 = B_y + C_y - A_y$$

This procedure was repeated until it appeared that the optimal constants had been located.

Two modifications were included in this basic procedure:-

1. If the worst point of the simplex was the new point, then the next worst point was rejected instead.

2. If a particular point was retained for three simplex operations, then its value was recalculated.

APPENDIX 7

PROCESS SIGNAL FILTERS.

A.7.1 ARITHMETIC MEAN.

This was the simplest form of filter considered for processing the raw signal. The arrangement was merely to consider the average signal read into the computer during the sampling interval. This may be represented thus:

$$\hat{S}* = \frac{\sum_{n=o}^{n=N} S_n}{N} \qquad \text{-------} \quad (A.7.1)$$

However, although this results in a relatively smooth signal, there is a time lag on the recorded variable. The usefulness of this filter is governed by the length of the sampling interval, and the speed at which the measured variable changes.

A.7.2 MOVING ARITHMETIC MEAN.

It is often useful to modify the previous filter to give a continuously updated mean value. The filter may be represented by the following equation:

$$\hat{S}_n * = \hat{S}_{n-1}^{*} - S_{n-a} + S_n \qquad \text{--------} \quad (A.7.2)$$

where a is the number of readings over which the signal is averaged.

A.7.3 WEIGHTED MEAN.

This form of digital filter merely weights the present signal, by a factor proportional to the previous valve. The processed signal may be represented thus:

$$S_n^* = \lambda S_{n-1}^* + (1 - \lambda)\, S_n \qquad \text{-------}\ (A.7.3)$$

where S_n^* and S_{n-1}^* are the present and previous weighted means values, S_n is the signal at any time n, and λ is the weighting factor.

Use of this filter still results in a constant lag in the recorded signal, but there is some reduction in this error. In addition, spurious readings have a markedly adverse effect on the recorded signal.

A.7.4 WEIGHTED LINEAR REGRESSION

The basis of this filter is the use of a best fit curve on the present and previous readings, and the use of this to predict the most likely present value.

The curve designated by equation A.7.4 may be used, where a and b must be chosen to suit the signal.

$$S = at + b \qquad \text{-------}\ (A.7.4)$$

The filtered signal is of the form

$$S_n^* = \lambda S_{n-1}^* + (1-\lambda)^2 \left(\frac{2Sn}{1-\lambda} - \sum_{i=o}^{i=n} \lambda^i S_{n-i} \right) \qquad \text{-----}(A.7.5)$$

Using this form, spurious signals still have a marked effect on the filtered signal.

However, their effect may be reduced by a weighting operation on the deviation of the present value from the weighted mean value.

The filtered signal may then be represented by the equation

$$S_n^1 = S_{n-1}^* + a_{n-1} \quad T - \frac{\alpha \delta n^*}{\alpha + \delta n^{*\ 2}} \qquad \text{-------} \quad (A.7.6)$$

where δ_n^* is the predicted valve of δ_n and α is a tuning constant for the filter.

This filter is explained in detail by Fowles and Smith (82). Its principal valve is that the smoothed signal does not lay behind the true signal. This is especially significant when the signal is changing rapidly.

A.7.5 FIRST ORDER STAGE

When this filter is employed, the raw signal is fed through the model of a first order stage. The filter may be illustrated by reference to equation (A.7.7).

$$\frac{dx}{dt} = \frac{Q}{V} \ (F - x) \qquad \text{-------} \qquad (A.7.7)$$

The raw signal is represented by F, and the filtered signal by x. The ratio $\frac{Q}{V}$ must be tuned to suit the nature of the raw signal.

APPENDIX 8

THE PROGRAMME USED TO DETERMINE THE OPTIMAL STRATEGY FOR A BATCH DISTILLATION TASK

A.8 INTODUCTION

This programme was produced in Argus Autocode for use on the Ferranti Argus 108 computer. It was written to optimise the operation of a batch distillation still by predicting an optimal reflux strategy, and used Dynamic Programming to carry out this task. The manner in which this was done has already been explained in the main text (Section 6.5). However, this Appendix will describe in more detail the individual stages of the programme. Since the programme may readily be sub-divided into four sub-sections, a detailed explanation of each of these will be given in turn.

A.8.1 THE STEADY-STATE MODEL

This section of the programme is an analysis of the separation achieved within the column. To do this, a steady-state model of the process is used, the basis of which is a two stage search routine. The first part of the search routine is a Mc-Cabe Thiele analysis of possible separations, and is detailed in Stage 1 of the programme flow diagram (Figure A8, Stage 1). The second part of the search routine is dependent on which stage the batch distillation task has reached, and is a mass balance analysis. This step occurs in Stages 2, 3 and 4 of the programme flow diagrams. (Figure A8, Stage 2, Part 1; Stage 3, Part 1; and Stage 4, Part 1). The search routine compares these analyses, and when suitable agreement between them is obtained, allows the programme to move onto the next section.

It is therefore used as a prelude to all the subsequent sections which merely analyse the results from the model, and, where necessary, set the initial parameters to it.

A.8.2 REMOVAL OF THE FIRST BATCH OF DISTILLATE

For this initial analysis, the computations are started from a fixed charge of material into the system. Hence the analysis requirement is merely to determine the resultant still composition after an appropriate amount of distillate has been removed. This is determined for the range of reflux ratios specified by the input data, and the results are both printed out, and stored in the computer memory. (Figure A8, Stage 2, Parts 1 and 2). In addition, the maximum and minimum values of X, are stored ready for use in the subsequent stages.

A.8.3 REMOVAL OF THE INTERMEDIATE BATCHES OF DISTILLATE

This sub-section is the most involved since it neither starts from a fixed point nor finishes at one. Infact, it uses two starting points which it obtains from the previous analysis — the Maximum and Minimum values of X_{n-1}.

From these two starting points, the range of permissable values of X_n are computed, using the range of reflux ratios specified by the input data. (Figure A8, Stage 3, Parts 1 and 2).

An analysis is then carried out to determine the best reflux strategy with which to achieve a given separation. (The criterian here being the reflux strategies required for a range of X_n). The first time this stage is used, this is achieved by referring to the values of X_1 and R_1 in store. Hence, for a given X_2, the combination of R_1 and R_2 whose sum is the minimum is stored as the best strategy. For subsequent reiterations through this stage, the latest results are compared with the reflux ratios from the store. Hence, for a given X_n, the minimum value of $FR_{n-1} + R_n$ is determined (Figure A8, Stage 3, Parts 3,4,5 and 6).

The results from this analysis are both stored in the computer memory, and printed out.

This sub-section may be repeated as often as is desired, the reiterations through it being fixed by the number of stages into which the distillation task has been split. At the end of each reiteration, the maximum and minimum values of X_n from the reflux strategy analysis are stored ready for the next analysis, as are also infact, various other counters (Figure A8, Stage 3, Part 7).

A.8.4 REMOVAL OF FINAL BATCH OF DISTILLATE

The main task of this stage is to determine the final reflux ratio which gives the overall optimum reflux strategy.

This is determined by ascertaining the value of X_{n-1} which corresponds to a given final reflux ratio (this is possible since the final still composition is fixed). The value of X_{n-1} thus obtained is then used to obtain the corresponding value of the reflux strategy for the previous sections. The overall reflux strategy is determined by summing the reflux ratio, and the reflux strategy from store.

This process is repeated using lower reflux ratios, until the optimum values of the reflux strategy is obtained. (Figure A8, Stage 4, Part 1 and 2).

Once the optimum reflux strategy has been obtained, by referring to the numbers in the computer store, all the intermediate values of the optimum strategy may be obtained (Figure A8, Stage 4, Parts 3, 4 and 5).

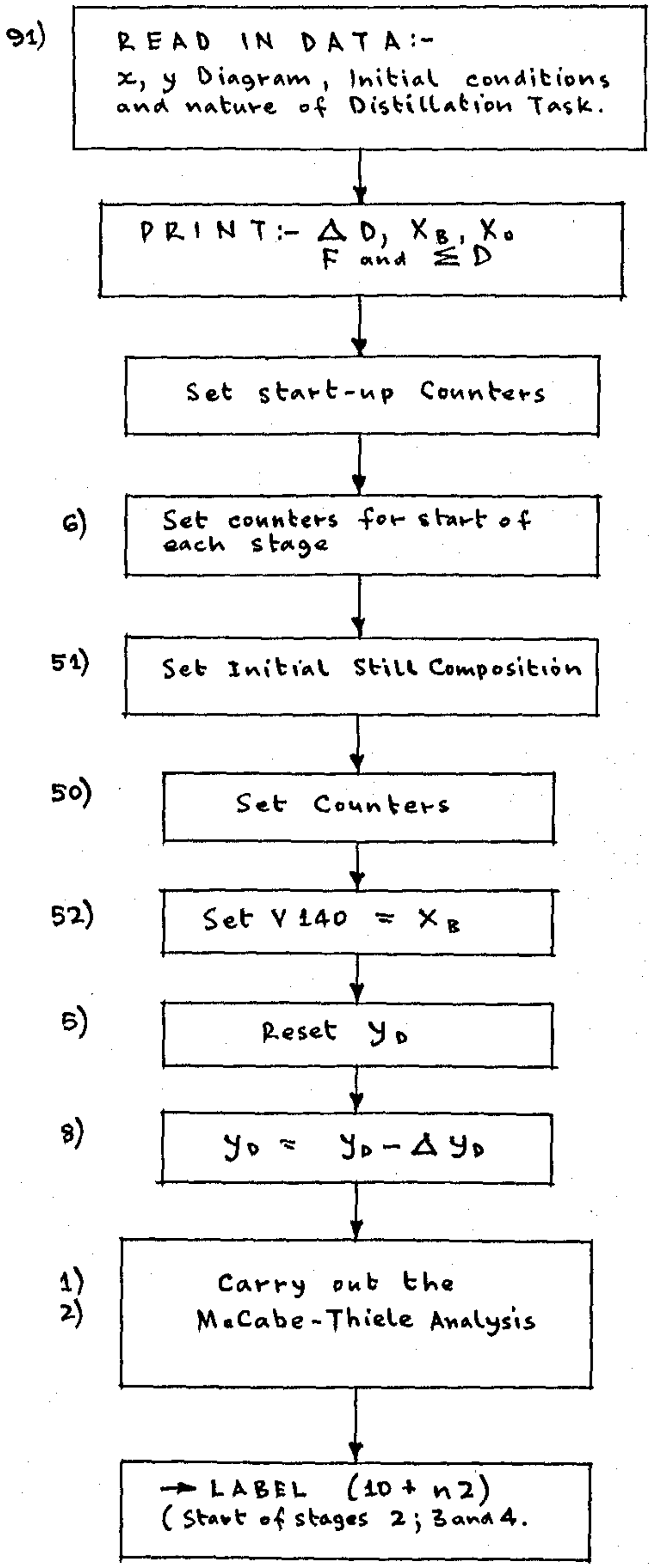


FIGURE : - A8

FLOW-DIAGRAM OF OPTIMIZATION PROGRAMME - STAGE 1.

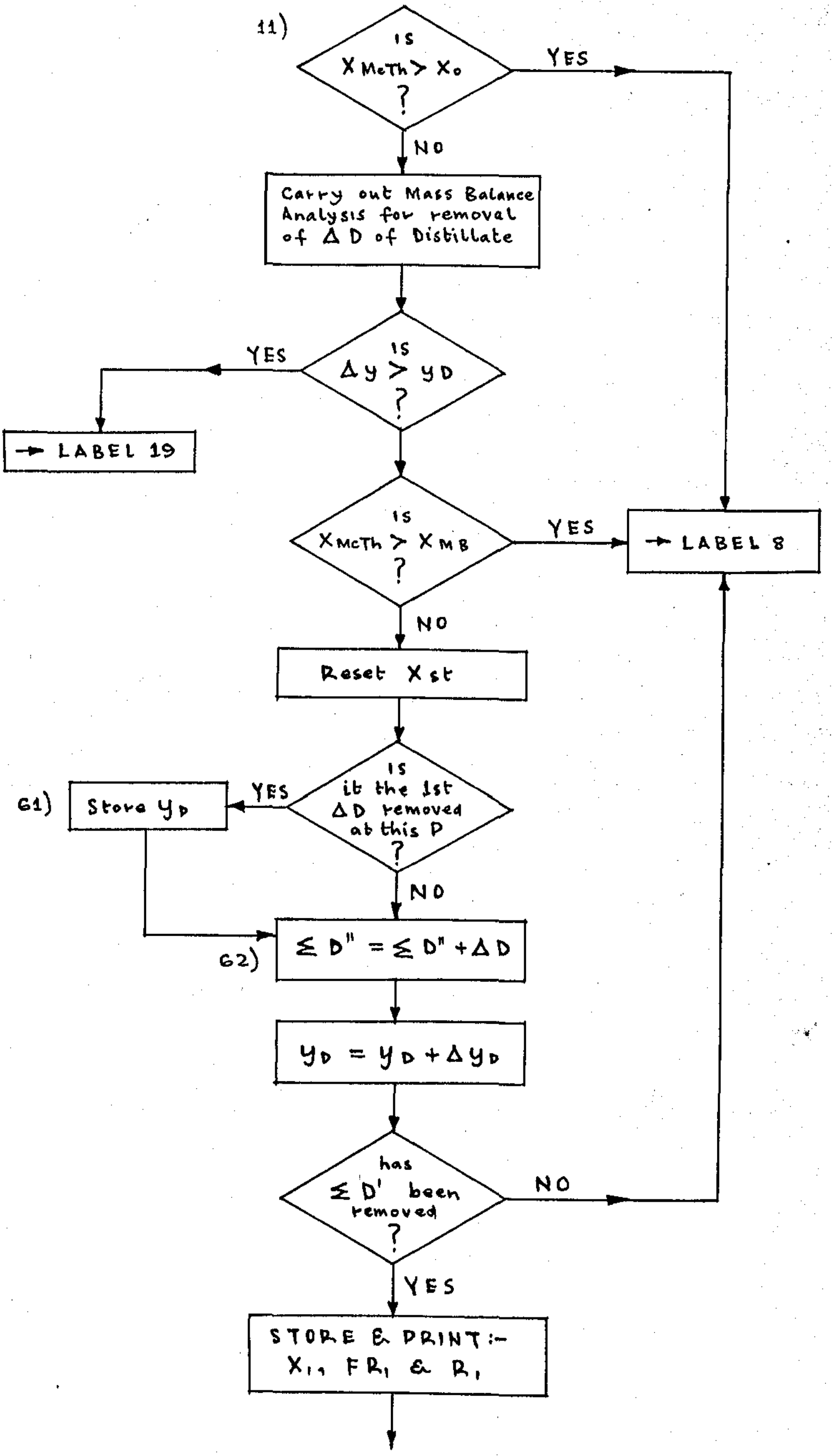


FIGURE : - A8

FLOW DIAGRAM OF OPTIMIZATION PROGRAMME - STAGE 2, PART 1.

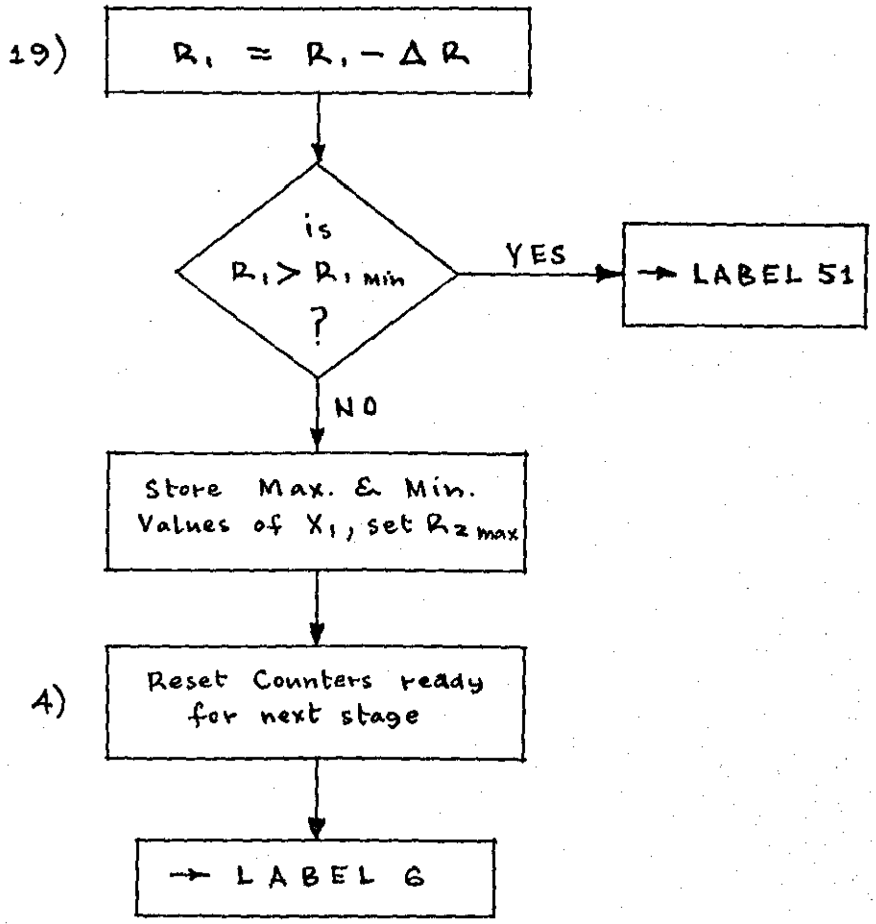


FIGURE : - A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 2, PART 2.

