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I am submitting herewith a thesis written by Marc Christopher Moss entitled "Methods and Challenges of Experimental System Identification of a Distillation Column." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science in Engineering, with a major in Chemical Engineering.

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Methods and Challenges of Experimental System Identification of a Distillation Column

A Thesis

Presented for the

Master of Science in Engineering: Chemical

Degree

The University of Tennessee, Chattanooga

Marc Christopher Moss

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ABSTRACT

The University of Tennessee at Chattanooga Engineering Department distillation column is used as a teaching tool, but is currently without a feedback control system. An eventual goal of developing such a control system requires that the behavior of the column be understood. It is necessary to identify which of the column parameters can change, either ones that change to cause a disturbance, or ones that the controller can change in response to a disturbance. There are four of these parameters in the UTC column: feed flow rate, feed composition, reboiler heat and reflux percent.

Experiments were performed on the column to identify the response when each of the four parameters was changed in isolation. A chart showing the concentrations on each of the trays both before and after the change was used to visualize how the change affected the individual trays. The operating lines from the McCabe-Thiele method were used to analyze the overall effect on the column. An analysis of the speed of the response of the individual trays was also performed. From these methods of analysis, it is apparent that the center of the column responds most quickly to a disturbance.

Three experiments were also performed in which manual feedback control schemes were tested on the column. In all three cases, the goal was to maintain one of the product streams at the starting concentration after a disturbance by manipulating only one of the controllable parameters, the reboiler heat.

A significant amount of material – primarily water, but also with some ethanol – is unaccounted for in a material balance of the column. Leaks were noted at some of the column joints, and there are some questions as to the reliability of the pump flow rates.

The missing material affects the behavior of the column; therefore these two problems need to be further evaluated.

The data obtained for this report can serve as the first steps in the development of a feedback control system. Gain and time constant values from the top, bottom and center of the column are presented for future reference. Suitable methods for control of a single stream composition (either distillate or bottoms product) are presented, but controlling both will require further work.

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1 Introduction

Understanding the behavior of a piece of equipment is vital to being able to control it. This is especially true in the case of a distillation column. Most systems have a linear (or approximately linear) response to changes; a standard control system using first order differential equations can therefore be used to control it. A distillation column is far from being a linear system, meaning that a standard control system will not work in most circumstances. Identifying which parameters in the column can change, and which of those are controllable, is therefore important for the eventual development of a feedback control system for the column.

Once the changeable parameters are known, it is necessary to understand how the column responds to a change in each. This involves monitoring the concentration of the liquid on each tray and determining the magnitude and direction of their changes, as well as the amount of time required for the change to occur.

During the analysis of the column, several challenges presented themselves. Unexplained behaviors, unquantified leaks and flow rate inconsistencies all complicated the process.

1.1 Column Description

The distillation column in the University of Tennessee, Chattanooga engineering controls lab (illustrated in Figure 1.1) has 12 stages with a partial reboiler and a total condenser. The main body of the column is composed of three sections: two sections each containing six trays and a small section between them where the feed enters (this means that the feed effectively enters on tray 7.) The reboiler is heated via resistance heating. The distillate is condensed with cooling water, and a magnetically switched valve controls reflux. Three constant displacement rotary pumps move material into and out of the column: one supplying feed to the column, one removing collected distillate from a receiver vessel (not shown in Figure 1.1) and the third removing bottoms product directly from the reboiler.

1.2 Column Parameters

An important consideration in how the operator of a distillation column should respond to a disturbance is the goal of the separation. The desired goal determines which variables need to be adjusted. The logical goals are a targeted purity for either the distillate or the bottoms product. In the experiments run for this report, a number of scenarios were considered:

1. maintaining the temperature of tray 1 at the level it was at when the disturbance occurred (and therefore the distillate composition);
2. maintaining the temperature of the reboiler at the level it was at when the disturbance occurred (and therefore the bottoms composition);
3. considering what would be necessary to maintain the entire column at the same level after the disturbance.

The variables in the UTC column that can be adjusted are the reboiler heating load, the reflux percent and the amount of feed being supplied to the column (via the speed of the reboiler pump.) In addition, it is possible to change the composition of the feed via manual manipulation of the feed pump tubing. Knowing what each of these four factors does in isolation is important for an eventual control system.

The column was analyzed under the assumption that both the feed composition and flow rate are uncontrollable variables, and that the column must respond to unanticipated changes in one (or possibly both) of these. The variables available to the controller are therefore limited to the reflux percent and reboiler heating load, which determine the column's reflux ratio and boil-up ratio, respectively.

In the analysis of a column, the parameter normally used is reflux ratio (R) instead of reflux percent. However, for the UTC column, reflux ratio is not an experimental parameter, while reflux percent is. The two parameters are related by the following relationship:

$$R = \frac{reflux\%}{1 - reflux\%}$$

For the operation of the column, the reflux percent is directly related to the percent of each minute the reflux is directed back down the column. For example, the 66% reflux used in many of the experiments for this report would result in the reflux being directed back down the column for 0.66 minutes, followed by 0.34 minutes where the distillate leaves the column. In addition to being useful in the operation of the column, the reflux percent is also useful in the analysis of the column. One graphical method, McCabe-Thiele, makes use of operating lines; the top operating line has a slope that is equal to the decimal form of the reflux percent.

1.3 Report Description

This thesis serves as a description of the experiments performed to understand the behavior of the UTC column and the parameters that affect this behavior. The section immediately following the introduction is a review of the literature covering experimental observation of distillation dynamics. Then comes a summary of all of the experiments run, detailing the objective of each experiment, the procedure used, any significant observations made during the performance of the experiment, and the conclusions drawn from the analysis of the data obtained. Following this is a section with a more thorough analysis of all the data obtained. Ending the thesis is a summary of the conclusions drawn, as well as a discussion of some problems encountered and suggestions on follow-up analysis.