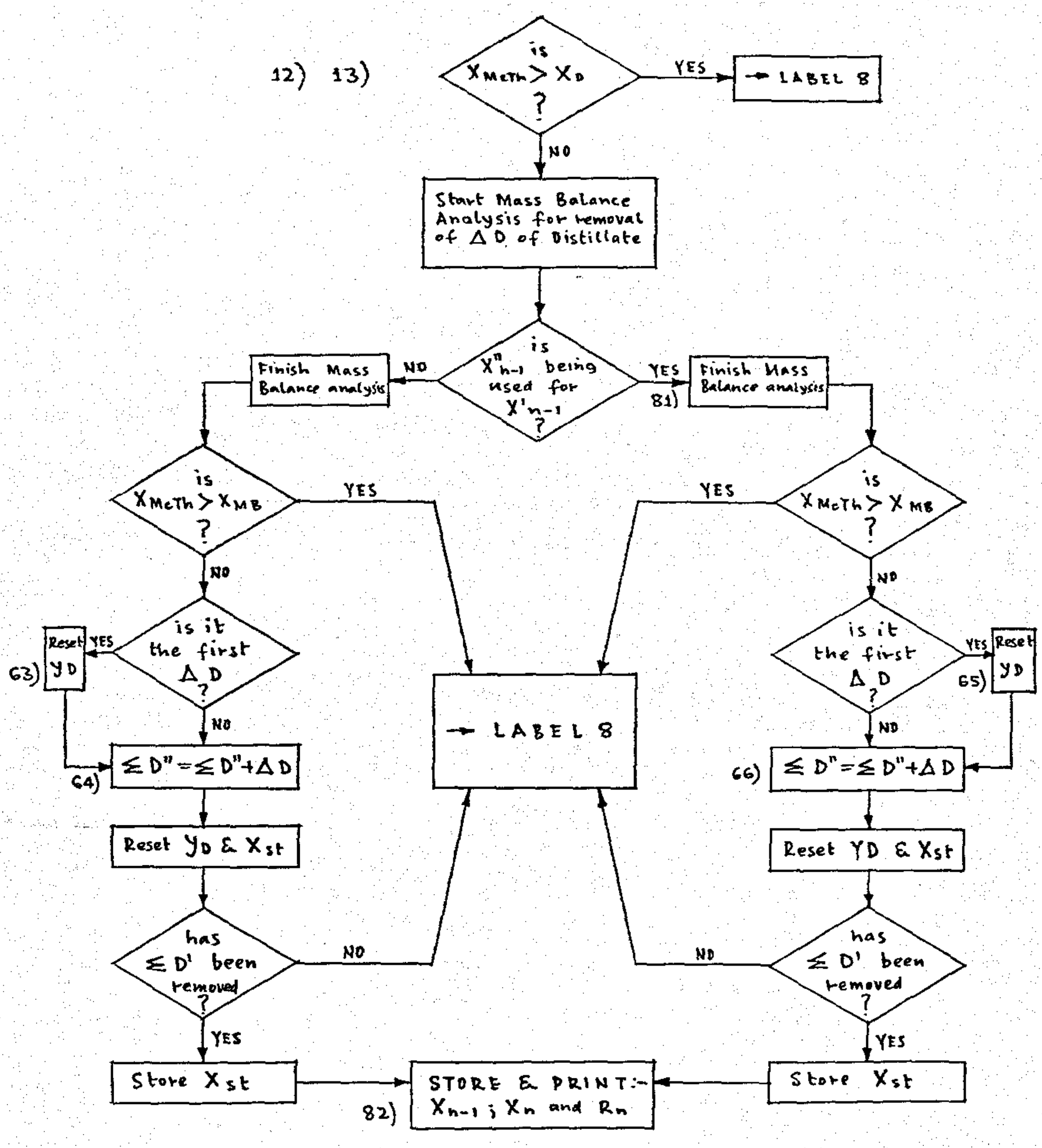


FIGURE :- A8

FLOW DIAGRAM OF OPTIMISING PROGRAMME - STAGE 3, PART 1.

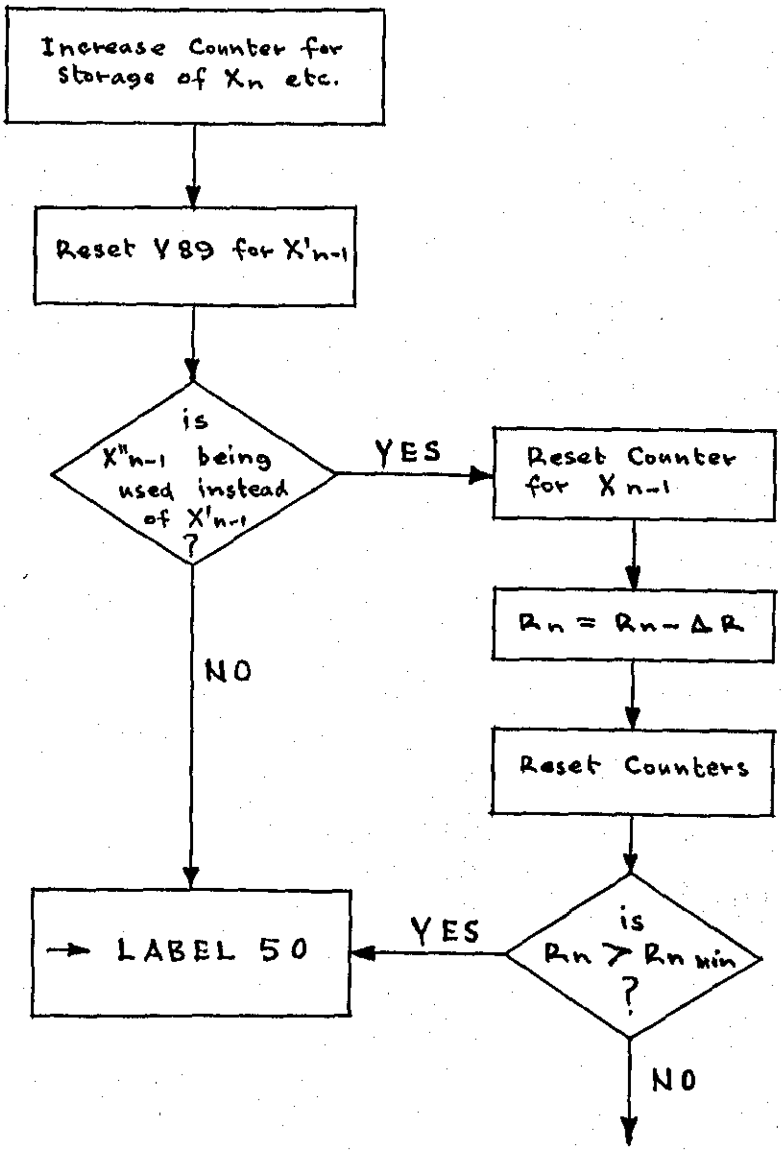


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 3, PART 2.

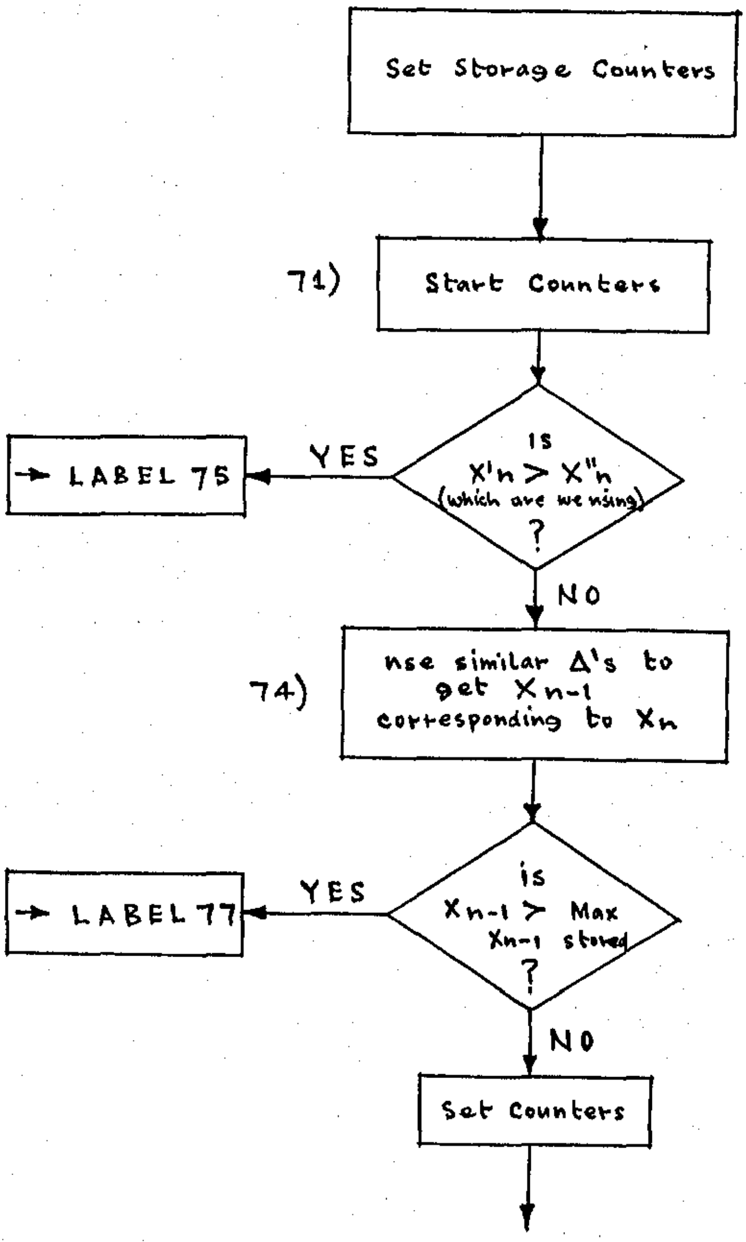


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 3, PART 3.

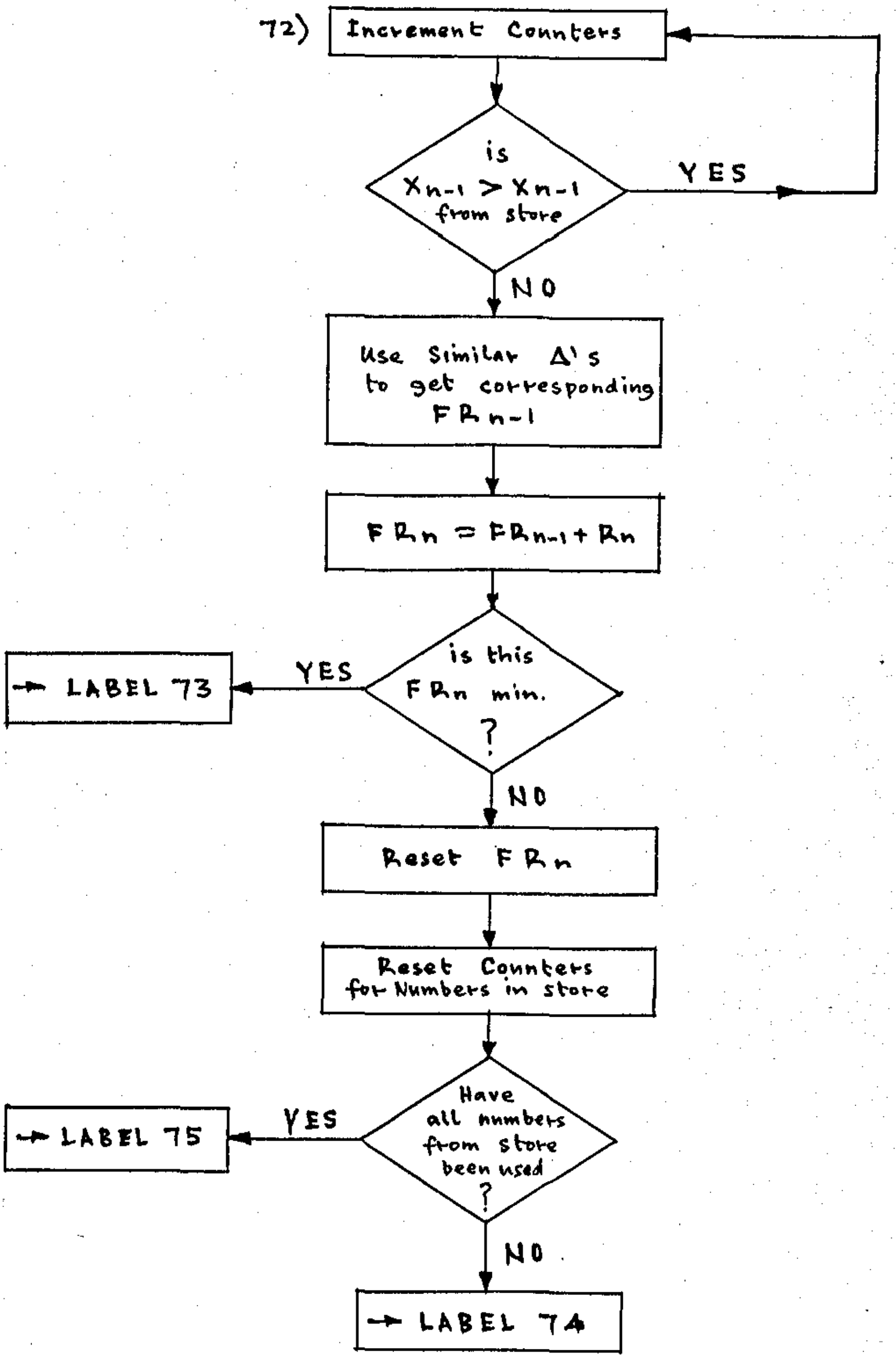


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 3, PART 4.

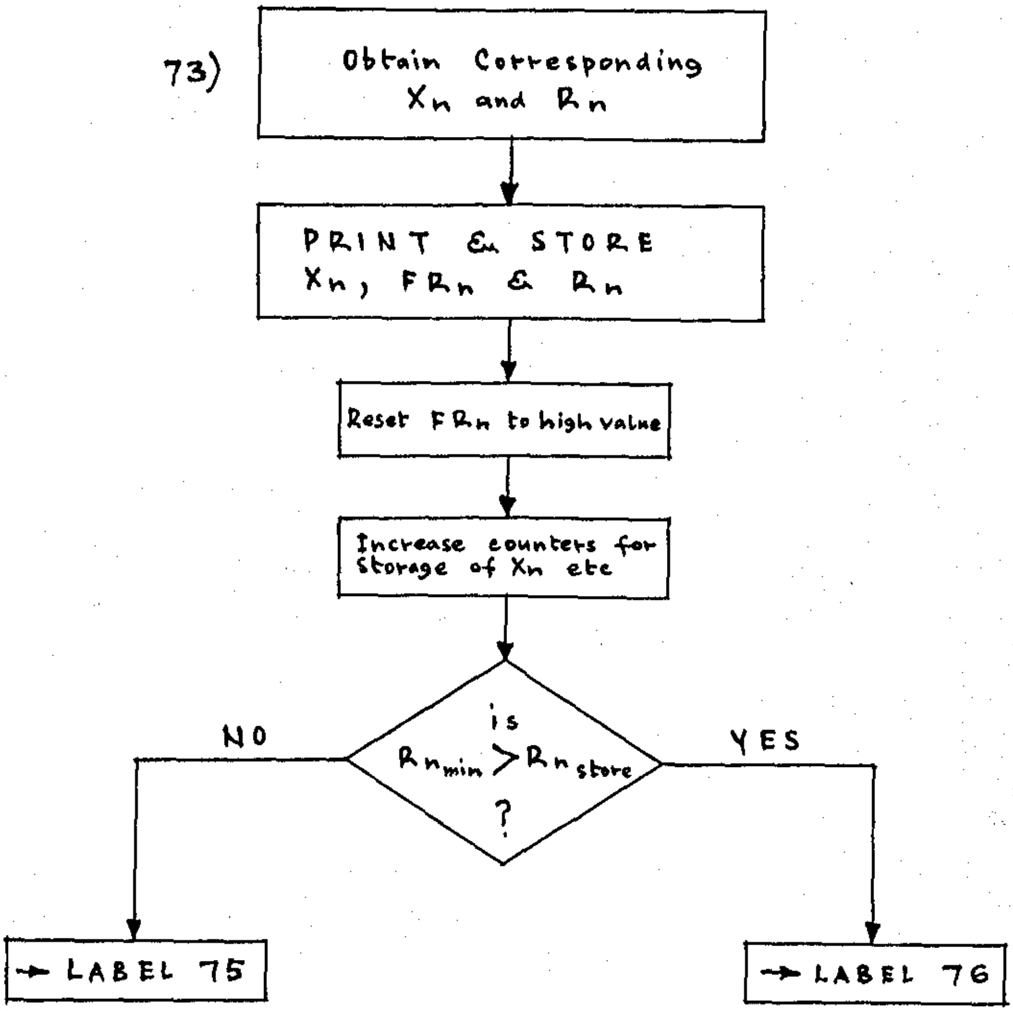


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 3, PART 5.

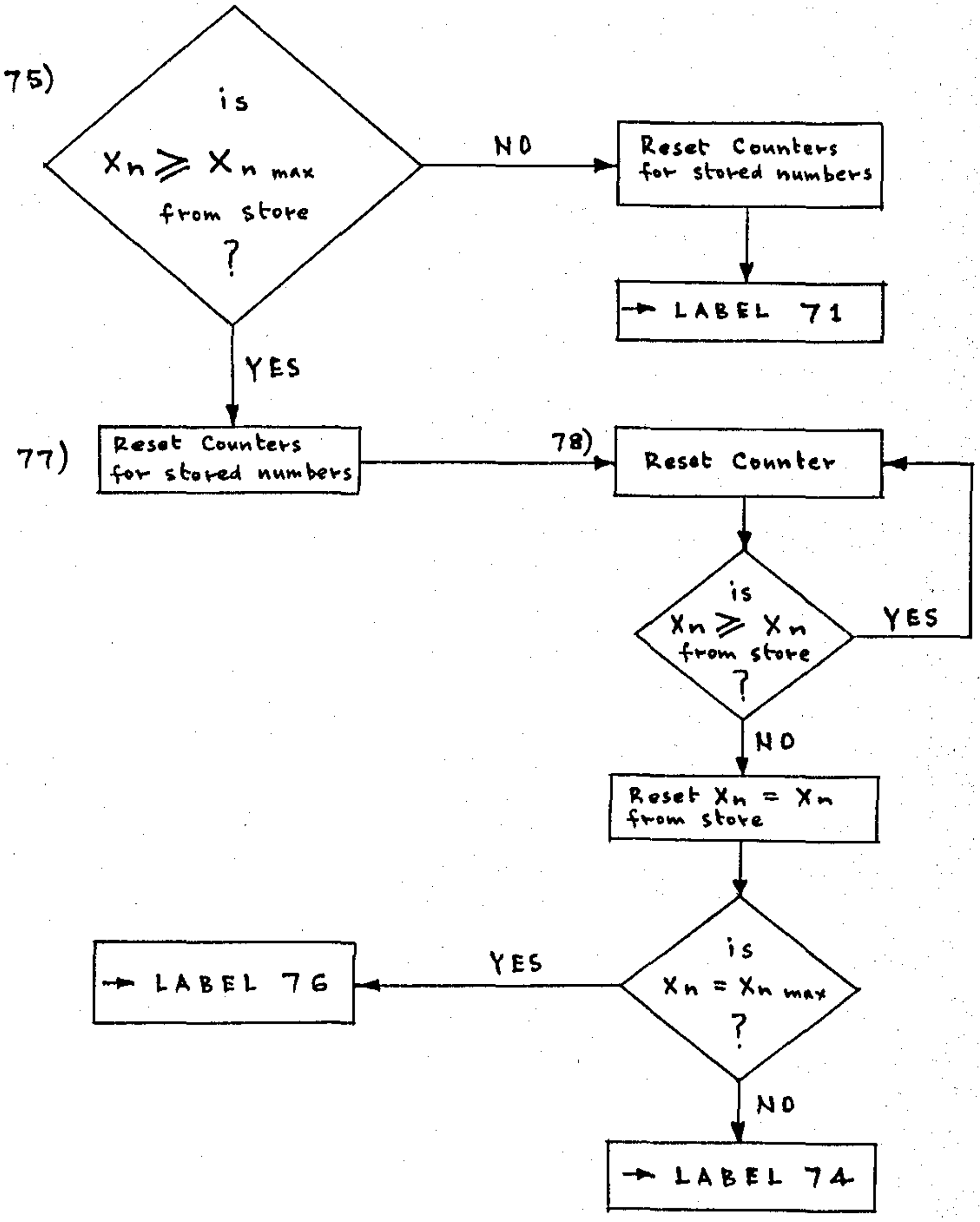


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 3, PART 6.