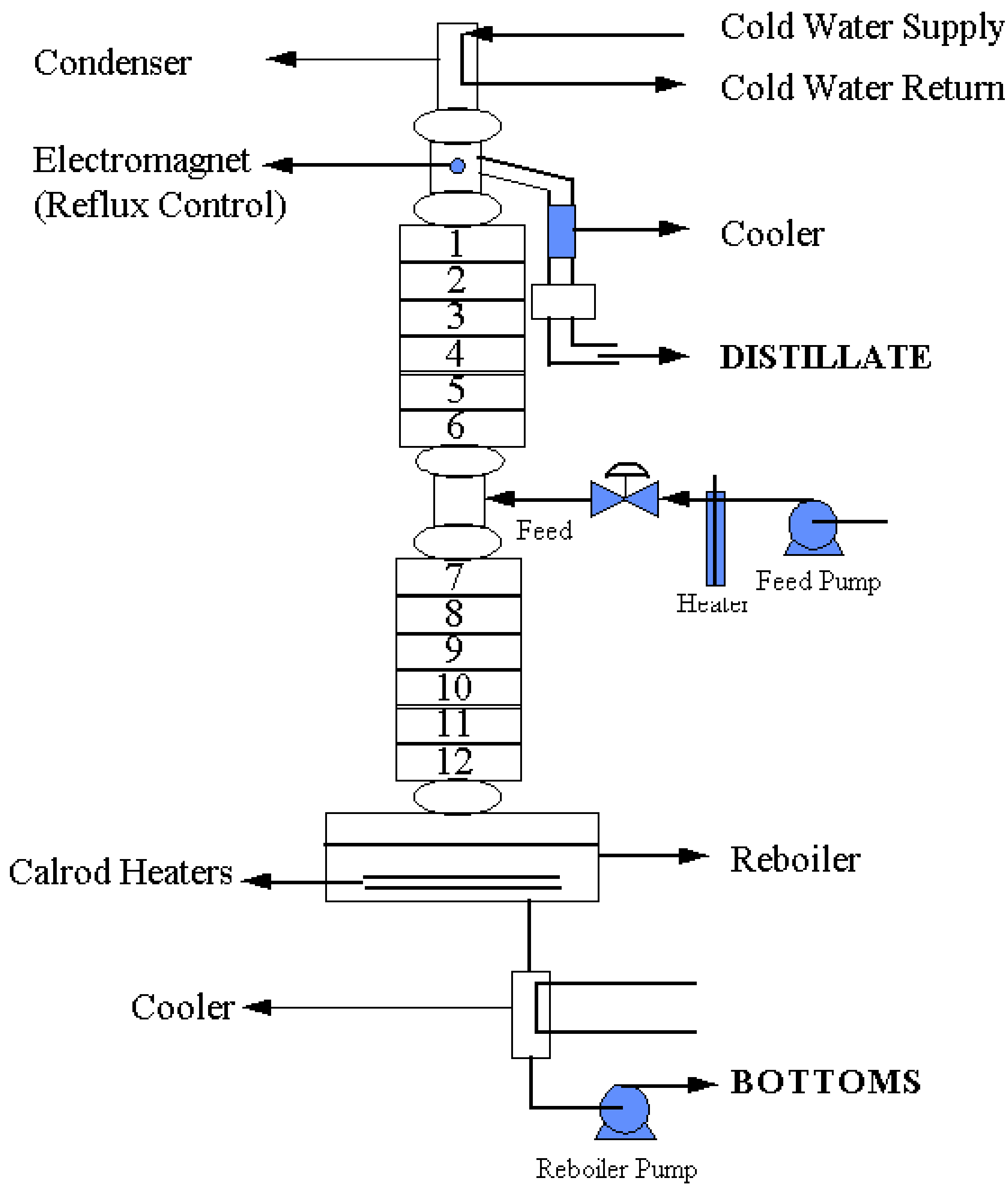

Figure 1.1. Column Diagram

2 Literature Review

Distillation is a very mature unit operation, and a great deal has been written on both the process itself and on its control. While a lot of material can be found relating to the theoretical analysis of distillation, there is less focused on experimental analysis of an actual column.

An excellent summary of the literature covering experimental analysis can be found in the master's thesis by Enagandula (2000.) To avoid unnecessary duplication, none of that material is covered here, and the reader is directed to this source for general coverage of the topic. On matters with direct impact to this thesis, the question of whether to focus on control of the composition of a single product stream or to try to control both has been analyzed on several occasions. The competing issues of complexity and efficiency have given rise to arguments supporting single-component control (Freuhauf and Hahoney, 1994) or dual-component control (Chiang and Luyben, 1985), respectively.

Previous work done on the UTC column primarily focused on manual control of the column (Cunningham, et. al., 1997.) One attempt was made to develop a fuzzy logic control system for the column (Ruta, 2003), but difficulties tuning the fuzzy controllers remained after their development.

3 Experimental Work

In order to understand the experimental work performed, several important standards need to be developed, behaviors described and concepts defined.

Probably the most important of these is the concept of steady state. Steady state is reached after six times the time constant has elapsed after a disturbance. For distillation columns, the time constant can be very long – in the hundreds of hours for some columns – meaning steady state won't be reached for hours, days, or even weeks in extreme cases. Because of the low number of trays and the small hold-up volume on each tray, the UTC column's time constant will be in the lower end of the range. Even then, however, true steady state could take several hours to reach. The term "steady state" will therefore be used to refer to the time when the tray temperatures can be visually judged to no longer be changing.

Because the UTC column is a teaching tool, it can be in operation at many times and at a wide variety of operating conditions. The contents of the reboiler (approximately 12-13 L of material) and the trays were therefore unknown at the beginning of most of the experiments. For this reason, despite attempts to standardize procedures, the amount of time necessary to reach steady state at the beginning of each experiment could vary considerably. In situations where more time was needed, the column was allowed to continue operating until it had reached steady state, even if this took it past a time specified in the procedure developed for the given experiment.

The feed supplied to the column was maintained at the same composition at all times for all but two of the experiments. A target concentration of 10 volume percent was decided on, since it is a reasonable concentration for a fermented ethanol mixture. The final mixture used was 3.33 mol% (10.12 vol%). The only time this feed was not used was for two experiments where the purpose was to examine the column behavior in which the feed was switched, in this case lowering the feed concentration to 2 mol%.

Table 3.1 Experiment summary

Exp. Name	**Date**	**Objective**
System Identification 1	Nov. 25, 2008	Obtain gain and response time values for changes in reflux percent, reboiler heat and feed flow.
System Identification 2	Feb. 5, 2009	Obtain gain and response time values for changes in reflux percent. Repeat of system identification 1.
System Identification 3	Feb. 14, 2009	Obtain gain and response time values for changes in reflux percent.
System Identification 4	Feb. 17, 2009	Attempt to repeat unexplained behavior present in system identification 3.
System Identification 5	Feb. 21, 2009	Repeat of system identification 3.
System Identification 6	Feb. 24, 2009	Evaluation of total reflux period at system start-up.
System Identification 7	Feb. 25, 2009	Further evaluation of total reflux period.
System Identification 8	Feb. 26, 2009	Obtain gain and response time values for changes in reflux percent. Length of step not based on observation, instead set at 50 minutes.
System Identification 9	Mar. 3, 2009	Analyze response of column to change in feed flow rate, from pump setting 2 to pump setting 3.
System Identification 10	Mar. 5, 2009	Analyze response of column to change in feed composition, from 3.33 mol% to 2 mol%.
System Identification 11	Mar. 10, 2009	Repeat system identification 9; trying for repeatability because of problems in system identification 10.
System Identification 12	Mar. 18, 2009	Analyze response of column to change in feed flow rate, from pump setting 2 to pump setting 3, and an increase in heat from 1500W to 2300W.
System Identification 13	Mar. 24, 2009	Repeat System Identification 10: change in feed composition, from 3.33 mol% to 2 mol%.
System Identification 14	Mar. 26, 2009	Analyze response of column to change in reboiler heat, from 1500W to 1660W.
System Identification 15	Mar. 28, 2009	Change in feed flow rate form pump setting 2 to pump setting 3 and change in reboiler heat from 1500W to 1660W. Feed is purchased ethanol water mixture.

Table 3.1 Cont.

System Identification 16	Apr. 25, 2009	Change in feed flow rate from pump setting 2 to pump setting 3 and change in reboiler heat from 1500W to 1660W. Feed is purchased ethanol water mixture with methanol removed.
Feedback Control 1	Mar. 21, 2009	Manual feedback control, attempting to maintain tray 1 temperature at same level after disturbance. Change in feed flow rate, from pump setting 2 to pump setting 3, and increase in heat from 1500W to 2300W.
Feedback Control 2	May 2, 2009	Manual feedback control, attempting to maintain reboiler temperature at same level after disturbance. Change in feed flow rate, from pump setting 2 to pump setting 3. Reflux percent 66%.
Feedback Control 3	May 6, 2009	Manual feedback control, attempting to maintain reboiler temperature at same level after disturbance. Change in feed flow rate, from pump setting 2 to pump setting 3. Reflux percent 75%.

3.1 Experiment: System Identification 1

Date Performed: November 25, 2008

3.1.1 Objectives

The objective of this experiment was to obtain gain (K_C) and response time (τ) values for the distillation column in the UTC Controls Laboratory. It was decided to try to obtain these values for the following circumstances:

Changing the reflux percent (holding heat and flow rate constant)

- 66% to 33%
- 33% to 66%
- 66% to 83%
- 83% to 66%

Changing the reboiler heat (holding reflux and flow rate constant)

- 1500 watts to 2000 watts
- 2000 watts to 1500 watts

Changing the feed pump flow rate (holding reflux and heat constant)

- setting 2 to setting 3
- setting 3 to setting 4
- setting 4 to setting 3
- setting 3 to setting 2

In all cases, the observed variable is the temperature in tray 1.

3.1.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. Once this happened, the column was changed to the desired operating point conditions for the first run:

- the heat at 1500 watts
- reflux at 66%
- the feed pump on setting 2.

The tray 1 temperature was observed until it appeared to reach steady state. At that point the reflux percent was lowered to 33%, and tray 1 was observed until it again appeared to reach steady state. This was repeated for the steps from 33% to 66%, 66% to 83%, and 83% to 66%. At this point, the experiment was ended because of time constraints.

3.1.3 Observations

The most significant observation was that the column response times were much longer than had been anticipated.

Using the live graph produced by LabView to determine when the system reached steady state has proven to be unreliable. The on-screen display shows approximately ten minutes worth of data; the temperature changes within this period of time can be small enough to make visual observation inadequate for making the judgment on when steady state has been achieved. The full-scale plot of the data shows that the temperature of tray 1 is still changing at the times the system was altered for the next step.

3.1.4 Conclusions

Since the 33% reflux resulted in a distillate composition that is very impure (approximately 4.25 mol% ethanol), it will no longer be used in the evaluation of the column performance.

The system never reached steady state, making the data acquired in this experiment unusable for the determination of controller variables; the experiment must therefore be repeated, allowing more time for the tray 1 temperature to reach steady state between steps. In addition, since so much time will be required for each step, the primary focus will be on just changing the reflux percent, leaving heat and flow rate changes for a later time.