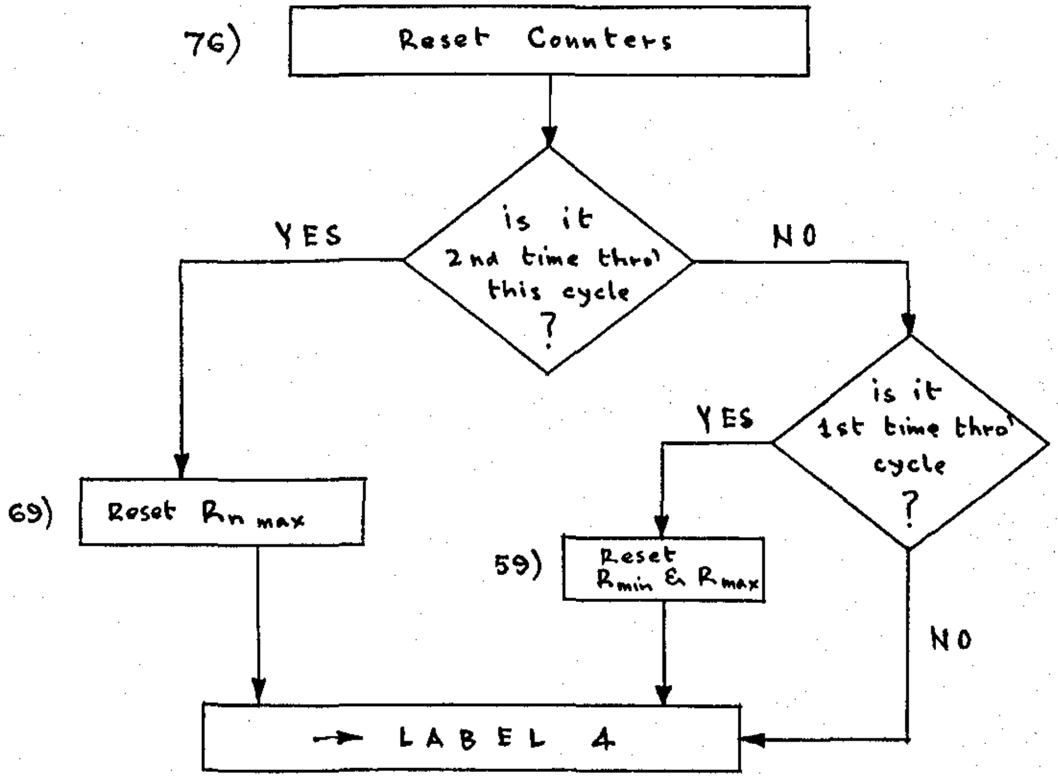


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 3, PART 7.

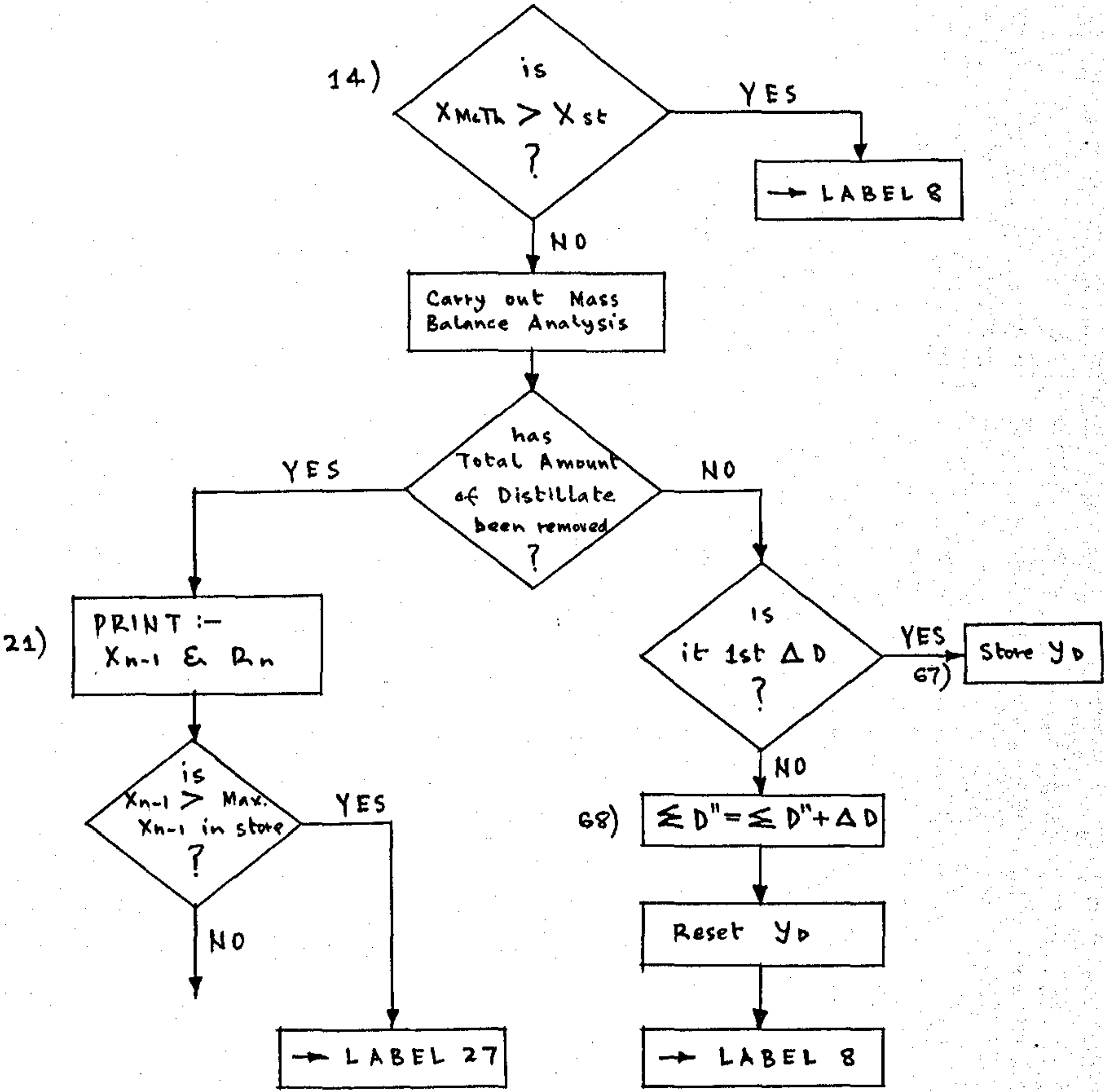


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 4, PART 1.

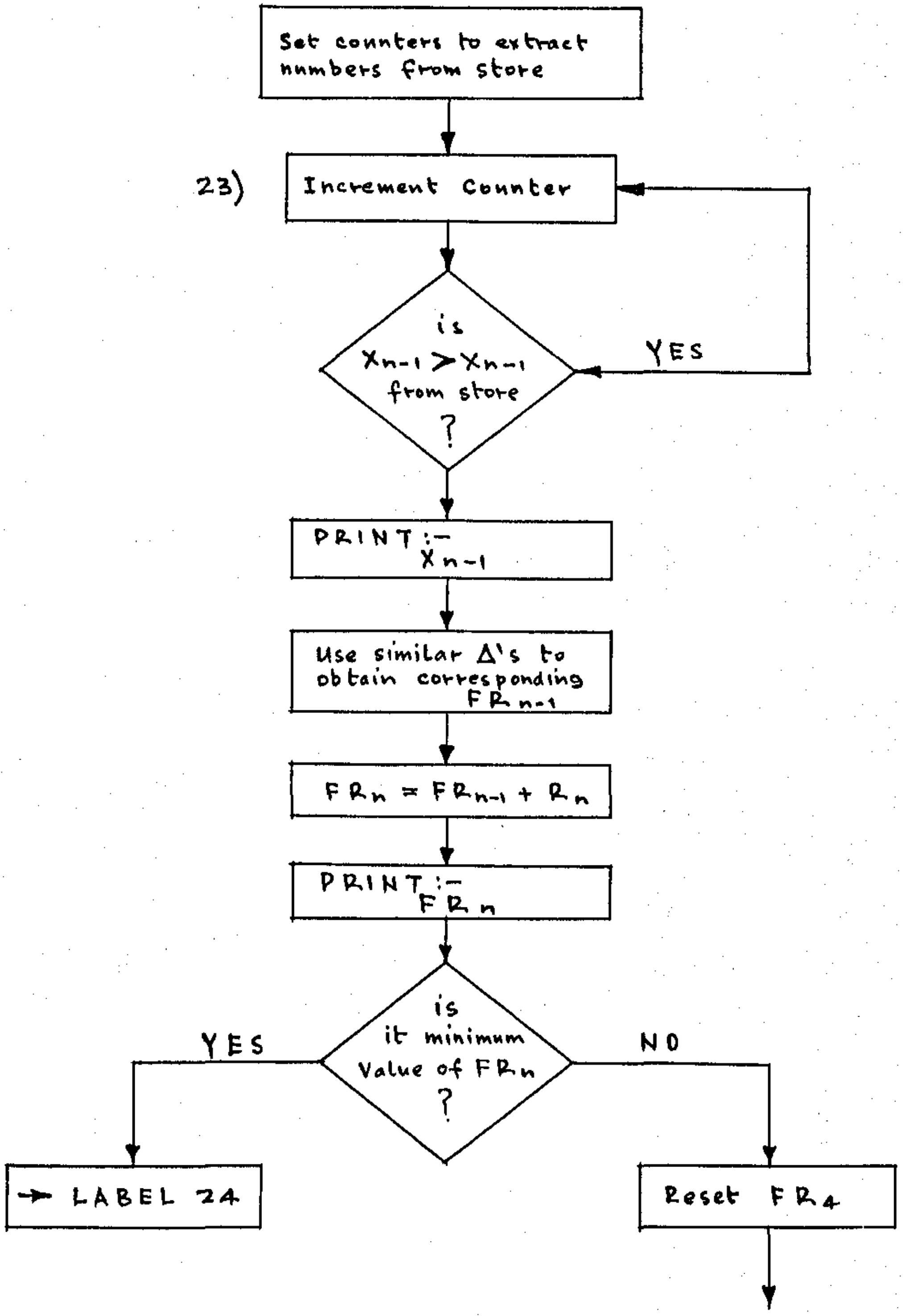


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 4, PART 2.

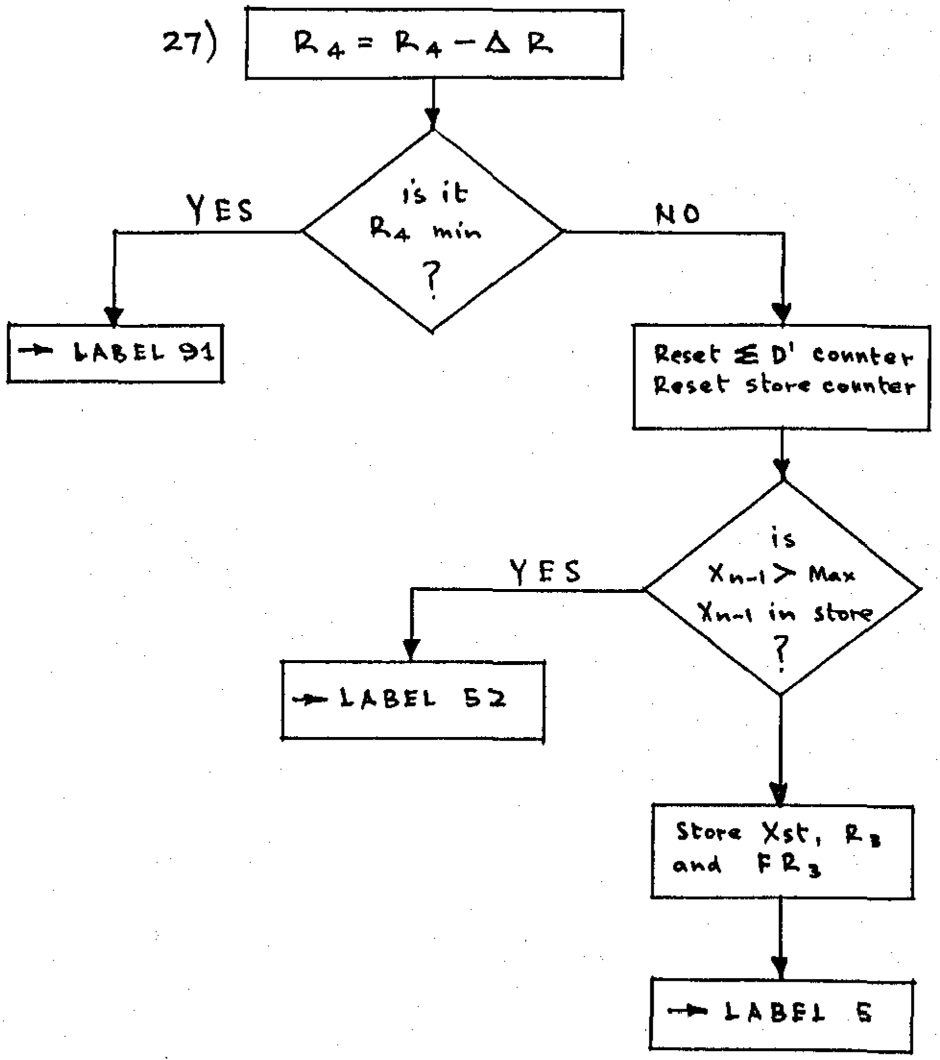


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 4, PART 3.

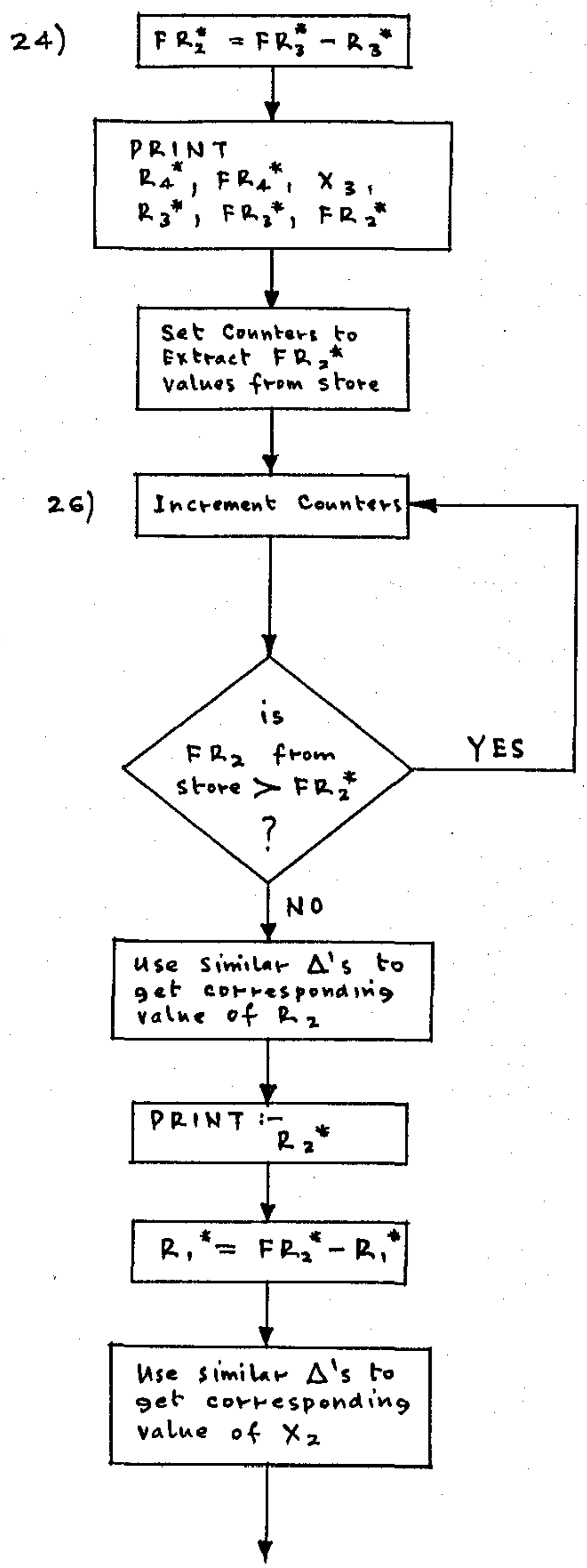


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 4, PART 4.