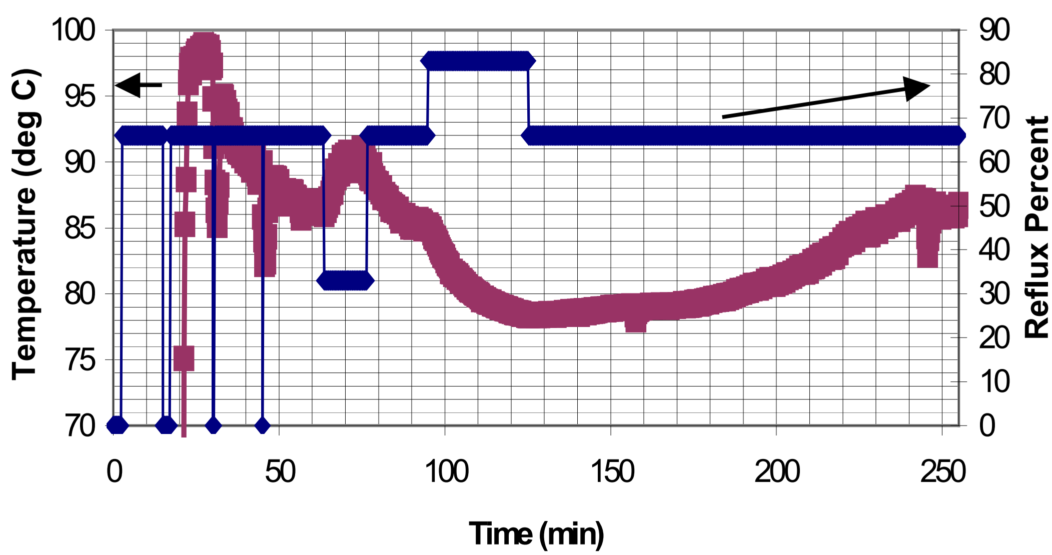

Figure 3.1. Tray temperatures never reached steady state

3.2 Experiment: System Identification 2

Date Performed: February 5, 2009

3.2.1 Objectives

This experiment was a repeat of the System Identification 1 experiment, with the same goal of acquiring gain and response time values. After the results of the previous experiment were analyzed, it was decided to limit the reflux percent values being tested to a smaller range than previously studied. The desired steps are:

Changing reflux percent (holding heat and flow rate constant)

- 66% to 70%
- 70% to 75%
- 75% to 70%
- 70% to 66%

3.2.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. Once this happened, the column was changed to the desired operating point conditions for the first run:

- the heat at 1500 watts

- reflux at 66%
- the feed pump on setting 2

The tray 1 temperature was observed until it appeared to reach steady state. At that point the reflux percent was raised to 70%, and tray 1 was observed until it again appeared to reach steady state. This was repeated for the steps from 70% to 75%, 75% to 70%, and 70% to 66%.

3.2.3 Observations

As in the previous experiment, the live graph produced by LabView was an unreliable indicator of when steady state had been achieved. The column was allowed to run for a longer time at each step to try to reach steady state, but it appears to still not be long enough.

One important observation is that the column reacts much more quickly to an increase in reflux percent than it does to a decrease in reflux percent. The response times for tray 1 are listed in Table 3.2, and show that the trays took twice as long to reach steady state after a drop in reflux percent as for an increase.

Table 3.2. Tray 1 Response Times

	Response time	
Reflux values	Step up	Step down
66% and 70%	7 min.	14.33 min.
70% and 75%	9.33 min.	17 min.

3.2.4 Conclusions

The chosen reflux percents seem to be a good range for the chosen feed composition and flow rate, since they show a reasonably good purification without saturating the top of the column with ethanol azeotrope. Follow-up analysis proved that the column was still undergoing change at the times when the steps occurred, so a longer period is needed between steps. In Figure 3.2 below, the temperature of tray 1 is still decreasing at the time the reflux percent is changed from 70% to 75%. To allow more time, it would probably be advisable to limit the number of steps on subsequent experiments. Repeating this analysis with only one or two steps will be the next experiment.

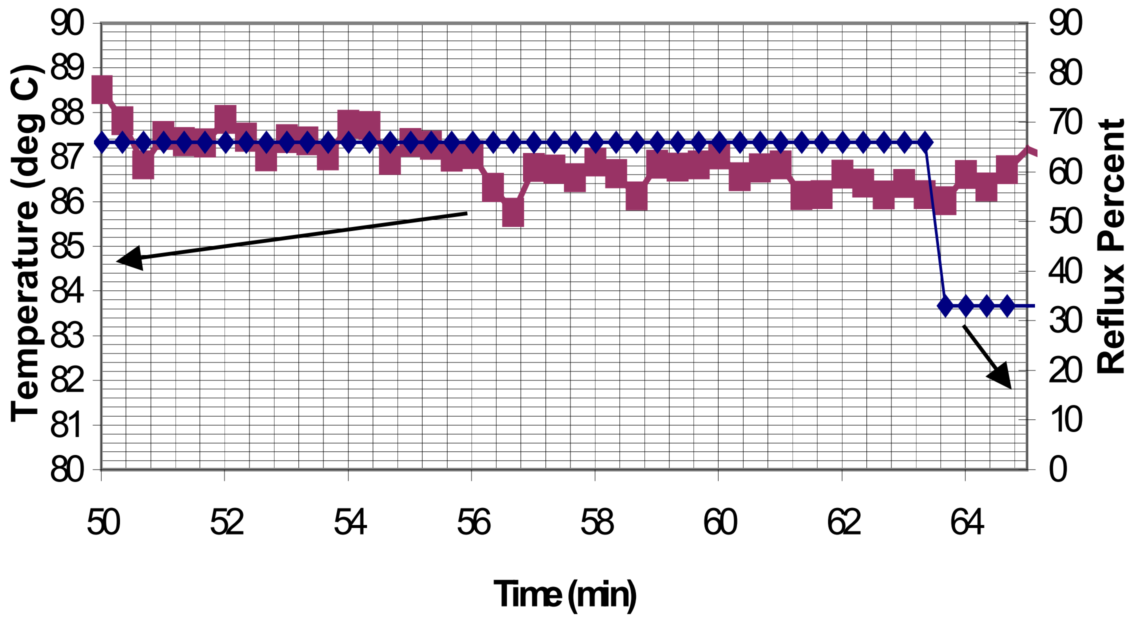

Figure 3.2. Temperatures still changing

3.3 Experiment: System Identification 3

Date Performed: February 14, 2009

3.3.1 Objectives

This experiment is a continuation of the previous two experiments, with the same goal of obtaining gain and response time values. For this run, the experiment will be limited to only two step changes in the reflux percent:

Changing reflux percent (holding heat and flow rate constant)

- 75% to 70%
- 70% to 66%

3.3.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. At this point, the procedure was changed from the previous experiments. Once the temperature in tray 1 had risen, the column was changed as follows:

- the heat to 1500 watts
- the feed pump on setting 2
- reflux was left at 100%.

The column was left on total reflux until the top portion of the column appeared to have started to stabilize, at which point the reflux was changed to 75%. The column was to be left alone for one hour, at which point the reflux would be changed to 70%.

3.3.3 Observations

Less attention was paid to the live graph this time, but after the hour at 75% reflux had elapsed, it was obvious that the column had still not achieved steady state. It was decided to allow the column to keep operating at 75% reflux until steady state had been reached. This did not occur for almost another four hours. A puzzling change in the trend of the tray temperatures occurred at approximately 350 minutes. They all showed a sudden drop, followed by a period of decline until they stabilized 40 minutes later, at which time the reflux percent was changed to 70%.

3.3.4 Conclusions

I have no explanation for the behavior of the temperatures in the period around 350 minutes. No unusual occurrences were noted in the column. The very long time required for the column to reach steady-state suggests that it might not be advisable to keep the column at 100% percent reflux for so long after start-up. An experiment repeating the 100% to 75% portion of this experiment might be necessary to try to determine the cause of the puzzling temperature behavior, as well as to evaluate whether the total reflux period should be shortened or even eliminated.

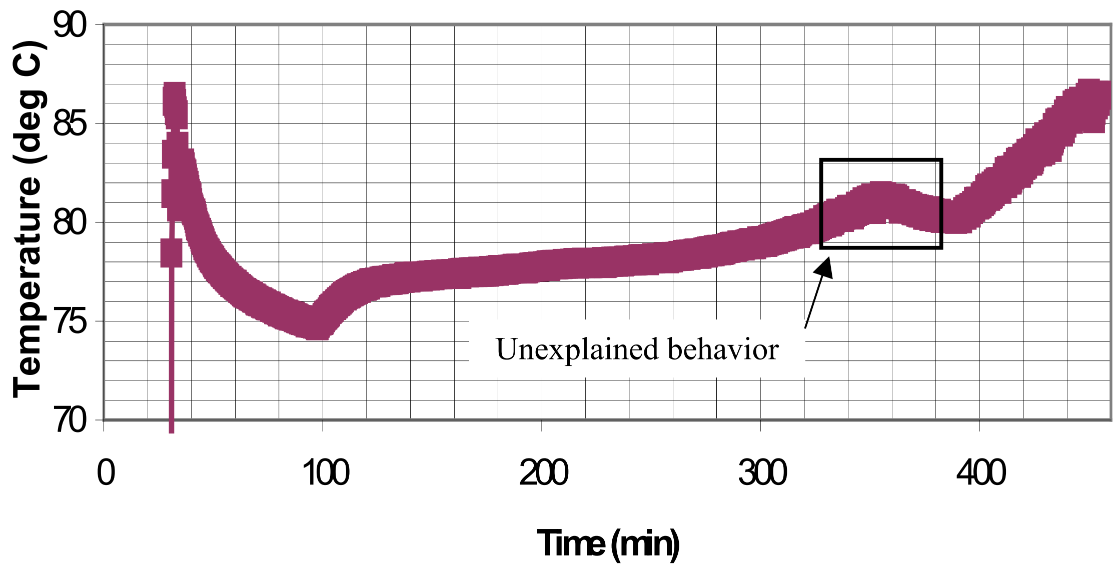

Figure 3.3. Unexplained bump in temperatures

3.4 Experiment: System Identification 4

Date Performed: February 17, 2009

3.4.1 Objectives

The purpose of this experiment was to evaluate the performance of the column at the same conditions used for the previous experiment, System Identification 3. There are two reasons for the repeat:

1. to see if the puzzling bump in the tray temperatures reoccurs and can be explained;
2. to determine if the total reflux period at the beginning of the experiment needs to be shortened.

3.4.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. At this point, the procedure from the previous experiment was to be repeated. Once the temperature in tray 1 had risen, the column was changed as follows:

- the heat to 1500 watts
- the feed pump on setting 2
- reflux was left at 100%.

The column was left on total reflux until the top portion of the column appeared to have started to stabilize, at which point the reflux was changed to 75%.

3.4.3 Observations

Neither the temperature bump nor the long transient period observed during the System Identification 3 experiment occurred during this experiment. In addition, the steady-state temperatures reached were very different from the previous experiment.

3.4.4 Conclusions

Based on a comparison to the steady state temperatures reached in the pervious experiments, it is apparent that an unknown factor has affected this experiment, making the data unusable. From the high tray temperatures and lower compositions recorded during the steady state period, it is possible that the batch of feed was mixed incorrectly, resulting in a feed with a lower concentration than desired.

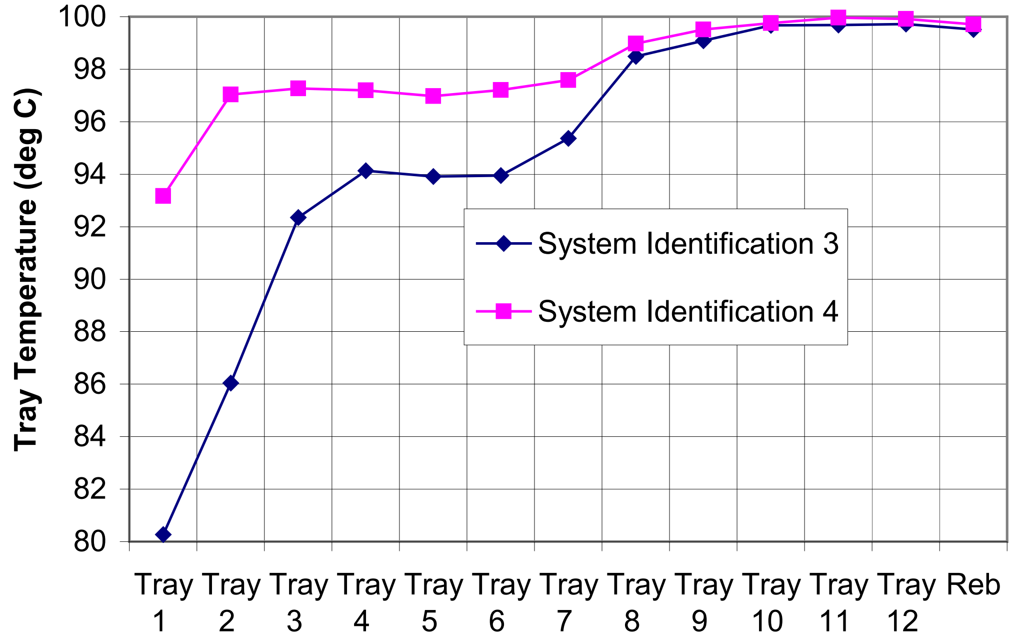

Figure 3.4. Lower temperatures than previously recorded

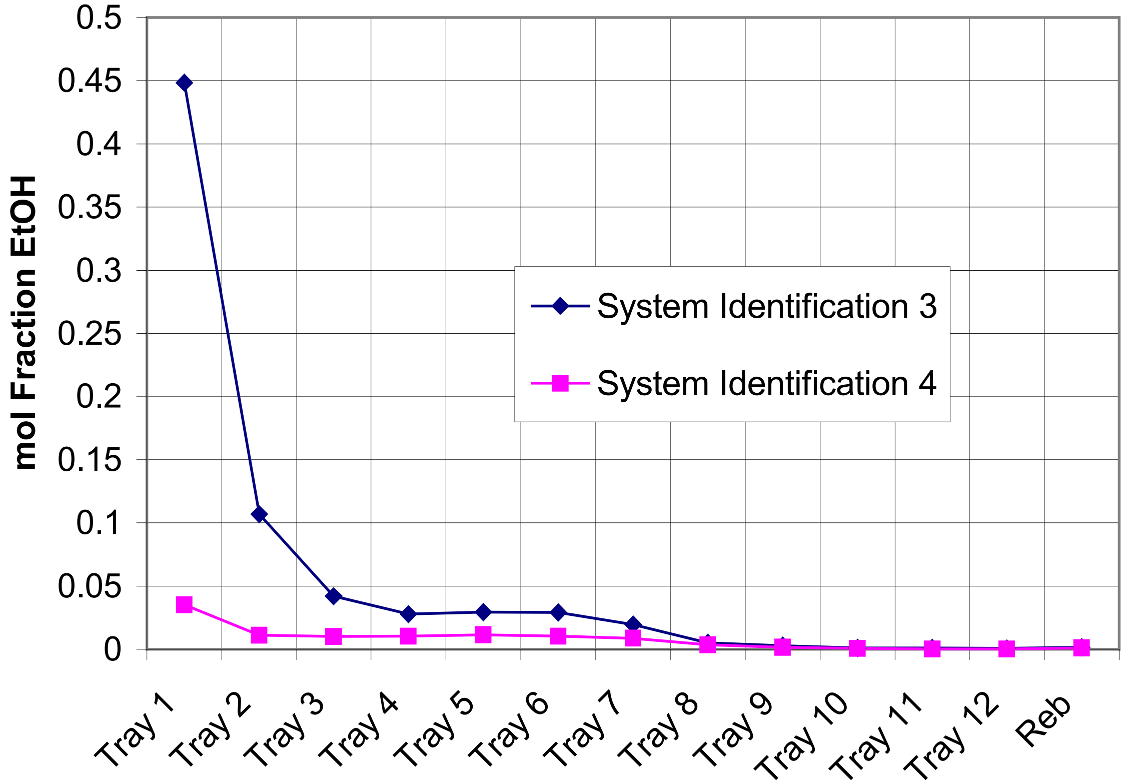

Figure 3.5. Tray compositions much lower

3.5 Experiment: System Identification 5

Date Performed: February 21, 2009

3.5.1 Objectives

Since the System Identification 4 experiment showed neither of the problems of the System Identification 3 experiment, this experiment will attempt to repeat the System Identification 3 experiment, and achieve the control data for the same operating conditions:

Changing reflux percent (holding heat and flow rate constant)

- 75% to 70%

- 70% to 66%

3.5.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. Once the temperature in tray 1 had risen, the column was changed as follows:

- the heat to 1500 watts
- the feed pump on setting 2
- reflux left at 100%

The column was left on total reflux until the top portion of the column appeared to have started to stabilize, at which point the reflux was changed to 75%. The column was to be left alone for one hour, at which point the reflux would be changed to 70%.