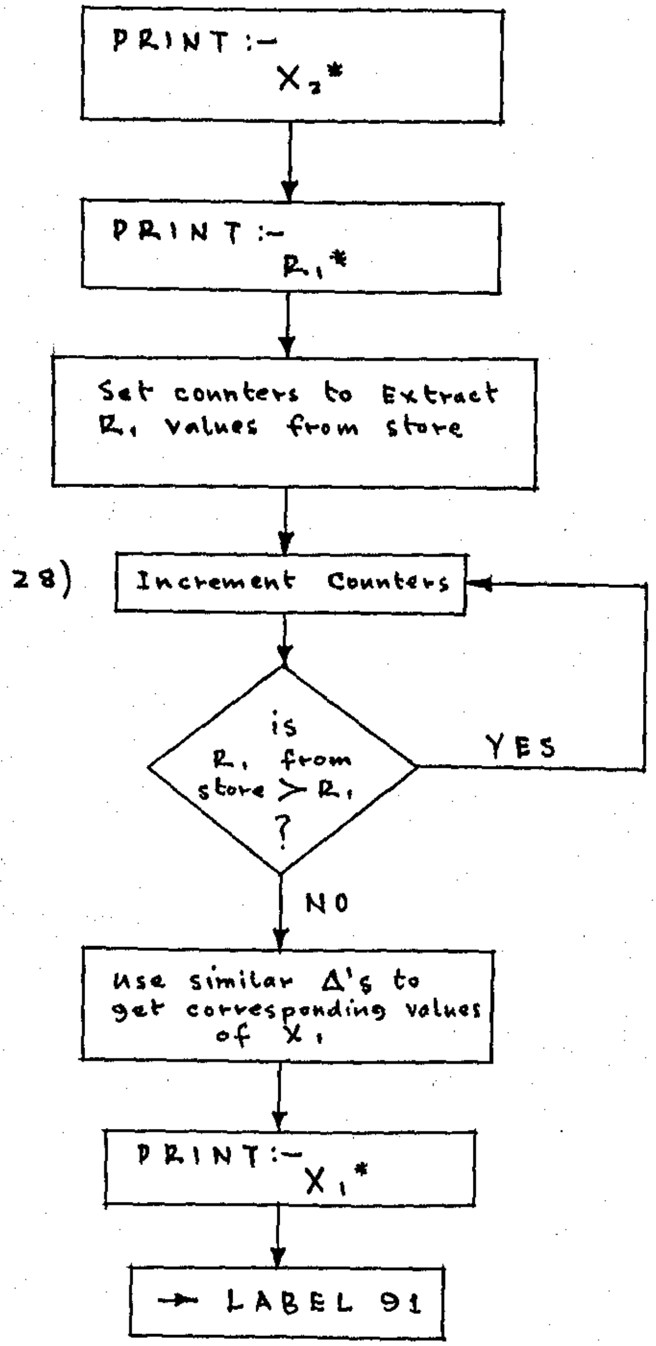


FIGURE :- A8

FLOW DIAGRAM OF OPTIMIZING PROGRAMME - STAGE 4, PART 5.

```
N
OPTIMUM REFLUX.

JV1
STOP
91)V891=TAPE
V1=TAPE44
V93=TAPE7
n4=TAPE
n19=TAPE
V830=TAPE 7
V91=V891/4.0
V91=V91/V94

PRINTV94,3065
PRINTV97,4065
PRINTV98,4065
PRINTV99,4065
PRINTV891,4065


V818=100.0
V815=V91
V77=V891/4.0

V140=V97
n2=1
n18=0
n21=0
V870=V93/2
6)n6=0
V89=0.0
n10=0
n12=0
V820=n4
51)V60=V98
50)V92=0.00
V75=0.00
V88=0.00
52)V140=V97
5)V100=V818
8)V100=V100-V95
n0=0
V801=V100
V50=0
1)V50=V50+1
n27=V50
V811=Vn27
2)→1,V811≥V801
V105=V801-V811
V106=V(21+n27)-V(22+n27)
V107=V(-1+n27)-Vn27
V108=V105XV106
V109=V108/V107
V110=V109+V(22+n27)
n0=n0+1
→3,n0=n19
```

```
V111=V820+1
V112=V820XV110
V113=V112/V111
V114=V100/V111
V801=V113+V114
→2

3)→(10+n2)
11)→8,V110>V98
V120=V92XV94
V79=V99-V120
V78=V79XV60
V121=V94XV100
V122=V79-V94
V123=V78-V121
V124=V123/V122
→19,V95≥V100
→8,V110>V124
V60=V124
n1=1+n2
V47=n1
n1=n1X100
n7=n1+n6
→61,1.0>V92
62)V92=V92+1
V100=V100+V95
→8,V91>V92

n6=n6+3
XPVn7=V124
SPV(1+n7)=V820
SPV(2+n7)=V820

PRINTV124,3065
PRINTV820,4065
PRINTV820,4065


19)V820=V820-V93
→51,V820≥V831
V(66+n2)=V200
V(67+n2)=V124
V905=V200
V906=V124
n4=V833
4)n2=n2+1
V96=900.0
V816=100.0
V817=100.0
V818=100.0
→6
61)V818=V100+V95
→62

12) 13)→8,V110>V98
n25=n2-1
V120=V92XV94
V128=n25
```

```
V129=V128XV77
V130=V99-V129
V131=V130-V120
V132=V94XV100
V133=V131-V94
V134=V133XV110
V61=V134+V132
→81,V89>0.5
V900=V905XV131
V901=V900-V132
V902=V901/V133
→8,V110>V902
→63,1.0>V92
64)V92=V92+1
V905=V902
V100=V100+V95
→8,V91>V92
V135=V(65+η2)
V(67+η2)=V902
→82
81)V900=V906XV131
V901=V900-V132
V902=V901/V133
→8,V110>V902
→65,1.0>V92
66)V92=V92+1
V906=V902
V100=V100+V95
→8,V91>V92
V135=V(66+η2)
82)η9=η2+η25
η9=η9X100
η8=η10+η9
XPVη8=V135
SPV(1+η8)=V902
SPV(2+η8)=V820

η10=η10+3
V89=1+V89

PRINTV135,3065
PRINTV902,4065
PRINTV820,4065

→50,V89=1
V89=0
V820=V820-V93
V905=V(65+η2)
V906=V(66+η2)
→50,V820>V830

η12=0
η14=0
η5=-3
η10=0
71)η10=η10+6
η0=η10
η8=η10+η9
```

```
V908=V(1+n8)
V96=900.0
→75,V908>V(4+n8)
V908=V908
74)V910=V(3+n8)-Vn8
V911=V908-V(1+n8)
V912=V(4+n8)-V(1+n8)
V913=V910XV911
V914=V913/V912
V915=V914+Vn8
→77,V915>V(66+n2)
n3=n25+n25
n3=n3X100
n5=-3
72)n5=n5+3
n11=n3+n5
→72,V915>Vn11
V850=V(-2+n11)-V(1+n11)
V851=Vn11-V915
V852=Vn11-V(-3+n11)
V853=V850XV851
V854=V853/V852
V855=V854+V(1+n11)
V916=V855+V(2+n8)
→73,V916>V96
V96=V916
n10=n10-6
n8=n10+n9
n5=-3
→75,n9>n8
→74
73)V916=V96
n11=n3+200
n13=n12+n11
XPVn13=V908
SPV(1+n13)=V916
SPV(2+n13)=V(2+n8)-V93

PRINTV908,3065
PRINTV916,4065
PRINTV(2+n13),4065

n12=n12+3
V96=900.00
→76,V830>V(2+n8)
75)→77,V908>V(67+n2)
n14=n14+6
n10=n14
→71
77)n10=n0
n7=-2
78)n7=n7+6
n8=n10+n9
n6=n7+n9
→78,V908>Vn6
V908=Vn6
→76,V908=V902
→74
```

```
76)V906=Vn13
V905=V400
V(66+n2)=V400
V(67+n2)=Vn13

→69,n2=3
→59,n2=2
→4
63)V817=V100+V95
V818=V816
→64
65)V816=V100+V95
V818=V817
→66
67)V818=V100+V95
→68
69)n4=V834
→4
59)n4=V835
V830=V836
→4

14)→8,V110>V140
n25=n2-1
V120=V91XV94
V128=n25
V129=V128XV77
V130=V99-V129
V131=V130-V120
V132=V94XV100
V133=V131-V94
V134=V133XV140
V135=V132+V134
V140=V135/V131
V597=V600
V598=V601
V598=V602
→21,1.0≥V91
→67,V815=V91
68)V91=V91-1
V871=9XV95
V100=V100+V871
→8
21)PRINTV140,3065
PRINTV820,4065
→27,V140>V(66+n2)
n24=n25X2
n24=n24X100
23)n12=n6+n24
n6=n6+3
→23,V140>Vn12
XPV140=V140
PRINTV140,3065
V850=V93/2
V145=Vn12-V(-3+n12)
V146=V(-2+n12)-V(1+n12)
V145=V146/V145
V146=V140-V(-3+n12)
```

```
V145=V145XV146
V852=V(-2+n12)-V145

V136=V820+V852
SPV136=V136
PRINTV136,4065
→24,V136>V96
V96=V136

27)V820=V820-V93
→91,V832>V820
V91=V815
n6=0
→52,V140>V(66+n2)
V141=V140
V142=V852
V145=Vn12-V(-3+n12)
V146=V(-1+n12)-V(2+n12)
V145=V146/V145
V146=V140-V(-3+n12)
V145=V145XV146
V143=V(-1+n12)-V145
V140=V97
n6=0
→5
24) XPV820=V820+V93
SPV136=V96
SPV141=V141
XPV143=V143
SPV142=V142
XPV137=V142-V143


PRINTV820,3065
PRINTV136,4064
PRINTV141,4065
PRINTV143,3065
PRINTV142,4065
PRINTV137,3065

n2=n2X100
26)n6=n10+n2
n10=n10+3
→26,V(1+n6)>V137
V860=V(-1+n6)-V(2+n6)
V861=V137-V(1+n6)
V862=V(-2+n6)-V(1+n6)
V860=V860XV861
V860=V860/V862
XP V865=V(2+n6)+V860
PRINTV865,3065
V860=Vn6-V(-3+n6)
V861=V137-V(1+n6)
V862=V(-2+n6)-V(1+n6)
V860=V860XV861
V860=V860/V862
SP V861=Vn6-V860
XP V138=V137-V865
```

```
PRINTV861,4065
PRINTV138,3065
n12=0
28)n13=n12+202
n12=n12+3
→28,Vn13>V138
V862=V(-3+n13)-Vn13
V863=V(-3+n13)-V138
V864=V(-2+n13)-V(-5+n13)
V863=V863XV864
V863=V843/V862
SP V863=V(-5+n13)+V863
PRINTV863,4065
→91
(→0)
```

APPENDIX 9

THE PROGRAMME USED TO PREDICT IDEAL SEPARATION WHEN USING THE SNATCH DISTILLATION TECHNIQUE

The programmes for this task were written in Fortran for use on the I.C.T. 1905 computer. Two programmes were written, one with a negligible hold-up in the reflux drum, and the other with a variable hold-up within it. The main point of difference between the two programmes was that with one, snatches of distillate were removed from the reflux drum, whilst with the other, the snatches were removed from the column hold-up itself.

If we consider now, in detail, the programme used to investigate the case with appreciable reflux drum hold-up, it will demonstrate the techniques used in both programmes.

The purpose of this programme was to use it to investigate the effects of such variables as column and reflux drum hold-ups, on the ideal separation that may be achieved within a distillation unit, when using the snatch technique.

The various steps in the programme have been detailed in its flow diagram (Figure A9), and a printout of the programme included later in the text (Page 285). By referring to the flow diagram, it can be seen that one first specifies the charge to the unit, and the unit characteristics. One then starts from a low initial still composition, and using this determines the composition profile of the column contents. Hence the total charge to the unit which corresponds to the guessed still composition is determined (Figure A9, Parts 1 & 2). Next, the composition found in this way is compared with the actual charge composition, and, if necessary, the initial still composition is increased. This procedure is repeated until the calculated charge composition corresponds to the actual charge composition, where upon the still composition, the charge composition, and the actual column profile are all printed out (Figure A9, Part 2).

Once the conditions within the unit have been ascertained, the effect of removing a snatch of distillate from the reflux drum is determined. This takes the form of determining the new residual contents composition. After this computation, the new residual composition of the unit contents and the total amount of distillate removed to-date, were printed out.

Finally, the programme checked whether the requisite amount of distillate had been removed, and, if so, read in fresh data. If the appropriate amount of distillate had not been removed, the still composition was reset, and using the new unit contents information, the programme was returned to the search procedure (Figure A9, Part 3).

The procedure was much the same for the other programme, except that the distillate was removed from the top of the column. This was done by determining at the start of the programme the hold-up on each plate, andhence, the number of plates emptied in order to produce the requisite quantity of distillate per snatch. Using this information, an with the knowledge of the composition profile of the column contents, it was a simple matter to determine the distillate composition as each snatch was removed.

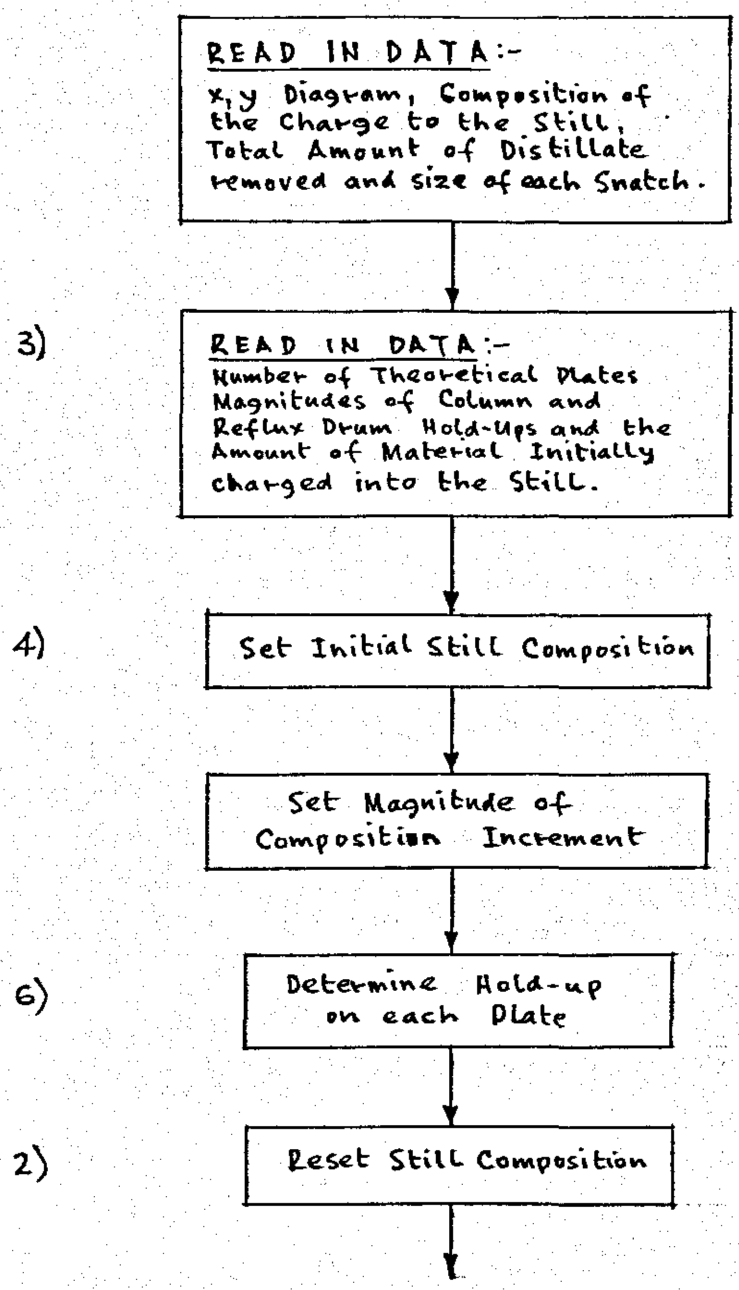


FIGURE :- A9

FLOW DIAGRAM OF PROGRAMME TO PREDICT IDEAL SNATCH SEPARATION – PART 1

1)

5)