3.5.3 Observations

The unexplained bump in the tray temperatures occurred several times during this experiment. This is shown in the chart below; tray 5 was used as a representative example.

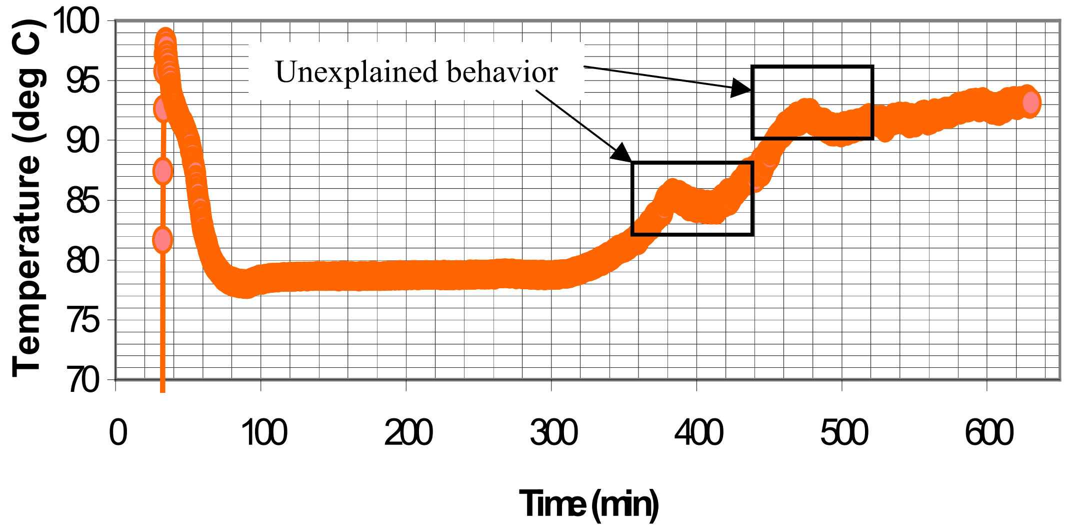

Figure 3.6. Unexplained bump in temperatures reoccurs

The column reached steady state much more quickly after the change from 100% to 75% reflux than occurred for the System Identification 3 experiment. The top five trays reached it within 40 minutes of the change, while trays 6-9 took roughly 3.5 hours. This is still a long time, but is considerably shorter than the five hours needed previously. The step from 75% to 70% needed roughly the same amount of time, while the change to 66% ran out of time before steady state was reached.

3.5.4 Conclusions

The cause of the bumps in the tray temperatures is still unknown. Careful observation of the column during operation revealed no clues. The column will be monitored closely on subsequent experiments to continue to find the cause.

Keeping the column at total reflux at the beginning of the experiment, while allowing for reliably repeatable start-up conditions, causes there to be a long delay before the first step can occur. The total reflux causes ethanol to build up to high concentrations in most of the column; at high reflux percents, it takes a long time to remove this excess ethanol. Further experiments to test this start-up procedure with a lower reflux percent (66%) will be conducted.

3.6 Experiment: System Identification 6 and System Identification 7

Dates Performed: February 24, 2009 and February 25, 2009

3.6.1 Objectives

These experiments will test the current total reflux start-up period with a change to 66% reflux. The goal is to observe the speed of the response with a lower reflux percent than was used previously to see if the column would reach steady state more quickly. The total reflux period will be run for two different lengths, to analyze the effect the amount of time spent at total reflux has.

3.6.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. Once the temperature in tray 1 had risen, the column was changed as follows:

- the heat to 1500 watts

- the feed pump on setting 2
- reflux left at 100%

The column was left on total reflux for a pre-determined length of time, at which point the reflux was changed to 66%. For the System Identification 6 experiment, the total reflux period lasted for roughly 2 hours; for the System Identification 7 experiment, it lasted for 1 hour.

3.6.3 Observations

For both experiments, the feed tank ran out before steady state had been reached. Comparing a few of the tray temperatures (shown in the table below), it can be seen that the trays reacted approximately 10 minutes faster with the shorter period of total reflux. (The temperature used is an arbitrary value.)

Table 3.3. Approximate time (in min.) to reach 82 ^{O}C

Experiment	Tray 4	Tray 5	Tray 6
System Identification 6	140	120	70
System Identification 7	130	110	60

3.6.4 Conclusions

While shortening the length of the total reflux period does speed the response of the column, the length of time to reach steady state is still too long. The experiment with the shorter period of total reflux had only begun to show a response in the temperature of tray 1 three hours after the change had occurred. This makes the experiments too long, and is wasteful of the ethanol. The period of total reflux will be reduced to no more that 15 minutes in all further experiments.

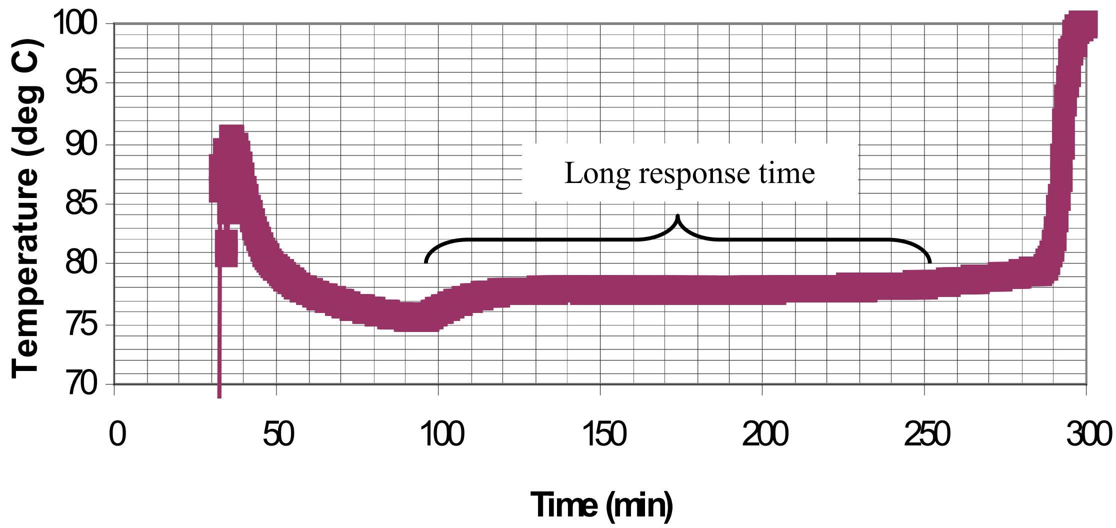

Figure 3.7. The long response time after total reflux

3.7 Experiment: System Identification 8

Date Performed: February 26, 2009

3.7.1 Objectives

After the analysis of many of the previous experiments showed the response times tended to be in the range of 10 to 15 minutes, it was decided to perform a single experiment changing the reflux percent several times, with each step restricted to a period of the same length, approximately 50 minutes. The goal is to see how close to steady state the system gets within this time constraint.

Changing reflux percent (holding heat and flow rate constant)

- 66% to 75%
- 75% to 80%
- 80% to 75%

- 75% to 66%

3.7.2 Procedure

The column was started up with the following settings:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise. Once the temperature in tray 1 had risen, the column was changed as follows:

- the heat to 1500 W
- the feed pump on setting 2
- and reflux left at 100%

The column was left on total reflux until the experiment reached the 50-minute mark (approximately 15 minutes). At the 50-minute mark, the reflux percent was lowered to 66%. After 50 minutes, the reflux percent was changed to 75%. This was repeated for the remaining 3 changes.

3.7.3 Observations

The 50-minute period was insufficient to allow all of the change to occur for each step, as they were all still showing changing temperatures at the time of the next step.

3.7.4 Conclusions

All attempts to allow multiple step changes within the scope of a single experiment have failed. It has become obvious that all further experiments must be limited to a single step.

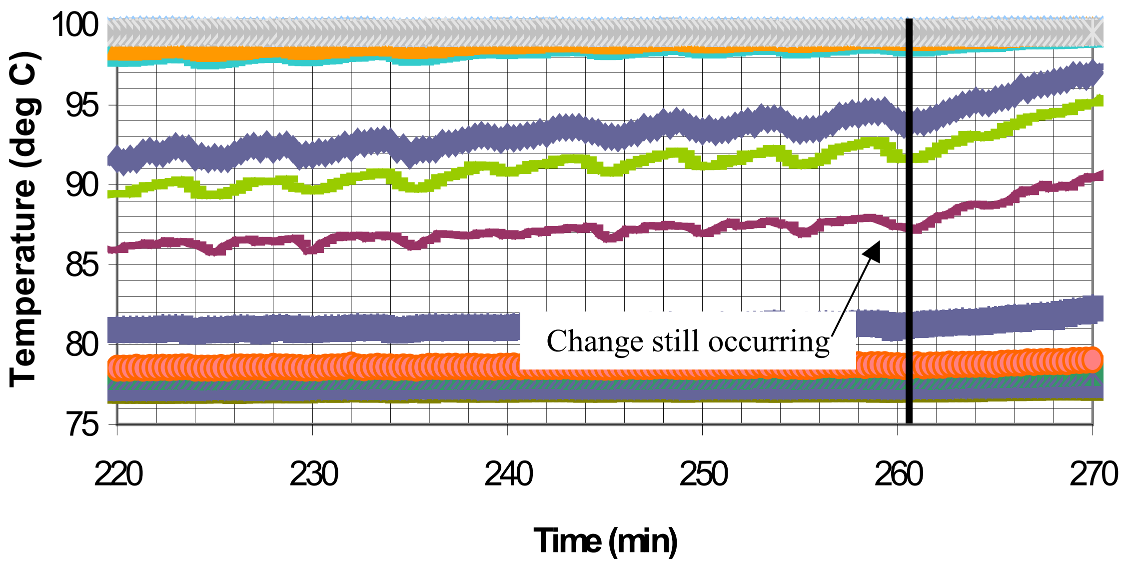

Figure 3.8. Temperatures still changing

3.8 Experiment: System Identification 9

Date Performed: March 3, 2009

3.8.1 Objectives

The focus of the thesis has shifted from an analysis of the control system for the distillation column to an analysis of the dynamic behavior of the column. From the previous experiments, 66% reflux has proven to be a reasonable reflux percent for a baseline, so that will be used for most of the following experiments. This experiment will examine the response of the column to a change in feed flow rate.

3.8.2 Procedure

A new start-up procedure has been developed to allow consistency and repeatability in all further experiments. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until 150 minutes had elapsed in the experiment. At that time, the feed pump was changed to a speed setting of 3 and all the other parameters were left the same. The column was then run for a further 150 minutes.

3.8.3 Observations

The new start-up procedure allowed enough time for the column to reach approximate steady state before the step occurred; the chart below shows some continuing change, but it is minor. The change in the feed pump showed a rapid and fairly dramatic decrease in the tray temperatures. The change was most noticeable in the bottom section of the column.

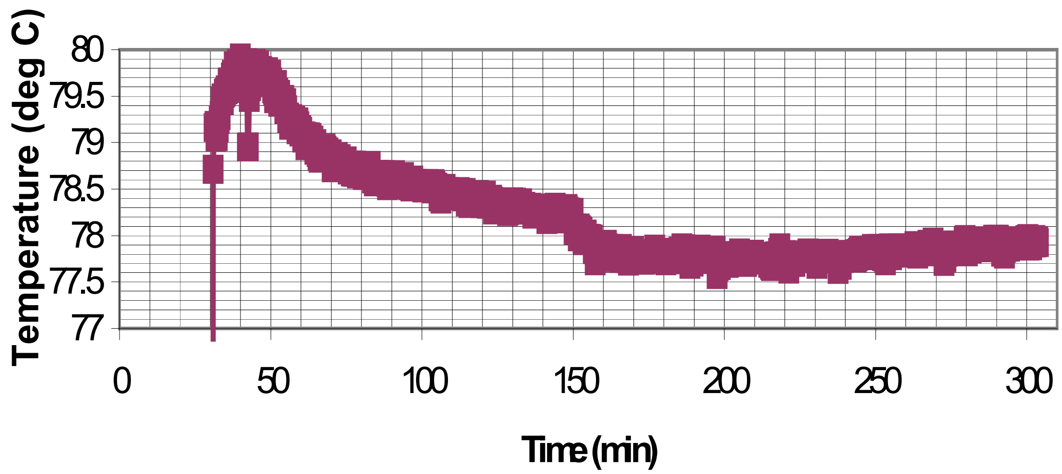

Figure 3.9. Tray 1 reaches steady state after the disturbance

3.8.4 Conclusions

The lowered temperatures on all trays in the column show that an increase in the flow rate of the feed into the column serves to effectively increase the effectiveness of the separation; the column produces a purer distillate. Unfortunately, the reboiler shows a higher ethanol concentration, meaning more ethanol is leaving the column in the bottoms product. Since the bottoms product is being treated as a waste stream and disposed of, this means that more ethanol is being lost.

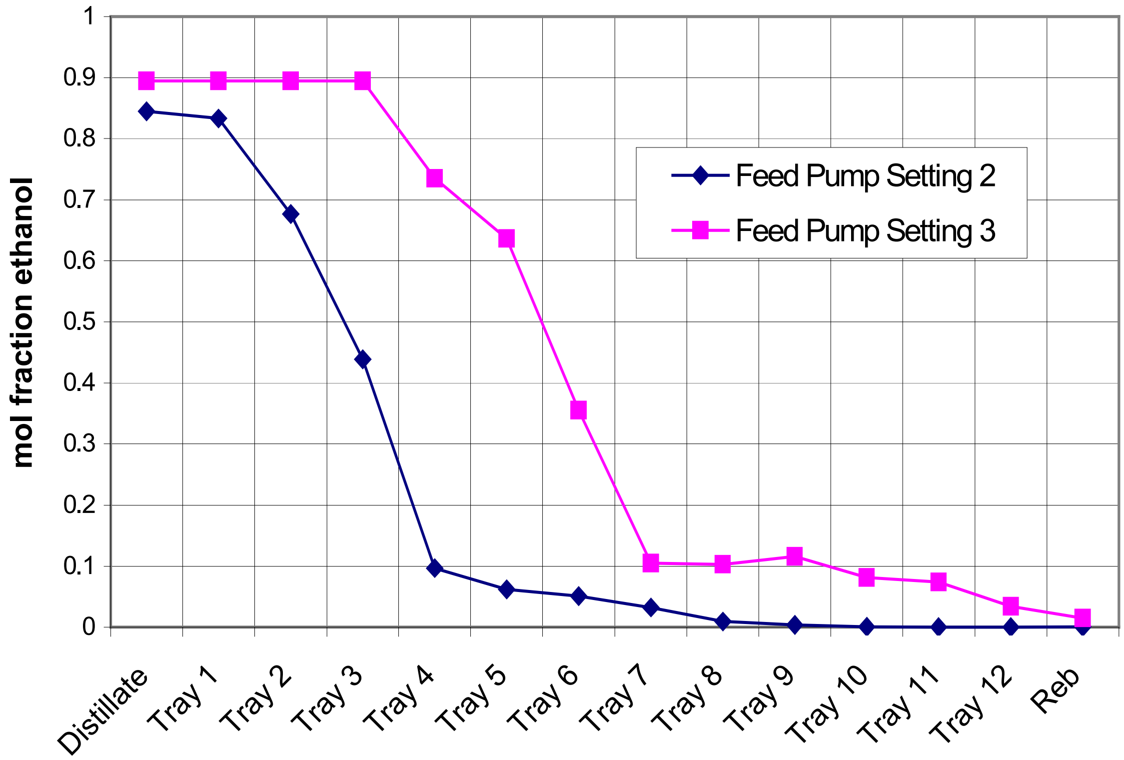

Figure 3.10. Concentration front, change in feed flow rate

3.9 Experiment: System Identification 10

Date Performed: March 5, 2009

3.9.1 Objectives

This experiment was a continuation of the evaluation of the dynamic response of the distillation column. For this experiment, the composition of the feed is what was changed, from a starting concentration of 3.33 mol% to 2 mol%.

3.9.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until 208 minutes had elapsed in the experiment. At that time, the intake for the feed pump was switched to a separate tank, which contained at feed at 2 mol% ethanol, while all the other parameters were left the same. The column was then run for a further 150 minutes.

3.9.3 Observations

With the column start-up being the same as the System Identification 9 experiment, the tray temperatures would be expected to behave in a similar manner. However, the trays were trending towards higher temperatures than had been observed previously. After some observation of the column, some air bubbles were noticed in the feed line. Since these could affect the feed flow rate into the column, they needed to be removed. The simplest way to accomplish this is to increase the speed of the feed pump; this occurred at approximately 155 minutes, and is the reason for the disturbance shown. However, this was obviously not the only problem with the experiment. While the temperatures did not continue increasing, they did not decrease back to the levels seen in the previous experiment. The feed composition was changed as planned, but the reliability of the data is questionable.