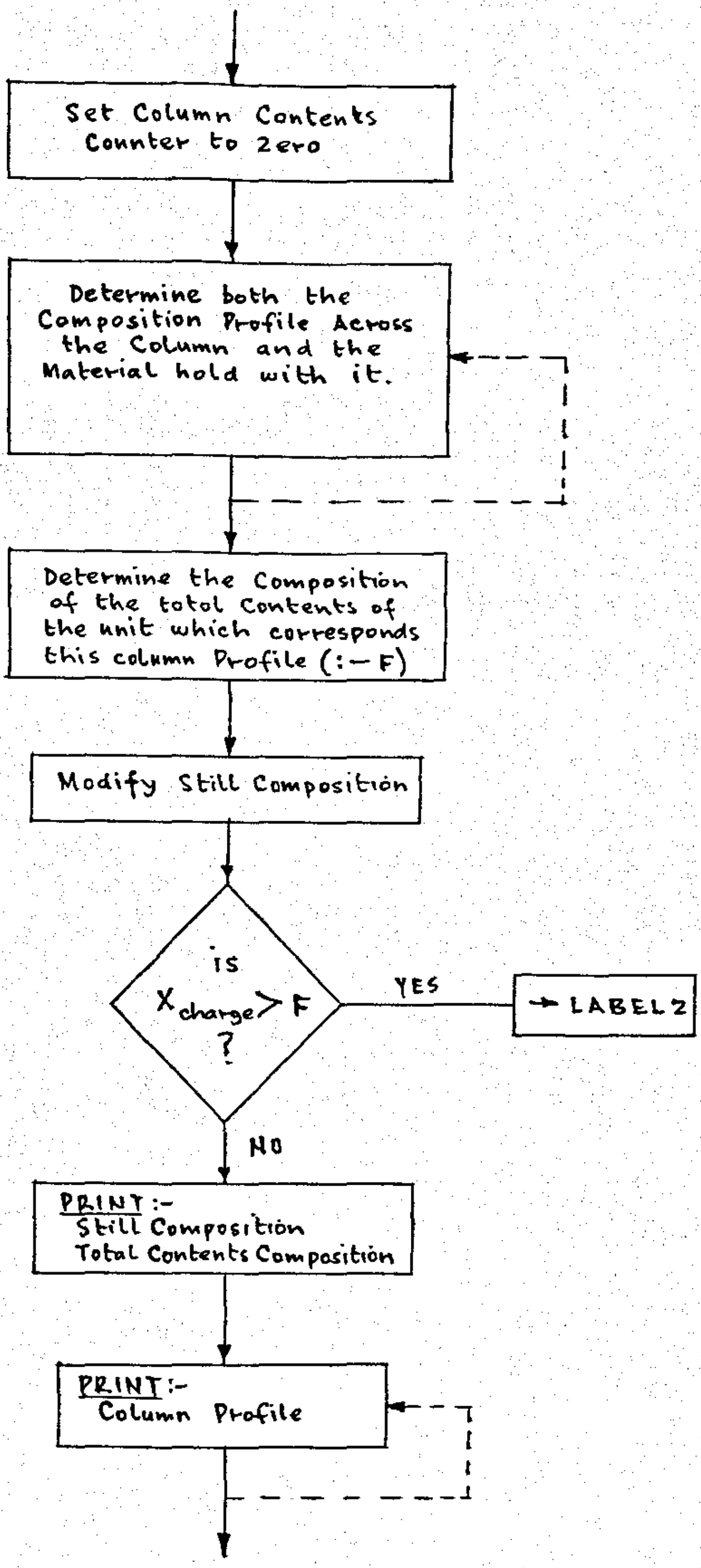


FIGURE :- A9

FLOW DIAGRAM OF PROGRAMME TO PREDICT IDEAL SNATCH SEPARATION – PART 2

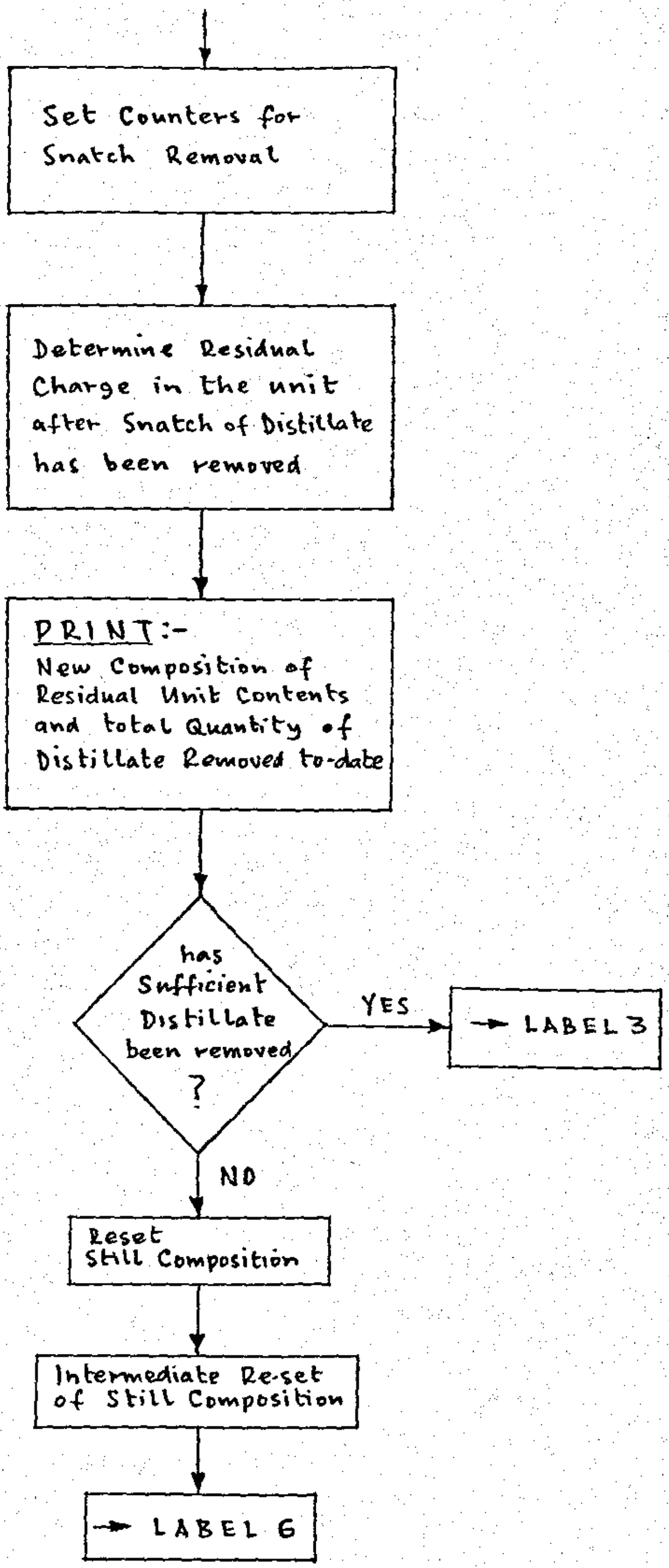


FIGURE :- A9

FLOW DIAGRAM OF PROGRAMME TO PREDICT IDEAL SNATCH SEPARATION - PART 3

```
      MASTER HUKSL
C     DISTILLATION AT TOTAL REFLUX
C     WITH HOLDUP
      DIMENSION X(26),Y(41)
      READ(1,400)M
      READ(1,300)(Y(I),I=1,M)
3     READ(1,300)XCHARGE,TOTDIS,SNATCH
      READ(1,400)N
      READ(1,300)S,H,HC
      WRITE(2,500)S,H,HC,N
      WRITE(2,100)SNATCH
      G=3.4
4     X1=.1
6     DX1=.002
      XN=N
      XH=H/XN
2     X(N+2)=X1
      F=0.
      L=N+1
      DO 1 J=1,N+1
      X2=X(L+1)*40
      MD=X2
      XMD=MD
      XMD=XMD/40.
      MD=MD+1
      X(L)=(Y(MD)+(Y(MD+1)-Y(MD))*(X(L+1)-XMD)*40.)/100.
      F=F+X(L)
1     L=L-1
      F=(XH*F+S*X(N+2)+(HC-XH)*X(1))/(S+XN*XH+HC)
      X1=X1+DX1
      IF(F.LT.XCHARGE)GO TO 2
      WRITE(2,99)X1
      WRITE(2,200)F
      LL=N+1
      DO 5 I=1,LL
5     WRITE(2,100)X(I)
C     SET UP NEW DATA
      S=S-SNATCH
      G=G+SNATCH
      XB=((S+SNATCH+XN*XH+HC)*F-SNATCH*X(1))/(S+XN*XH+HC)
      XCHARGE=XB
      WRITE(2,600)XB,G
      IF(G.GE.TOTDIS)GO TO 3
600   FORMAT(1X,5H XB= ,F8.4,8X,4H G= ,F8.4,/)
      X1=.1
      IF(G.GE.2.4)X1=.01
      GO TO 6
99    FORMAT(//15H COLUMN PROFILE,/F10.4)
100    FORMAT(F10.4)
200   FORMAT(/28H COMPOSITION INTIIAL CHARGE=,F10.4)
300   FORMAT(8F10.4)
400   FORMAT(2I3)
401   FORMAT(1X,19HSNATCH COMPOSITION=,F8.4)
500   FORMAT(34H HOLD-UP  STILL   COLUMN CONDENSER,/8H        ,3F9.3,//,
     11H ,I3)
      STOP
      END


END OF SEGMENT, LENGTH   330, NAME  HWKSL
```

APPENDIX 10

AN ILLUSTRATION OF THE PRACTICAL APPLICATION OF DYNAMIC PROGRAMMING OPTIMISATION TO A BATCH DISTILLATION UNIT

A.10 INTRODUCTION

The object of the investigation was to compare the experimental results obtained during actual batch distillation runs, with those predicted from theoretical computations off-line. The batch distillation task investigated was based on the separation of a 20% Methyl Cyclohexane/80% Toluene mixture. The experimental run was based on a theoretical computation which optimised the production of a high yield of distillate with an average composition of 93.0% M.C.H.

A.10.1 THEORETICAL OFF-LINE COMPUTATION

The analysis of the batch distillation task was carried out off-line, using the programme developed to investigate the use of Dynamic Programming to optimise batch distillation tasks (Section 6.5).

Although the number of stages into which the process may be split is optional, it was decided to employ four steps. Using this technique, the optimum reflux strategy, ($\underline{R}^* = R_1^*, R_2^*, R_3^*, R_4^*$), and the corresponding composition profile was predicted (Table A.10.1).

A.10.2 EXPERIMENTAL PROCEDURE

The theoretical computations were based on the fact that the still was charged with 28 litres of a mixture of 20% Methyl Cyclohexane and 80% Toluene (i.e. 253 Moles of charge). However, because the theoretical routine assumed zero hold-up in the distillation column and auxilliary equipment, extra solvents were added to the experimental unit to compensate for the actual liquid hold-up in the system.

$R_4 = 26.7$	$FR_4^* = 93.2$	$X_4 = 3.06\%$
$R_3 = 24.5$	$FR_3^* = 66.5$	$X_3 = 7.93\%$
$R_2 = 22.1$	$FR_2^* = 41.9$	$X_2 = 12.2\%$
$R_1 = 19.8$		$X_1 = 15.85\%$
		$X_0 = 20.6\%$

TABLE A.10.1

RESULTS FROM THEORETICAL COMPUTATION TO PREDICT THE OPTIMUM REFLUX STRATEGY FOR A NEGLIGIBLE HOLD-UP UNIT

(Initial Charge was 254 Moles of Methyl Cyclohexane/ Toluene Mixture, from which 45 Moles of Distillate were withdrawn).

These extra solvents were added whilst the column was maintained at total reflux at the requisite boil-up rate. This ensured that the additional solvents had the appropriate composition, allowing for the column profile etc.

The addition of this quantity of extra solvents was monitored by use of the automatic on-line chromatograph, which for this purpose was switched to sampling from the still continuously. However, once the extra solvents had been added, the chromatograph was returned to its normal mode ofoperation, namely sampling material from both the column head and the still.

During the initial start-up period, the boil-up loop was controlled manually, however, once the appropriate boil-up rate was reached, the loop was switched to automatic control using a Taylor pneumatic controller.

Although the column was held at total reflux initially, during the experimental runs the reflux loop was also controlled by using a Taylor pneumatic controller. In addition, the off-take rate and the total amount of distillate collected were also measured. In fact, because of the inadequacies of entire reliance on the reflux return flow loop (Section 4.7), these measurements were used in the determination of the actual reflux ratio.

After the column had been charged with the appropriate material, and maintained at total reflux until steady-state within the unit had been achieved, the off-take of distillate was commenced. Collection of distillate at the requisite rate was continued until the appropriate amount of material to complete the first stage of the experimental run had been collected. At this instance, the set point on the reflux return flow controller was altered, and hence, the reflux ratio changed to the second value indicated by the off-line computation. The appropriate amount of distillate was then collected in order to complete the second stage of the experimental run. This procedure was repeated for stages three and four. Once all the distillate had been collected, the unit was returned to total reflux, and the steady-state still composition at the end of the run determined.

Throughout the experimental distillation operation, the automatic on-line chromatograph was used to analyse the material in the still and at the column head. In addition, the analysis programme (Appendix 1), was employed to determine the compositions of these samples. These analyses provided detailed information on the composition profiles throughout the experimental runs, and were compared with the predicted results from the theoretical computations.

A.10.3 EXAMINATION OF THE EXPERIMENTAL RESULTS

The quantity of charge to the still was the desired 31 litres, but at total reflux the steady-state still composition at the start of the run was 20.5% Methyl Cyclohexane.

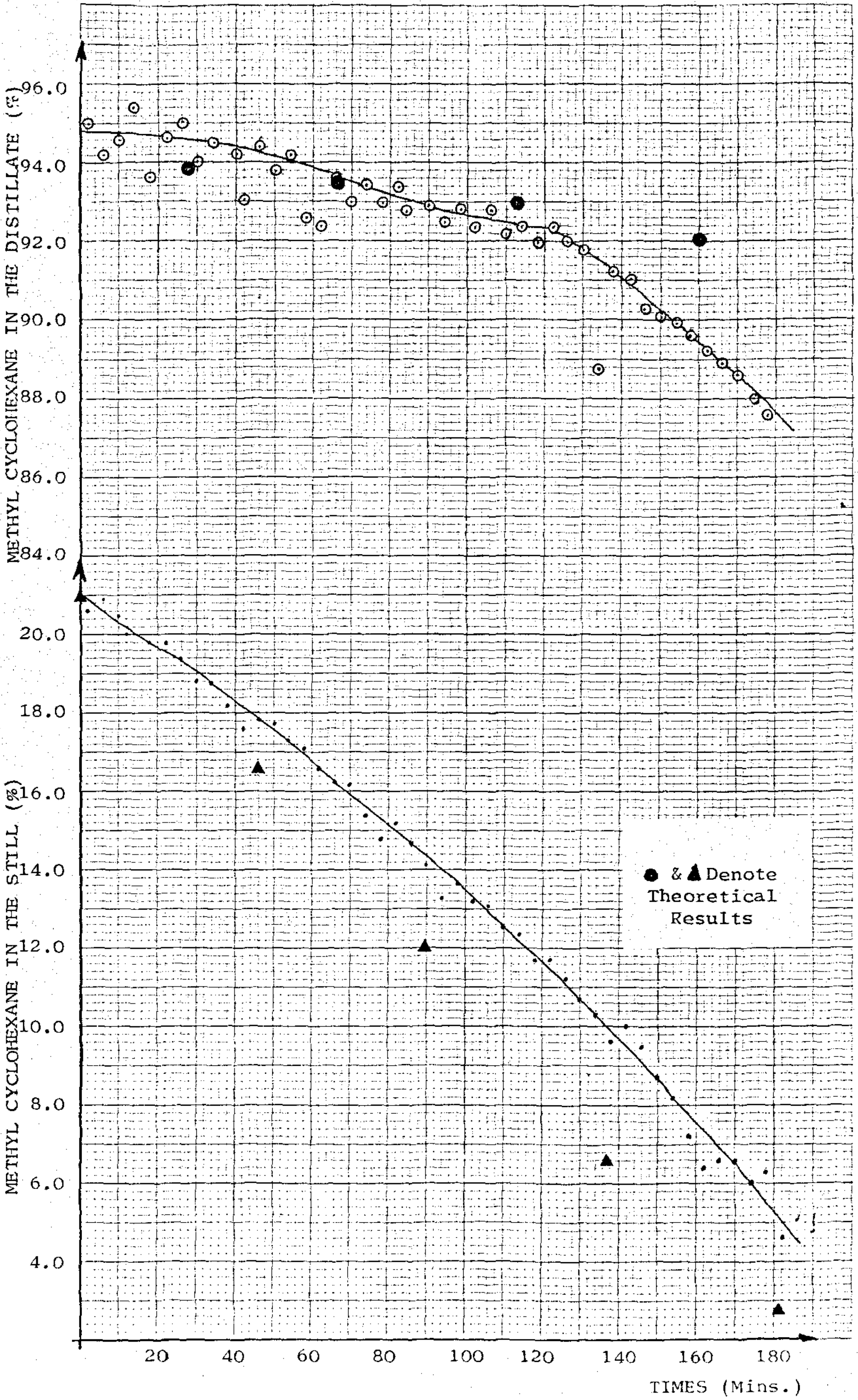
For ease of comparison, the pertinent details from the actual and theoretical distillation tasks have been tabulated (Table A.10.2), and the theoretical and actual composition profiles plotted (Graph A.10.1).

Considering first the operating details of the experimental run, it can be seen that experimental run followed the theoretical steps rather well. A particular problem which had been envisaged had been the control of the reflux ratio. However, by monitoring the col-

	Theoretical Results	Experimental Results
STAGE 1		
X_0	20.6% M.C.H.	20.6% M.C.H.
R_1	19.8	18.75
D_1	36 cc/min.	38 cc/min.
X_1	15.86% M.C.H.	17.3% M.C.H.
$\hat{y}_{D_1}$	94.0% M.C.H.	94.1% M.C.H.
Amount of Distillate Collected	1,350 cc.	1,352 cc.
STAGE 2		
R_2	22.1	24.0
D_2	32.5 cc/min.	30.1 cc/min.
X_2	12.2% M.C.H.	14.0% M.C.H.
$\hat{y}_{D_2}$	93.4% M.C.H.	92.8% M.C.H.
Amount of Distillate Collected	1,350 cc.	1,345 cc.
STAGE 3		
R_3	24.6	24.0
D_3	29.5 cc./min.	30.1 cc/min.
X_3	7.93% M.C.H.	9.8% M.C.H.
$\hat{y}_{D_3}$	92.8% M.C.H.	91.8% M.C.H.
Amount of Distillate Collected	1,344 cc.	1,350 cc.
STAGE 4		
R_4	26.7	25.8
D_4	27.5 cc/min.	28.0 cc/min.
X_4	3.06% M.C.H.	5.4% M.C.H.
y_{D_4}	92.0% M.C.H.	88.5% M.C.H.
Amount of Distillate Collected	1,342 cc.	1,340 cc.
Overall Average y_D	93.0% M.C.H.	91.5% M.C.H.

TABLE A.10.2

COMPARISON BETWEEN KEY THEORETICAL & EXPERIMENTAL RESULTS



GRAPH A.10.1 THEORETICAL AND ACTUAL COMPOSITION PROFILES lection rate continuously, satisfactory control was possible. This was particularly true since the technique allowed up-dated corrective action to be taken. (This was because the accumulative rates and totals were always considered). In this way, any flow measurement errors were not allowed to build-up. As a result, although the error in actual collection rate at any time was as much as ± 7%, the overall collection rate for a particular step could be controlled to an accuracy of the order of ± 2%.

The separation achieved in the experimental run will be considered now. As was to be expected, the actual separation was worse than had been predicted. This is particularly true when one considers that initially, the hold-up of high purity material in the reflux flowmeters improved the performance of the unit. In addition, as the composition of the material in the still rose above its predicted value, the separation became somewhat easier. (In particular, a significant enrichment of the still contents was a result of the high Methyl Cyclohexane content of the initial liquid hold-up).

APPENDIX 11

AN ILLUSTRATION OF A PRACTICAL SNATCH DISTILLATION RUN

A.11 INTRODUCTION

For the purpose of illustrating the relative merits of the snatch distillation technique, the distillation task undertaken was one which had been previously investigated by a Dynamic Programming Analysis (Appendix 10 - Run 1). Hence, the still was charged with the same material as has been employed previously, and the same total run time allowed for the removal of the distillate (Since a constant boil-up rate was employed throughout the experimental work, this resulted in the same total heat input to the unit). For the final variable of snatch strategy, it was decided to remove the snatches of distillate at a constant rate throughout the experimental run. It was therefore merely necessary to specify the number of snatches to fix their size, and the frequency with which they were removed.

A.11.1 EXPERIMENTAL PROCEDURE

The arrangements for charging material to the distillation unit were the same as had been employed previously (Appendix 10). The control of the distillation unit was somewhat easier for this work, since the reflux control merely required the maintenance of the column at total reflux (except when the snatches of distillate were removed from the reflux drum). Once again, the boil-up loop was controlled by the use of a Taylor pneumatic controller.

The on-line chromatograph was employed initially to ensure that the appropriate material was charged to the distillation unit, and to determine the composition profile of the material in the still and at the column head during the experimental run. In addition, at the end of the run, the snatches of distillate which had been removed were also analysed. This was necessitated by the fact that the reflux drum was not a well mixed vessel, and hence, their composition could not be determined from the composition profile at the column head.

Once the distillation unit had been charged with the appropriate material, it was maintained at total reflux until steady-state within the unit had been reached. When this had been achieved, the first snatch of distillate was removed by operating the valve on the reflux flowmeter (Figure A.11.1). The measured quantity of distillate was removed, and the column then returned to total reflux. It was subsequently maintained at total reflux until it was time to remove the second snatch of distillate. The snatching procedure was repeated throughout the experimental run until the appropriate amount of distillate had been removed.

A.11.2. ANALYSIS OF EXPERIMENTAL RESULTS

The unit was charged with 31 litres of the Methyl Cyclohexane Toluene mixture with a composition of 20.2% Methyl Cyclohexane. The quantity of distillate which had to be collected was 5.6 litres, and the total run time three hours. It was decided to collect this distillate by removing fourteen snatches. It was therefore necessary to remove the 400 cc. of distillate for each snatch every 13.75 minutes.

The actual snatch sizes and their compositions have all been

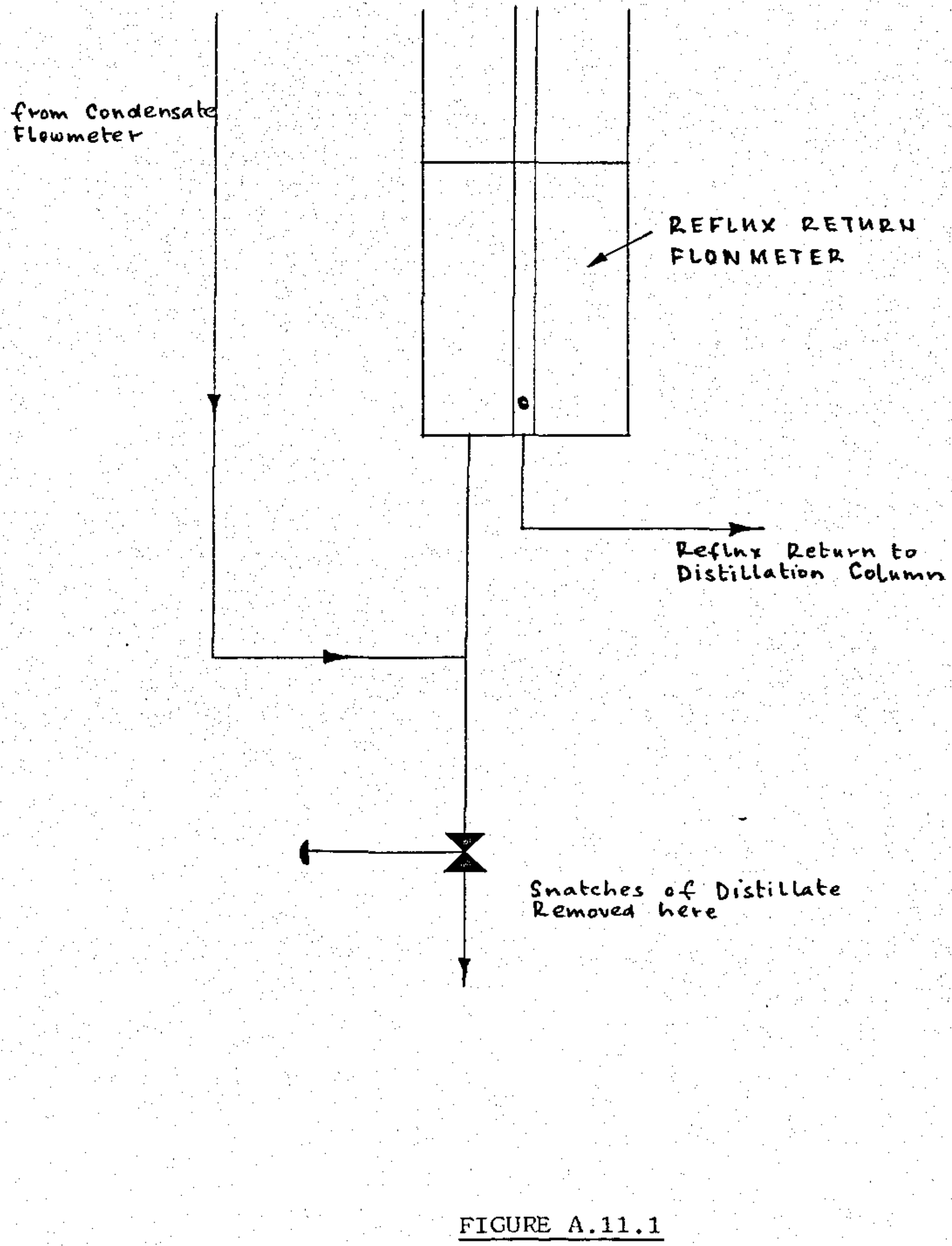


FIGURE A.11.1

ARRANGEMENT TO SNATCH DISTILLATE FROM THE REFLUX FLOWMETER tabulated together with other pertinent details from the experimental analysis (Table A.11.1). In addition, the experimental and ideal distillate composition profiles have been plotted on the same graph (Graph A.11.1).

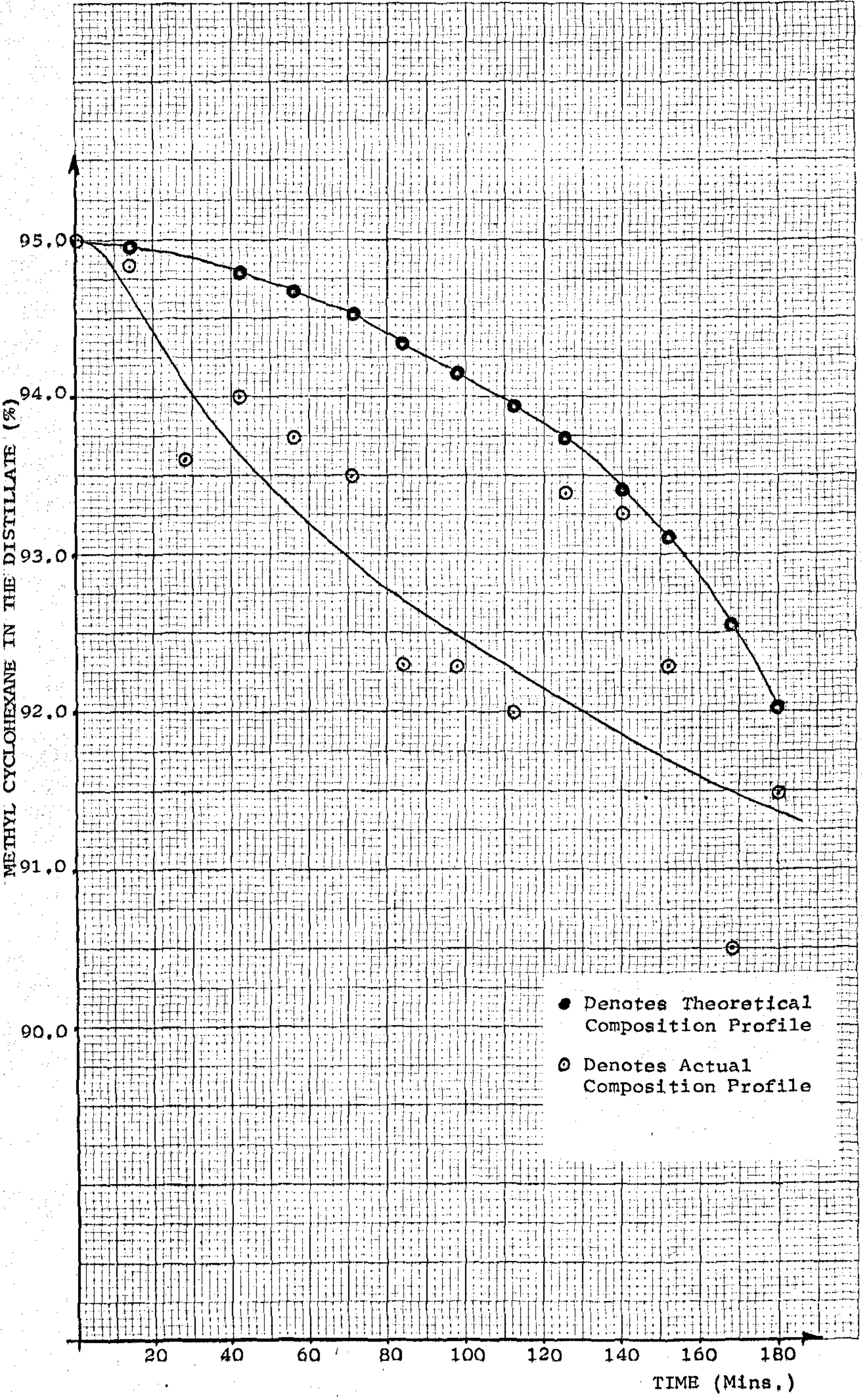
By referring to the results, and comparing them with those obtained when using other techniques, it can be seen that considerable savings are possible by use of the snatch technique.

SNATCH NUMBER	SNATCH SIZE (cc.)	SNATCH COMPOSITION (% M.C.H.)
1	415	95.0
2	385	95.0
3	460	93.6
4	320	94.0
5	440	93.75
6	340	93.5
7	390	92.3
8	395	92.3
9	495	92.0
10	375	93.5
11	365	93.5
12	400	91.8
13	375	90.5
14	380	91.5

TABLE A.11

ANALYSIS OF THE SNATCHES OF DISTILLATE REMOVED DURING THE 3HR. - SNATCH RUN

(Snatches whose Average Volume was 400cc., Removed Every 14 Minutes).



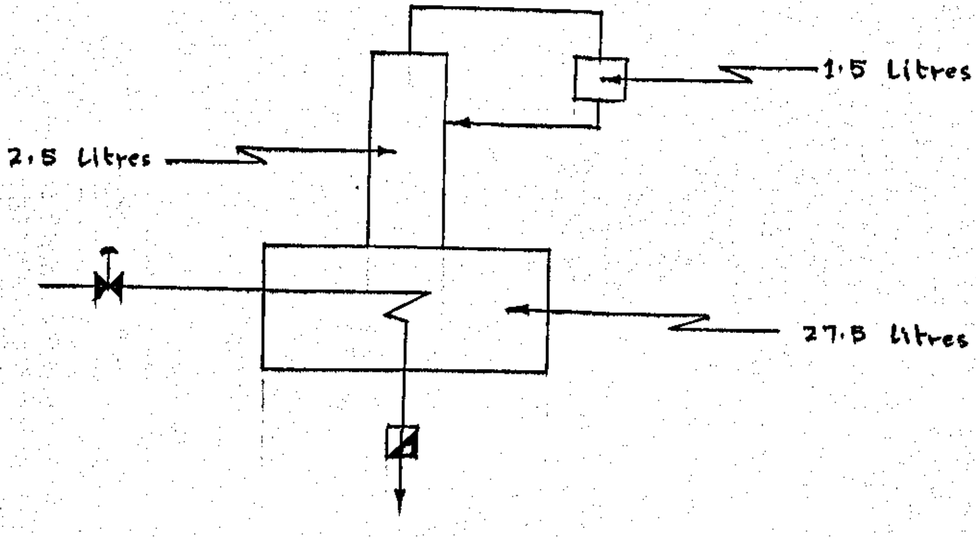
GRAPH A.11.1 IDEAL & ACTUAL DISTILLATE COMPOSITION PROFILES

APPENDIX 12

ANALYSIS OF HOW THE EXPERIMENTAL RUNS SATISFY THE MATERIAL BALANCE EQUATIONS

INTRODUCTION

The experimental runs all utilised information from the chromatograph, and were also dependent on precise information with regard to the initial charge to the unit. For this reason it was decided to carry out a cross check on the results by determining point instantaneous material balances during the runs



For a Typical Run

Total Charge to the Unit was 31.5 litres

namely, 8.830 litres of M.C.H.
22.670 litres of Toluene

Total Charge Analysis

1. Initial Still Composition = 20.5% M.C.H.
2. From the Steady-State Analysis the material Hold-Up Composition (in the Column) = 72.35% M.C.H.
3. Distillate Composition (Flowmeters) = 94.5% M.C.H.

A.12.1 ANALYSIS OF THE SNATCH RESULTS FROM THE 3 HOUR – 14 SNATCH RUN

Actual Methyl Cyclohexane Charged to the Unit = 8.79 litres

Material Balance for Methyl Cyclohexane at the End of the Run, but Prior to the Removal of the Last Snatch

1. Column Contents (Ideal) = 63.25% M.C.H.
2. Still Composition (Actual) = 7.1% M.C.H.
3. Distillate Composition (Actual) = 93.1% M.C.H.
4. Top Product Composition (Actual) = 91.0% M.C.H.

Using these compositions as a basis, the material balance gives a total Methyl Cyclohexane charge of 9.31 litres.

To agree with the actual charge to the unit, the non-ideal column composition would have to be 43% Methyl Cyclohexane. (This assumes that the flowmeters were well mixed drums).

After the Removal of the Final Snatch

Assessment of the Ideal Still Contents = 5.1% M.C.H.

(this is equivalent to a Column Contents composition of 60.54%, and top product of 90.21%)

This would have resulted from a column charge including 9.19 litres of Methyl Cyclohexane

It can be seen from this analysis that, although it was impossible to determine the precise instantaneous composition of the unit contents, the results may be shown to indicate that the compositions are approximately correct.

A.12.2 ASSESSMENT OF RESULTS FROM THE OTHER 3 HOUR SNATCH RUNS

A similar analysis was also undertaken in order to assess the other experimental runs. The basis of the assessment was to ascertain whether the experimental results from the chromatograph were feasible when compared with the known initial charge to the unit. The basis of the material balance examination was to assess the ideal still composition after the last snatch of distillate had been removed, and utilising this information to ascertain the corresponding ideal column composition. The results from this have been outlined below (Table A.12.1).

A.12.3 ASSESSMENT OF HOW THE OPTIMUM REFLUX RESULTS SATISFY THE MATERIAL BALANCE EQUATIONS

A similar analysis to that carried out in the previous section was undertaken for the experimental run utilising the optimal reflux strategy. A detailed description of the run has been outlined in Appendix 10, and from this, an analysis of how the experimental results compared with the actual charge to the units was carried out. The basis of the comparison was to utilise the results in Table A.10.2 in order to assess the column composition at the end of each stage, and the results from this analysis have been outlined in the following Table (Table A.12.2).

Run Number	Number of Snatches	M.C.H. Actually Charged to the Unit	Assessed Still Composition after Removal of Last Snatch	Corresponding Column Composition	Equivalent Total Charge of M.C.H.
1	20	8.86 litres	4.1% M.C.H	55% M.C.H.	8.5 litres
2	7	8.83 litres	5.4% M.C.H.	60% M.C.H.	9.02 litres
3	14	8.79 litres	4.8% M.C.H.	59.5% M.C.H.	8.95 litres
4	(Constant Reflux)	9.0 litres	5.5% M.C.H.	60% M.C.H.	9.1 litres

TABLE A.12.1

MASS BALANCE ANALYSIS OF SNATCH EXPERIMENTAL RUNS

	Still Composition at the End of Stage	Total Average Distillate Composition	Corresponding Column Contents Composition
Stage 1	17.3% M.C.H.	94.1% M.C.H	65% M.C.H
Stage 2	14.0% M.C.H	93.45% M.C.H	58% M.C.H
Stage 3	9.8% M.C.H	92.9% M.C.H	54% M.C.H
Stage 4	5.4% M.C.H	91.6% M.C.H	53% M.C.H

TABLE A.12.2

MASS BALANCE ANALYSIS OF OPTIMAL REFLUX STRATEGY EXPERIMENTAL RUN

Study of Digital Control Algorithms as Applied to a Batch Distillation Column

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Synopsis

The purpose of the paper is to compare the practical application of a number of analogue and digital control schemes. These control schemes form part of the system required for controlling a batch distillation column.

A number of digital control algorithms were tested. In particular a control algorithm designed to reduce the amount of digital logic was found to give satisfactory control. The addition of rate constraints on one of the loops was found to be essential.

Fast reflux flow control was easily implemented A cascade control loop used to run the column just below the flooding point was successfully implemented.

Résumé

Cet exposé a pour objet de comparer l'application pratique d'un certain nombre de schémas de contrôle analytique et de contrôle digital.

Ces schémas constituent une partie du système nécessaire pour le contrôle d'une colonne de distillation continue.

Un certain nombre d'algorithmes de contrôle digital ont été essayés. Plus particulièrement un algorithme de contrôle établi pour réduire la quantité de logiques digitales a fourni un contrôle très satisfaisant. L'addition de constantes de vitesse sur l'une des boucles s'est avérée être essentielle.

Un contrôle rapide du débit de reflux a été facilement mis en oeuvre. Une boucle de contrôle en cascade utilisée pour faire fonctionner la colonne immédiatement au-dessous du point de noyage a été expérimentée.

Kurzreferat

Der Zweck des Aufsatzes ist ein Vergleich der praktischen Anwendung einer Anzahl von analogen und digitalen Regelsysteme. Diese Regelsysteme stellen einen Teil eines Systems dar, das für die Regelung einer Chargen-Destillationskolonne benötigt wird.

Eine Anzahl von digitalen Regelalgorithmen wurde untersucht. Insbesondere wurde gefunden, daß ein Regelalgorithmus, der darauf abgestellt war, den Aufwand an digitaler Logik zu reduzieren, eine befriedigende Regelung ergab. Die Eingfügung von Geschwindigkeitsbegrenzungen in einen der Kreise wurde als wesentlich erkannt.

Eine schnelle Regelung für den Rücklaufstrom war leicht zu bewerkstelligen. Eine Kaskadenregelung mit dem Zweck, die Kolonne knapp unterhalb der Flutgrenze zu betreiben, wurde erfolgreich eingesetzt.

Introduction

The purpose of this paper is to compare the use of analogue controllers for the control of batch distillation with the use of equivalent digital control systems. It was also attempted to discover how realistically optimum reflux strategies, described, for example, in Refs 1 and 2, could be applied to a real batch distillation column.

Continuous distillation columns need very complex control systems and some of the methods that have been suggested are summarized in Ref. 3. The batch distillation column used in this investigation was operated at atmospheric pressure and the outlet temperature of the cooling water could easily be controlled manually. For these reasons it was found that only the boil-up rate, the reflux return and the top-product composition needed control systems to ensure satisfactory operation.

The control is needed to satisfy two requirements, *viz.* control of column hydraulics and control of mass transfer. For the column used the hydraulics could be controlled by controlling the boil-up and the reflux. To make use of the composition measurements, for some strategy of optimum operation, a model describing the dynamics is needed (*e.g.* Ref. 4). As an alternative to the optimal operation described in Refs 1 and 2 the possibility of using sub-optimal control (see Refs 5, 6, and 7) was considered.

The subsequent sections of this paper deal with the theoretical and practical attempts to implement the various levels of control described above.

Symbols Used

C = constant.
D = product take-off rate (mol/h ft^2).
$\left.\begin{matrix}E\\e\end{matrix}\right\}$ = controller error signal.
H = liquid hold-up (mol/ft^3).
h = height of packing (ft).
H^1 = vapour hold-up (mol/ft^3).
K = controller gain.
L = liquid rate (mol/h ft^2).
m = constant.
M_n = valve offset from nth time increment.
P = valve position.
S = set point.
s = complex variable in Laplace transform.
T = controller constant.
t = time (min).
Δt = sampling interval (s).
U = controller constant.
V = vapour rate (mol/h ft^2).
x = composition of liquid phase (mol fraction).
y = composition of vapour phase (mol fraction).
z = distance along column (ft).

Subscripts

$_D$ = derivative mode.
$_h$ = value at $z = h$ (head of column).
$_I$ = integral mode.

$_{\mathrm{I}}$ = inlet.
$_{\mathrm{n}}$ = current time period.
$_{0}$ = initial time period.
$_{\mathrm{p}}$ = proportional mode.
$_{\mathrm{s}}$ = still.
$_{\mathrm{ss}}$ = steady state.
$_{z}$ = at distance z along column.

Feedback Control Algorithms

Pneumatic controllers

Both boil-up and reflux return loops were controlled using Taylor pneumatic controllers. These controllers nominally have a control algorithm described by the equation:

$$P = K_c\,[1 + T_s + U/s]\,E \qquad (1)$$

The actual pneumatic controller characteristics may be approximated by equation (2), which was supplied by the manufacturer:[8]

$$P = K\left(\frac{1+UT}{1-UT}\right)\left(1+\frac{T}{1+UT}s+\frac{U}{1+UT}\cdot\frac{1}{s}\right) \qquad (2)$$

The recommended procedure for setting these controllers is to obtain a step response with a 4 : 1 decay ratio. For the digital controllers it was more convenient to find the controller settings which would minimise the integral of the error squared. The derivative action of the pneumatic controller was not found to be necessary for satisfactory control and was therefore not included in the digital control experiments.

Digital controllers

Three algorithms were tested, the first two, the position and velocity algorithm were suggested by Cox, Hellums, and Williams[9] and the third, which made use of a truncated logarithmic quantization, was proposed by Peatman and Mergler.[10]

The position algorithm represents a three-term controller analogous to that given by equation (1) and is given by equation (3), in finite difference form:

$$P_n = K_p e_n + K_D\frac{(e_n - e_{n-3} + 3e_{n-1} - 3e_{n-2})}{6\Delta t} + K_I\sum_{i=0}^{n} e_i\,\Delta t + P_0 \qquad (3)$$

In the applications of this control algorithm the derivative term, K_D, was set to zero. In this case the control algorithm is equivalent to that described by Allen, Freshwater, Kropholler, and Spikins.[11]

The velocity algorithm is given by:

$$V_n = P_n - P_{n-1} = K_p(e_n - e_{n-1}) + \frac{K_D}{6\Delta t}(e_n + 2e_{n-1} - 6e_{n-2} + 2e_{n-3} + e_{n-4}) + K_I e_n\,\Delta t\,. \qquad (4)$$

Cox, Hellums and Williams[9] stressed the fact that in using equation (4) it was always necessary to include some integral action, *i.e.* $K_I > 0$, as this is the only term which includes a measure of the set-point of the controller. The velocity algorithm was also used with K_D set to zero.