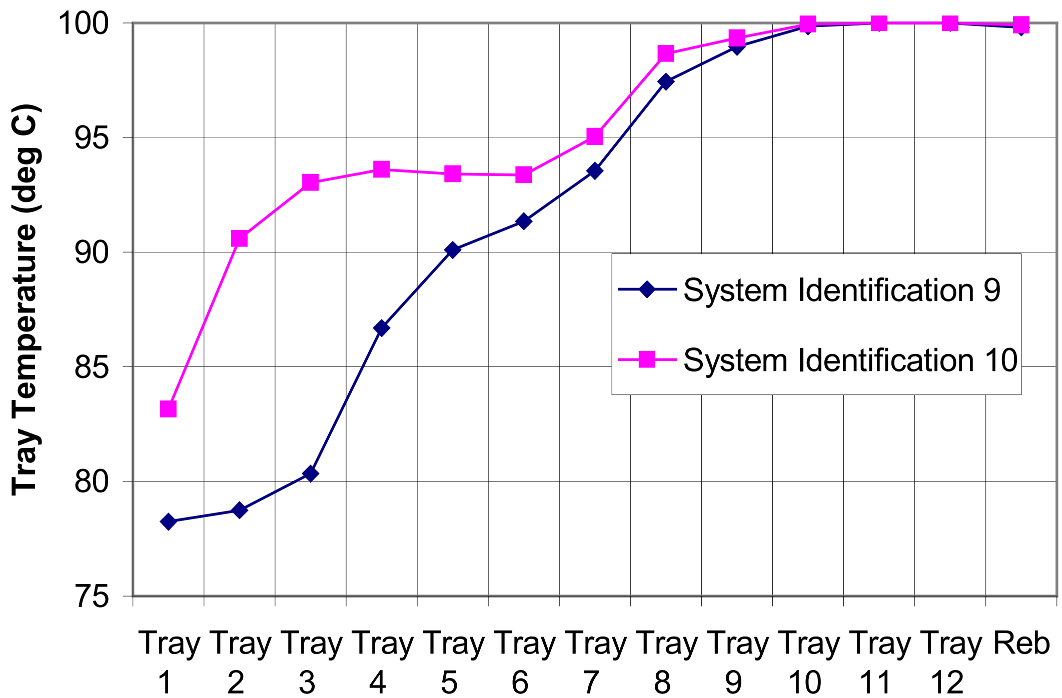

Figure 3.11. Tray temperatures for Sys. ID 9 and Sys. ID 10

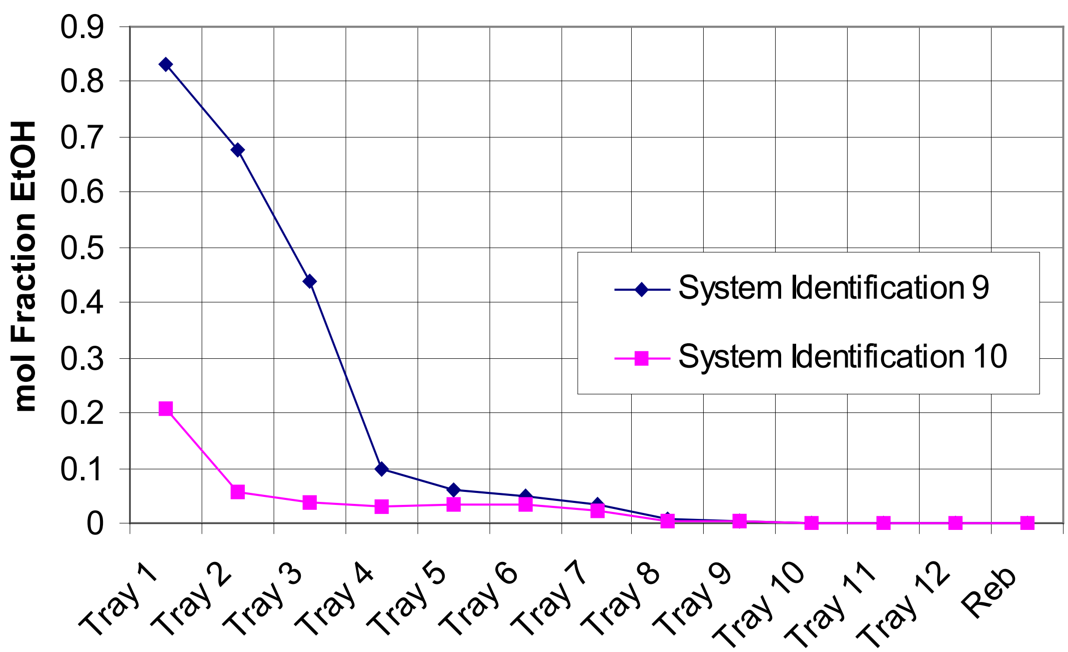

Figure 3.12. Tray compositions very different

3.9.4 Conclusions

The higher tray temperatures and lower compositions at the steady state condition suggest that the feed might have been prepared incorrectly, resulting in a lower concentration than desired. Because of the questions, this experiment will need to be repeated.

3.10 Experiment: System Identification 11

Date Performed: March 10, 2009

3.10.1 Objectives

After the previous experiment failed to reach the same steady-state conditions during the start-up phase, it was decided to repeat the experiment where the feed flow rate was changed (System Identification 9), to see if it was reproducible.

3.10.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W

- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until 150 minutes had elapsed in the experiment. At that time, the feed pump was changed to a speed setting of 3 and all the other parameters were left the same. The column was then run for a further 150 minutes.

3.10.3 Observations

The data from this experiment (shown in the concentration fronts, below) match closely with that from the previous attempt (System Identification 9), so the System Identification 10 experiment will be thrown out and repeated.

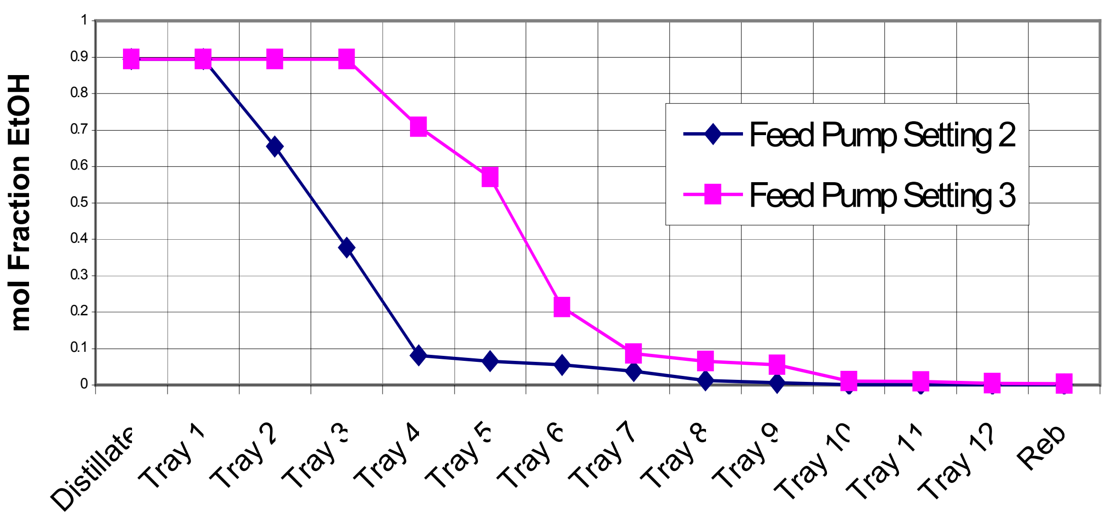

Figure 3.13. Concentration front, change in feed flow rate

3.11 Experiment: System Identification 12

Date Performed: March 18, 2009

3.11.1 Objectives

It was decided to run an experiment where the feed flow rate was increased as in the System Identification 9 and System Identification 11 experiments, but an attempt would be made to account for the increased fluid entering the column by increasing the reboiler heat by the amount necessary to bring that fluid to the operating conditions of the column.

3.11.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until 150 minutes had elapsed in the experiment. At that time, the feed pump was changed to a speed setting of 3 and the reboiler heat was increased to 2300W, while all the other parameters were left the same. The column was then run for a further 150 minutes.

3.11.3 Observations

The increase in heat maintained the bottom of the column at the same conditions after the step as before; the top of the column, however, showed a noticeable decrease in purity.