The truncated logarithmic algorithm was specifically designed[10] to keep the digital logic as simple as possible. The equation for the controller is given by:

$$\Delta M_n = K_E\,E_n + K_{\Delta E}(E_n - E_{n-1}) \qquad (5)$$

The operations implied by equation (5) are carried out in such a fashion that core-store requirements are reduced and multiplication is simplified. The operations may be formally defined by the expression:

$$\langle\log E_n\rangle = \begin{cases} 0; & E_n = 0 \\ (\mathrm{sign}\,E_n)\langle 1+\log_2|E_n|\rangle & E_n \neq 0 \end{cases} \qquad (6)$$

The brackets ⟨ ⟩ are used to indicate that the error signal is truncated at the decimal point, *e.g.* 2·96 is represented by 2. Therefore, in binary form, $1 + \langle\log_2|E_n|\rangle$, is a number equal to the position of the most significant digit. The simple logical operations required for this controller are illustrated in Fig. 1.

The final controller settings for any of the digital controllers was made on the basis of minimising the integral of the error squared and using, off-line, the simplex technique described elsewhere.[11] In carrying out this integration the maximum sampling rate was used throughout.

Model for the Distillation System

The modelling for the batch distillation column can be separated into hydraulic behaviour and mass transfer behaviour.

As shown in Fig. 5 and Table I, the hydraulic response of the system is fairly fast compared to the other dynamic effects. The inclusion of the boil-up and reflux return control loops

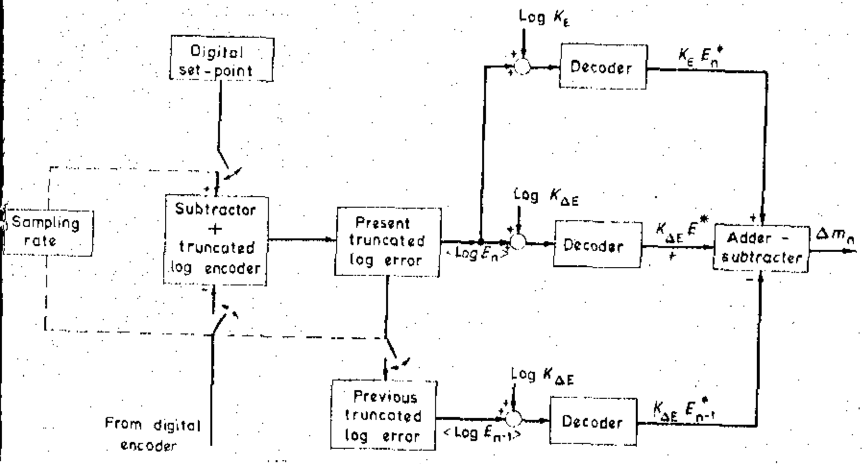


Fig. 1.—Outline of logic for controller employing truncated logarithmic.

TABLE I.—*Summary of Results for Investigation of Process Characteristics (Reflux Flow Loop)*

Initial Signal on Valve	Final Signal on Valve	ΔV on Valve	ΔV on Process	Dead Time on Process	Time Constant of Process	Gain
(V)	(V)	(V)	(V)	(s)	(s)	—
6·0	7·0	1·0	0·94	2	88	0·94
7·0	8·0	1·0	1·07	6	144	1·07
8·0	9·0	1·0	0·61	11·5	111	0·61
5·55	4·8	0·75	1·10	2	107	1·42
6·3	5·55	0·75	1·11	8·0	150	1·48
7·05	6·30	0·75	0·51	10·0	96	0·68

ensures that the hydraulic response is sufficiently fast to be negligible compared to the mass transfer response. Therefore the equations describing the model represent only mass transfer dynamics.

The slowest response is that of the reboiler and the dynamics are adequately described by equations (7) and (8):

$$\frac{\mathrm{d}sx_s}{\mathrm{d}t} = Lx_0 - Vy_s \qquad (7)$$

$$\frac{\mathrm{d}s}{\mathrm{d}t} = -D \qquad (8)$$

A number of simplifying assumptions have been made in describing the column section dynamics and these are described elsewhere.[4] This model was developed as a transfer function for the system but in applying some of the more complex control strategies a matrix formulation of the equation is more convenient.

Instead of using the partial differential equation, as described earlier,[4] for the column section the following set of ordinary differential equations were found to give a good representation of the column dynamics:

$$H\frac{\mathrm{d}\Delta x_n}{\mathrm{d}t} = \frac{L}{\Delta Z}(\Delta x_{n+1} - \Delta x_n) - K\left\{\frac{\Delta x_{n+1} + \Delta x_n}{2} - m_n\frac{(\Delta y_n - \Delta y_{n-1})}{2}\right\} + \frac{\Delta L}{\Delta Z}(x_{ss,\,n+1} - x_{ss,\,n}) \qquad (9)$$

$$h\frac{\mathrm{d}y_n}{\mathrm{d}t} = -\frac{V}{\Delta Z}(y_n - y_{n-1}) + K\left\{\frac{\Delta x_{n+1} + \Delta x_n}{2} - m_n\frac{(\Delta y_n + \Delta y_{n-1})}{2}\right\} \qquad (10)$$

The boundary conditions are obtained by solving the equations:

$$L(x_{ss,\,n+1} - x_{ss,\,n}) = V(y_{ss,\,n} - y_{ss,\,n-1}) \qquad (11)$$

$$\frac{L}{\Delta Z}(x_{ss,\,n+1} - x_{ss,\,n}) = K\left\{\frac{x_{ss,\,n+1} + x_{ss,\,n}}{2} - m_n\frac{(y_{ss,\,n} + y_{ss,\,n-1})}{2}\right\} - Kc_n \qquad (12)$$

The subscript $_{ss}$ refers to the conditions at the beginning of the increment of time under consideration. It was found that by using this approximation the column packing, which was equivalent to about 30 theoretical plates, could be represented satisfactorily by ten space increments.

The number of space increments needed to give an adequate representation of the system dynamics is very sensitive to the finite difference form used for the mass transfer relationship. The following alternative forms were tried:

$$K(\Delta x_n - m_n\,\Delta y_n) \qquad (13)$$

$$K(\Delta x_n - m_n\,\Delta y_{n-1}) \qquad (14)$$

$$K(\Delta x_{n+1} - m_n\,\Delta y_n) \qquad (15)$$

$$K\left\{\frac{\Delta x_{n+1} + \Delta x_n}{2} - m_n\frac{(\Delta y_n + \Delta y_{n+1})}{2}\right\} \qquad (16)$$

For the same accuracy of solution, equation (13) required 1000 space increments, equations (14) or (15) 100 space increments, and equation (16) only 10 space increments when used for the solution of equations (9), (10), and (12).

If the vapour hold-up is neglected the system may be represented by an 11-variable system. This is a reasonable assumption as the vapour hold-up is only one-fiftieth of the liquid hold-up.

The equation for the still is included and equation (17) is used as a piece-wise linear representation in time of the system dynamics:

$$H\dot{\mathbf{x}} = A\mathbf{x} + F \qquad (17)$$

The model represented by equation (17) has only one parameter which is an unknown function of the system provided that the liquid and vapour rates are known and also that the equilibrium relationship is known. This parameter is the mass transfer coefficient, K. However, as there is some error in the measurement of the compositions and flow-rates a further simplification of the model is useful, particularly for sub-optimal control. The form of the equations derived by the use of moments andor cumulants are given by equations (18) and (19) for the composition at the top and the bottom of the packed column section:

$$X_N/L = K_N/(T_1 s+1)(T_2 s+1) \qquad (18)$$

$$X_0/L = K_0 \times (T_1 s+1)^2/[(T_2 s+1)(T_3 s+1)^3] \qquad (19)$$

Equations (18) and (19) are suitable forms for on-line parameter estimation as described by Rees.[6]

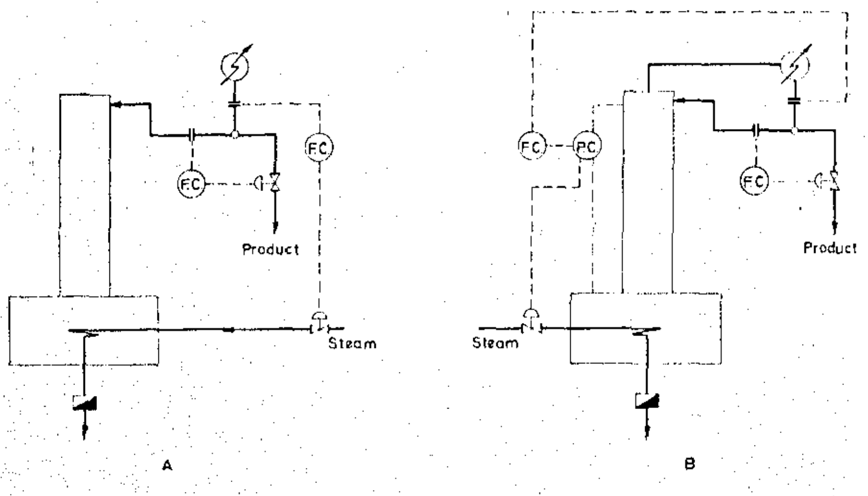
Apparatus Employed in the Experimental Work

The distillation column was constructed in standard Q.V.F. glassware. The still was a 50-litre spherical vessel fitted with a steam-heated thermo-syphon reboiler. The column consisted of a total of 80 in of high-efficiency stainless steel packing in four-inch diameter glass pipe.

The overhead product was condensed in a total condenser and its flow-rate measured using a submerged orifice meter. The outlet from this meter may be split to give product or reflux return and the reflux return rate may be measured using a second submerged orifice meter. The pressure drop across the column was measured by connecting a differential pressure transducer across the column. Differential pressure transducers were used to convert the readings obtained by the two submerged orifice meters to pneumatic signals in the range 3 → 15 lbf/in² gauge.

The various signals from the differential pressure transducers may either be connected to conventional analogue pneumatic controllers or to pneumatic to electrical transducers for feeding into the computer.

Outputs from the pneumatic controllers may be connected directly to pneumatically operated control valves



A: Separation control
B: Column capacity control

Fig. 2.—Line diagrams of distillation units showing control loops

governing the flow of steam to the reboiler and the reflux return rate. However, the outputs from the computer must first be converted to pneumatic signals by current-to-air transducers.

The composition of the liquid in the still or the column head was measured by means of a chromatograph fitted with an automatic sampling device. Liquid from the process was pumped continuously through stainless steel tubing and intermittently injected into the chromatograph. The analysis was processed automatically by feeding the output signals from the chromatograph into the computer. The composition analysis was printed out at the end of each two-minute sample period.

The computer used for the control work was a Ferranti Argus 108 and the analogue to digital/digital to analogue peripheral equipment has been considered as part of the unit.

Outline of Experimental Procedure

Control of the reflux return rate was considered first, then control of the boiling rate, and subsequently attempts at following an optimum reflux strategy were tried.

The first loop may be considered as determining the separation achieved by the column, whilst the second loop affects the column capacity. The control loops are as outlined in Figs 2A and 2B. The mixture used throughout the experimental examination was methyl *cyclo*-hexane—toluene, and it was decided to fix the initial still composition at 30% methyl *cyclo*-hexane throughout the experimental work.

Examination of the analogue controller systems

Using the pneumatic analogue controllers both control loops were tuned approximately using the procedure recommended by the manufacturer. The loops were then further adjusted by using the simplex technique as described elsewhere.[11] The value of $\int e^2\,dt$ was taken as the performance index throughout.

The response of the reflux flow loop is shown in Fig. 3.

Examination of the digital control loops

Approximate values for the controller constants and for the sampling frequency were determined from the open loop responses of the systems as described elsewhere.[11] For the purpose of the experimental investigation, the control loops were considered to include their peripheral equipment. The characteristics of the process were investigated by making step changes in the valve position.

The computer was used to actuate the valve and the open loop response of the system was logged. The open-loop responses for the reflux loop were plotted linearly and on a log-linear plot, and the results have been summarised in Table I. It can be seen that the time constants and gains of the loops varied non-linearly over the whole range of operation.

In addition, it was found that for both the reflux flow loop and pressure drop loops, the time constant of the electrical to pneumatic transducers had a significant effect on their response.

The digital control algorithms described by equations (3), (4), and (5) were tested initially using a process simulated on an analogue computer.

Examination of the reflux flow loop

It was decided to investigate the reflux flow loop by using three sampling periods:

(1). 0·1 second: this was as fast as the interrupt routine would allow.

(2). 2 seconds: frequent sampling, but allows for other loops to be examined as well.

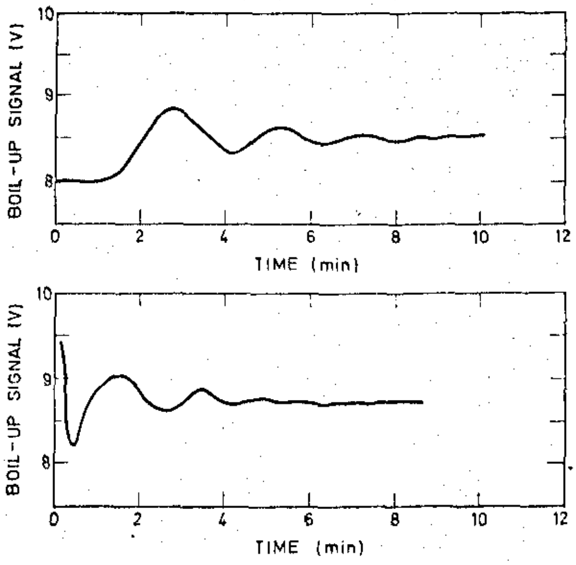


Fig. 3.—Control-loop responses using pneumatic analogue controllers

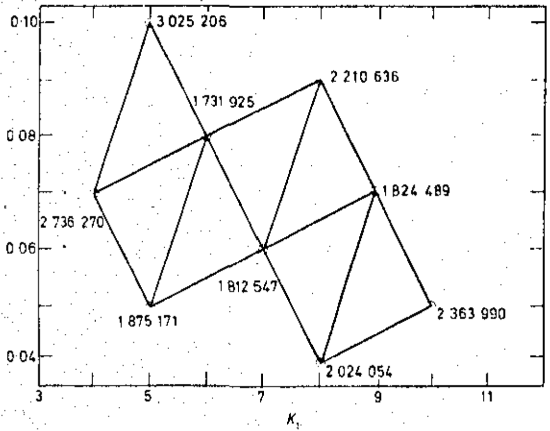


Fig. 4A.—Simplex using velocity algorithm to control reflux flow loop. (Sampling interval = 2 s)

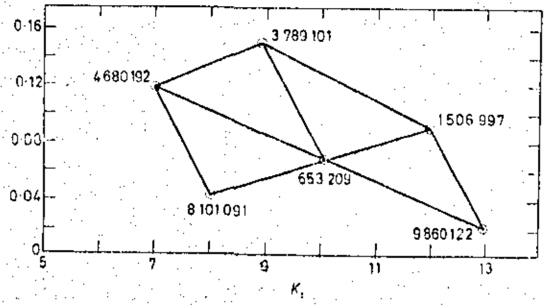


Fig. 4B.—Simplex using truncated algorithm to control reflux flow loop. (Sampling interval = 2 s)

TABLE IIA.—*Results of Simplex Analysis using Velocity Algorithm to Control Reflux Flow Loop (Sampling interval* = 0·1 s). *Without Rate Constraint*

Point No	K_1	K_2	$\int e^2\,dt$
1	10	0·006	1 762 122
2	8	0·008	2 971 846
3	7	0·004	2 479 134
4	9	0·002	1 935 056
5	12	0·004	2 391 211
6	13	0·008	2 527 261
7	10	0·006	1 623 296
8	11	0·010	2 672 763

TABLE IIB.—*Results of Simplex Analysis Using Velocity Algorithm to Control Reflux Flow Loop (Sampling Interval* = 0·1 s). 2% *Valve Movement per Sampling Period*

Point	K_1	K_2	$\int e^2\,dt$
1(a)	10	0·006	2 498 151
5(a)	12	0·004	1 722 151
8(a)	*11*	0·010	*1* 628 869

TABLE IIC.—*Results of Simplex Analysis Using Velocity Algorithms to Control Reflux Flow Loop (Sampling Interval* = 0·1 s). 1% *Valve movement per Sampling Period*

Point	K_1	K_2	$\int e^2\,dt$
1(*b*)	10	0·006	2 273 468
5(b)	12	0·004	2 191 381
8(b)	11	0·010	2 241 985

(3). 30 seconds: considered as the largest sampling period which could control the process satisfactorily.

The simplex operation outlined previously was first used to determine the best controller constants at the various sampling intervals, using the position algorithm. The results have been outlined in Table IIA.

Once the investigation with the standard algorithm had been completed the effect of using a rate restraint to govern the valve movement was considered. Various values for the rate restraint were employed in conjunction with certain specified controller constants that had already been examined. Their effects on the performance index was then determined by comparison (see Tables IIA and IIB).

It can be seen that the use of the restraint tends to result in greater stability of the process over a wider range of controller constants.

The procedure was then repeated for the other controller algorithms and the effects of tuning the controllers are outlined in Fig. 4.

Examination of the boil-up loop

BOIL-UP—FLOW-METER LOOP

The flow-meter gives a measure of the condensate leaving the condenser. The vapour rate up the column is this plus the vapour condensed by the cool reflux. Because of the hold-up in the column and auxiliary equipment there is a substantial time lag in the response of this system.

To examine this system its open loop response was first determined.

From Fig. 4A it can be seen that the system is non-linear, and in addition, there are significant differences in its characteristics depending on the direction of the forcing function.

The system may be examined in a similar manner to that outlined for the reflux flow loop.

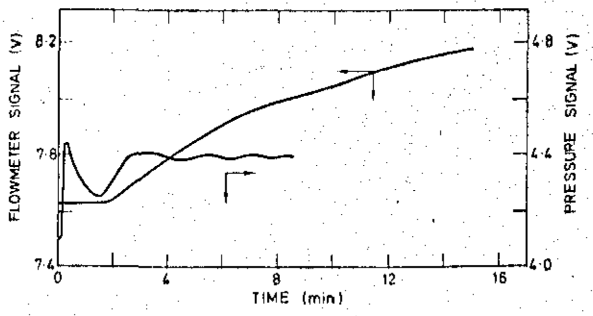


Fig. 5A.—Open-loop responses of boil-up flow-meter and pressure-drop systems

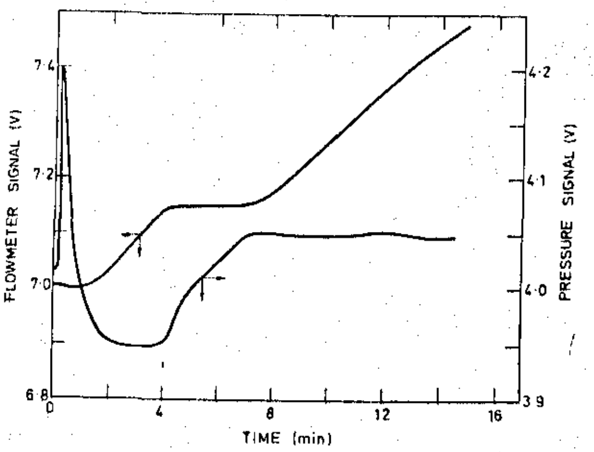


Fig. 5B.—Open-loop responses of boil-up flow-meter and pressure-drop systems

BOIL-UP—PRESSURE DROP SYSTEM

The open-loop response of the boil-up—pressure drop system was examined in the manner previously explained. Typical results are illustrated in Fig. 5B.

The relationship between the flow-meter reading and the pressure drop is illustrated by the plot of the steady state flow-meter and pressure drop readings (Fig. 5C).

Once again the measured variable is the vapour rate up the column but in this case it is assessed by determining the pressure drop across the column. For this reason, the dynamic response is a measure of the balance between vapour rate up the column, liquid rate down the column, and the column hold-up.

The advantage of the loop is that its time constant is small. However, its disadvantage is that the signal/noise ratio is quite low. In addition, its response to a load change may include pulsations. Because of this pulsation a large sampling interval would be desirable. However, the small time constant of the process does not permit this.

The controller constants were again tuned by use of the simplex evolutionary technique (Fig. 6A).

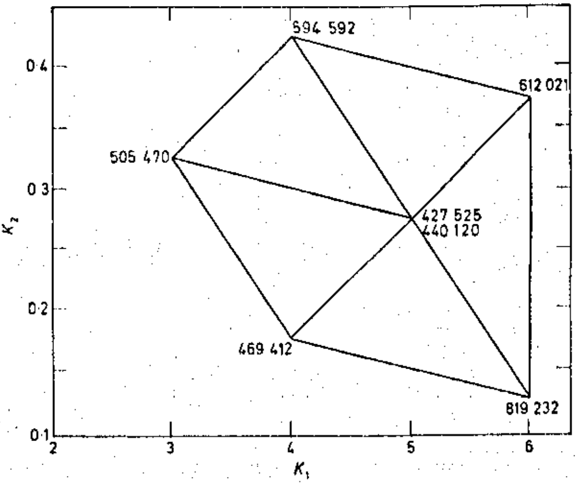
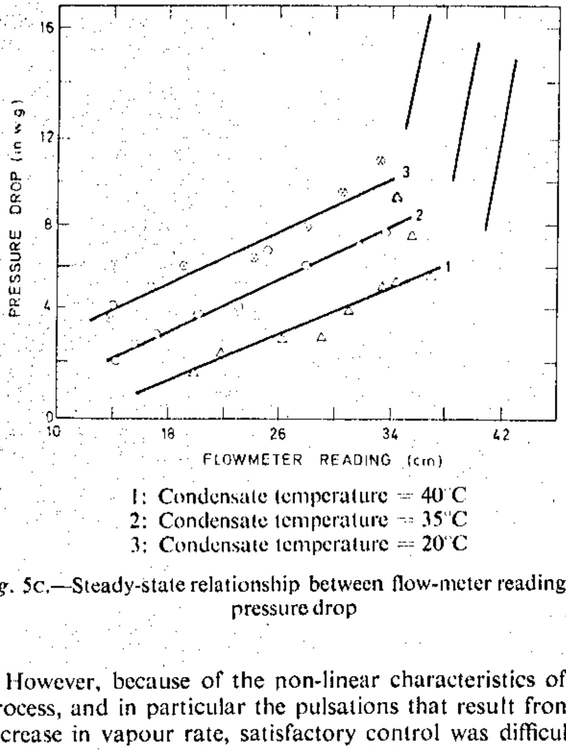


Fig. 6A.—Simplex using position algorithm to control pressure drop. (Sampling interval = 2·0 s)



1: Condensate temperature = 40°C
2: Condensate temperature = 35°C
3: Condensate temperature = 20°C

Fig. 5C.—Steady-state relationship between flow-meter reading and pressure drop

However, because of the non-linear characteristics of the process, and in particular the pulsations that result from an increase in vapour rate, satisfactory control was difficult to obtain. The effect of these characteristics on the controlled response are illustrated in Fig. 6B.

CASCADE SYSTEM

The operation of the cascade system was considered in two ways. First the pressure-drop measurement was used for the master controller and second the condensate flow-rate measurement was used for the master controller.

The throughput of a distillation column, for a given separation, is a function of the boil-up rate. Hence we tried to develop a system to allow the column to be operated as near its flooding point as possible and this requires close control of the boil-up. Figure 5 indicates the open-loop responses of the flow-meter loop and the pressure-drop loop. It has previously been explained that the flow-meter loop measured the amount of liquid leaving the condenser and passing through the submerged orifice meter. The hold-up in the condenser and flow-meter was of the order of one litre, and the hold-up of the column was less than three litres. Hence there was a dead time of several minutes before a change in vapour rate up the column was completed.

Although a sudden increase in hot vapour flow sometimes results in transient flooding without, however, inducing permanent instability, the pressure-drop sensor may be used to detect a change in the dynamic equilibrium of the system almost immediately.

If the pressure drop across the column is used as the controlled variable, then the response of the system to a step change is as indicated in Fig. 7.

Adequate control of boil-up could be obtained by using the flow-meter loop, providing that the boil-up rate was well below the flooding rate. However, when the flow-meter loop was used to control the column just below the flooding point, the system was unstable (Fig. 8).

Observation of the dynamic pressure drop across the column suggested that a rate constraint on the steam-valve movement could be used to avoid flooding (Figs 5A and 5B).

Restriction on the movement of the steam valve was therefore included in the controller programme.

By referring to Fig. 9, it can be seen that the rate of valve movement had to be restricted to 4% per minute before an appreciable improvement was detected. However, even then, satisfactory control of the system was not obtained, since the process was still liable to become unstable.

Although a change of controller constants gave an improved response, the control of the boil-up rate was still not satisfactory.

As a result of the work outlined above it was decided that to operate the column near its flooding point it would be necessary to control both the pressure drop across the column and the boil-up flow-meter reading. It was felt that by using

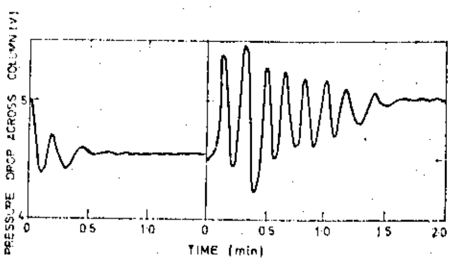


Fig. 6B.—Pressure-drop control loop response using digital velocity controller

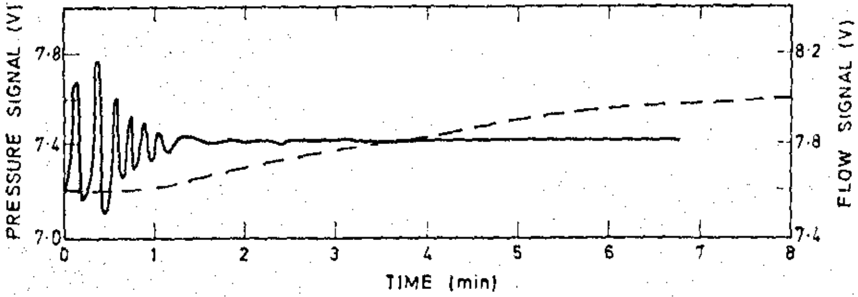


Fig. 7.—Pressure-drop control-loop response and corresponding open-loop response of flow-meter

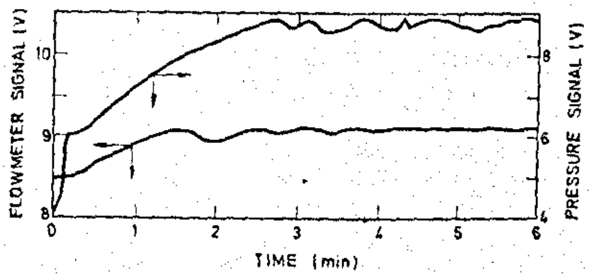


Fig. 9A.—Boil-up flow-meter responses using digital controller with rate restraint on controller output. No limit on ΔV

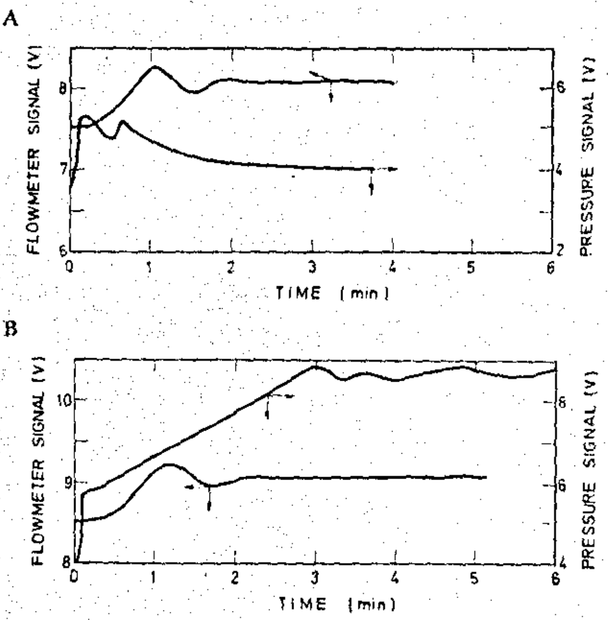


Fig. 8.—Boil-up flow-meter control-loop response and corresponding open-loop pressure drop

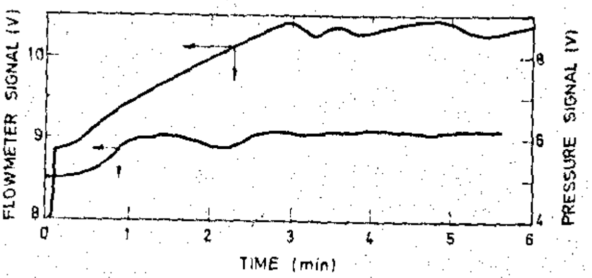


Fig. 9B.—Boil-up flow-meter responses using digital controller with rate restraint on controller output. Maximum value of ΔV = 10%

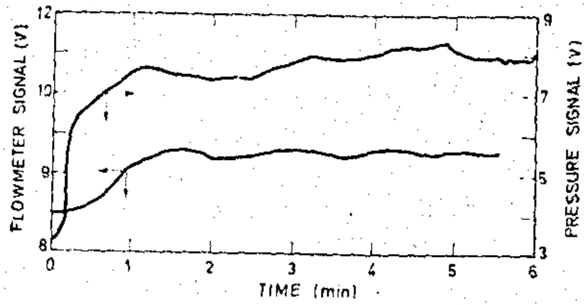


Fig. 9C.—Boil-up flow-meter response using digital controller with rate restraint on controller output. Maximum value of ΔV = 4%

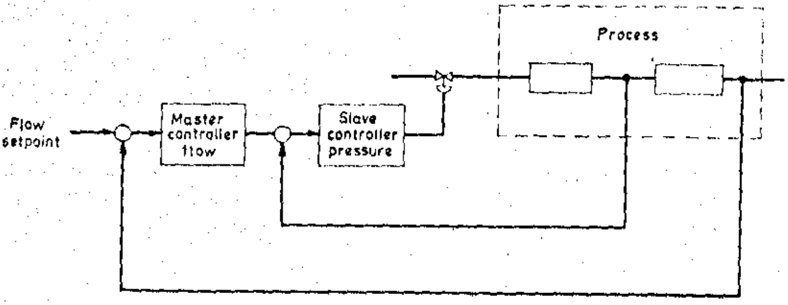


Fig. 10.—Schematic outline of cascade arrangement

LY 7

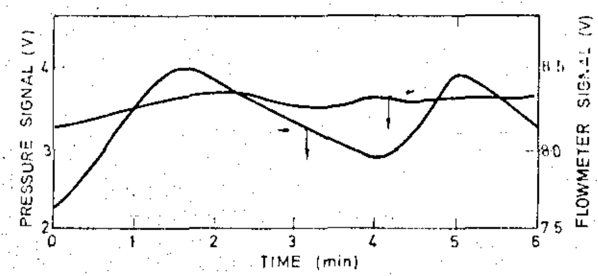


Fig. 11A.—Response of boil-up loop using digital cascade controller. Maximum value of ΔV = 12%

both the variables, the rate of off-take at the top of the column could be controlled at its maximum whilst measurement of the pressure drop ensured that pressure surges through the column could be detected and dealt with before they caused flooding to occur within the column.

The system configuration is outlined in Fig. 9. However, certain modifications were included in the digital cascade programme to allow for the individual characteristics of the system. The noise level of the flow loop was low, and therefore no filtering of the process signal was included. However, it was decided that a rate limitation on the output signal from the master controller be included. Satisfactory control of the pressure loop was more involved. The pressure signal from the plant was read by the computer and because of the noise level a moving average of the signal was used. The error signal was then computed and if it was below a specified value no corrective action was taken by the controller. This was to prevent unnecessary oscillations of the control valve, in particular the oscillations that could result from a noisy signal. In addition, if the signal from the plant was above a specified value then flooding conditions were considered to be on the point of occurring. As a result an alarm message is sent out and the signal to the steam valve is set to zero. In addition the output from the master controller (i.e. the set-point to the secondary loop) was not allowed to exceed the pre-flooding pressure for the column. Finally, the rate of valve travel was constrained to reduce the occurrence of pressure surges within the column.

As has been mentioned previously, the pressure loop is particularly sensitive to flooding conditions within the column. Together with its quick response time this meant that its use in the cascade system allowed the column to be operated successfully near its flooding point.

The sampling intervals for the two loops were independent. After some investigation, it was found that sampling rates of 15 s for the flow loop, and 2 s for the pressure loop handled the system satisfactorily.

The cascade controller was tuned to control the process satisfactorily using the information gathered from the open loop responses and the control of the two loops separately.

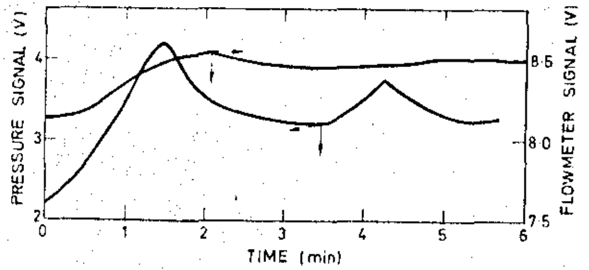


Fig. 11B.—Response of boil-up loop using digital cascade controller. Maximum value of ΔV = 18%

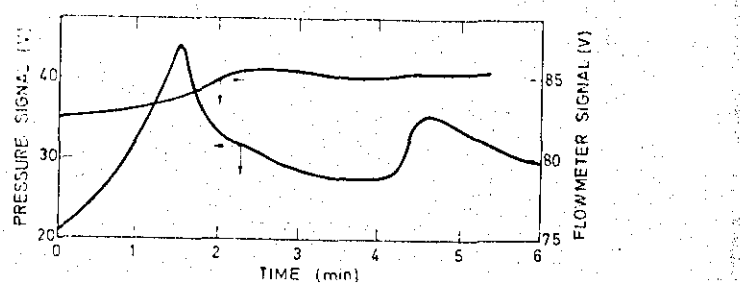


Fig. 11C.—Response of boil-up loop using digital cascade controller. Maximum value of ΔV = 24%

Suitable rate constraints for the outputs from the primary and secondary control loops were determined experimentally.

Typical response curves are presented in Fig. 11.

From these results it can be seen that by use of the cascade system the boil-up loop may be operated satisfactorily near the flooding point of the distillation column.

Optimum reflux strategy

As shown by Price[2] the optimum trajectory for a batch distillation is determined solely by the reflux strategy used. The boil-up rate must be held constant at its maximum value and the experimental results described above show how this was attained by this column.

Both Price[2] and Mitten and Prabhaker[1] showed that it was practicable to calculate an optimum reflux strategy provided that the column hold-up could be neglected. The procedure proposed by Mitten and Prabhaker was used for a batch distillation. Because of the column dynamics which gave a dominant mass-transfer time constant of the same order of magnitude as the still time constant it was not possible to attain the optimum trajectory. At the end of the first stage of the distillation the accumulated product was below specification. As pointed out by Price,[2] the inclusion of additional dynamics considerably complicates the application of Pontriagin's method.

Conclusions

In the development of strategies for the control of a batch distillation column it has been shown that the response of the hydraulics to changes in reflux rate can be speeded up considerably. The use of a digital cascade control system which includes a rate constraint on the demand for an increased pressure drop across the column and a rate constraint on the rate of movement of the steam flow valve allows the column to be operated close to the flooding point and hence at maximum capacity. The simple algorithm proposed by Peatman and Mergler[10] has been shown to be as good as more complex digital control algorithms.

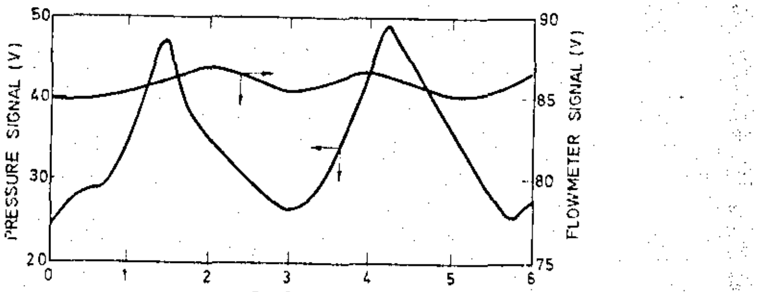


Fig. 11D.—Response of boil-up loop using digital cascade controller. Maximum value of ΔV = 36%

Optimum reflux strategies which do not take into account column section dynamics do not appear to be useful. cause of the "curse" of dimensionality the solution to the mplete optimization problem does not appear to be acticable. A possible alternative is the application of a b-optimal control theory as described, for example, in fs 5, 6, and 7. In this case it is necessary to define an tial and final state. If this were feasible then it might be ssible to design a relatively simple control system which uld approach the optimal solution. Further work needs be done in this field.

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NOMENCLATURE*

C	Constant
D	Product take-off rate (Mol./h.ft^2)
E e	Controller Error Signal
F	Feed to Batch Still (Mols.)
F	Function
H	Liquid Hold-up (Mol./ft^3)
h	Height of Packing (ft.)
K	Controller Gain
L	Liquid Rate (Mol./h.ft^2)
Δm	Step Change in Controller Output
m	Constant
P	Valve Position
R	Reflux Ratio
S	Set Point
s	Complex Variable Laplace Transform
t	Time (min.)
Δt	Sampling Interval
U	Controller Constant
V	Vapour Rate (Mol./h.ft^2)
x	Composition in Liquid Phase (Mol. Fraction)
y	Composition in Vapour Phase (Mol. Fraction)
z	Distance along Column (ft.)

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* Unless otherwise stated in the text the symbols used have have the following definitions.

Subscripts

D	Derivative Mode
h	Value at $z = h$ (Head of Column)
i	Integral Mode Inlet
n	Current Time Period
o	Initial Time Period
p	Proportional Mode
s	Still
ss	SteadyState
z	Distance along the column