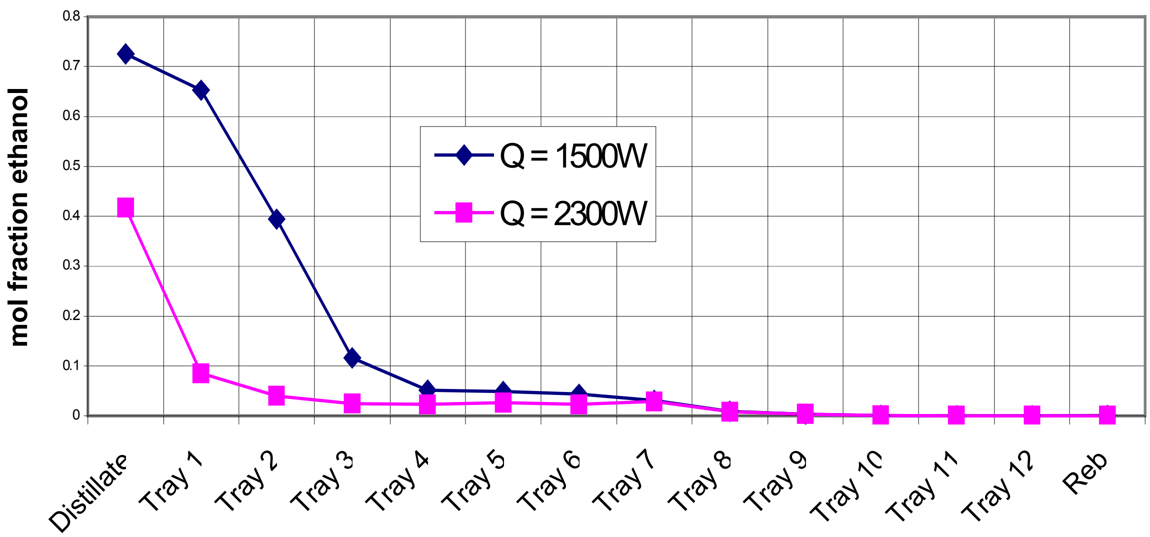

Figure 3.14. Concentration front, change in reboiler heat and feed flow rate

3.11.4 Conclusions

Since the behavior of the column above and below the feed depend on different ratios (the reflux and boil-up ratios), and since the ratios are controlled via different variables, it is apparent that a change in only one of these variables is insufficient to maintain the column at the identical conditions after the disturbance as before.

3.12 Experiment: Feedback Control 1

Date Performed: March 21, 2009

3.12.1 Objectives

Since the previous experiment showed that a simple heat increase by the amount necessary to account for the increased feed would not maintain the distillate at the same level, a new experiment was devised. The System Identification 12 experiment would be repeated exactly, with the following exception: a manual feedback control system would be implemented to attempt to maintain the tray 1 temperature at the level it was at when the step occurred.

3.12.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until 210 minutes had elapsed in the experiment. At that time, the feed pump was changed to a speed setting of 3 and the reboiler heat was increased to 2300W, while all the other parameters were left the same. The temperature in tray 1 was observed after the step had occurred; every minute, the heat was lowered by 10W per ^{o}C the temperature was above where it was at the step. This was continued until the temperature was less than a degree high and the system had reached steady state.

3.12.3 Observations

It took nine minutes for the temperature in tray 1 to increase by 1 degree, and it took another 35 minutes for it to return to less than one degree above its starting value. This resulted in the reboiler heat being lowered to a final value of 1660W. While the tray 1 temperature was the same as its starting value, none of the other trays were close. The rest of the column ended up being considerably richer in ethanol.

3.12.4 Conclusions

It is again obvious that a single control variable is not sufficient to maintain the entire column at the same point after a disturbance.

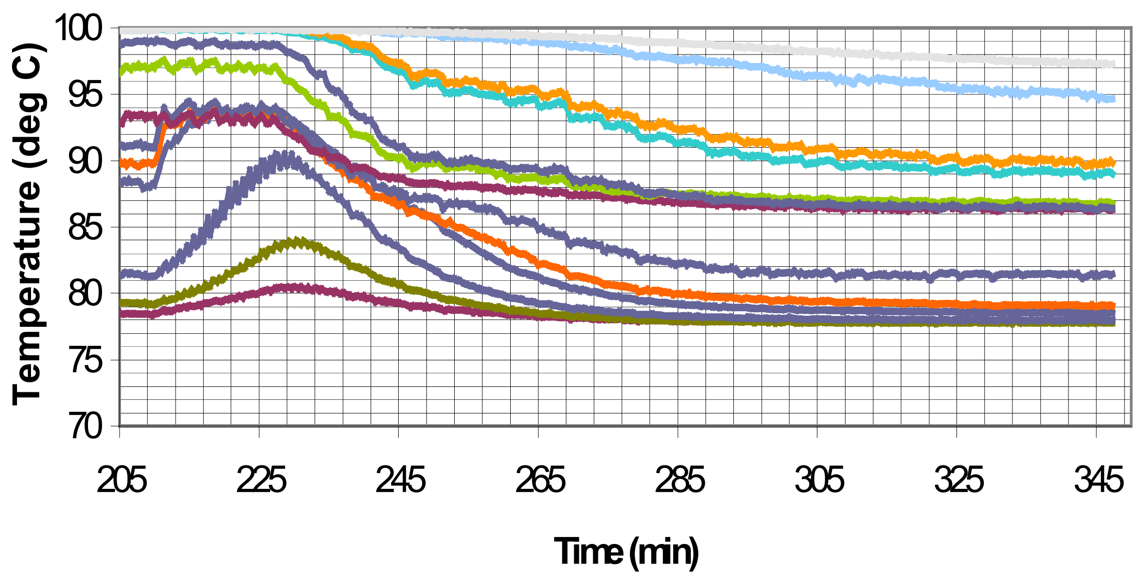

Figure 3.15. The effect of manual feedback control

3.13 Experiment: System Identification 13

Date Performed: March 24, 2009

3.13.1 Objectives

This e~~xperiment is a repeat of~~ the feed composition change experiment performed in System I Feedback control period

3.13.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until 160 minutes had elapsed in the experiment. At that time, the intake for the feed pump was switched to a separate tank, which contained at feed at 2 mol% ethanol, while all the other parameters were left the same. The column was then run for a further 150 minutes.

3.13.3 Observations

The start-up temperatures are much closer to all the other runs than they were during the original performance of this experiment (System Identification 10). As in the other experiments, the trays showed a rapid response to the disturbance. The bottom third of the column showed little response to the composition change, but the final concentration of the distillate dropped significantly from it's starting value.

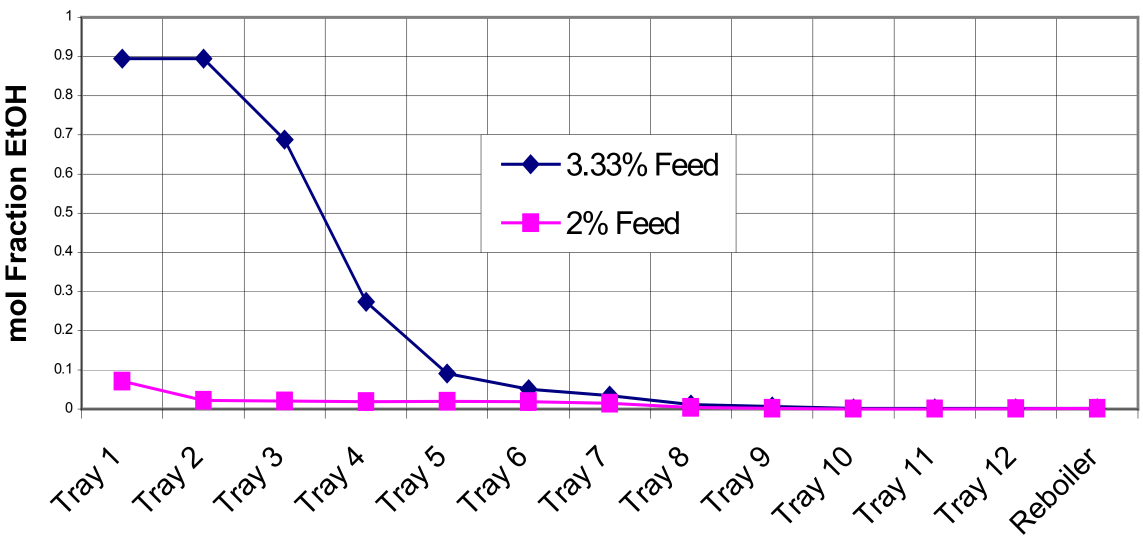

Figure 3.16. Concentration front, change in feed concentration

3.13.4 Conclusions

Since a change in the feed composition changes where the q line intersects the equilibrium diagonal (Figure 3.17), recovering after the disturbance to the same conditions as before will be more difficult than adjusting for a change in flow rate.

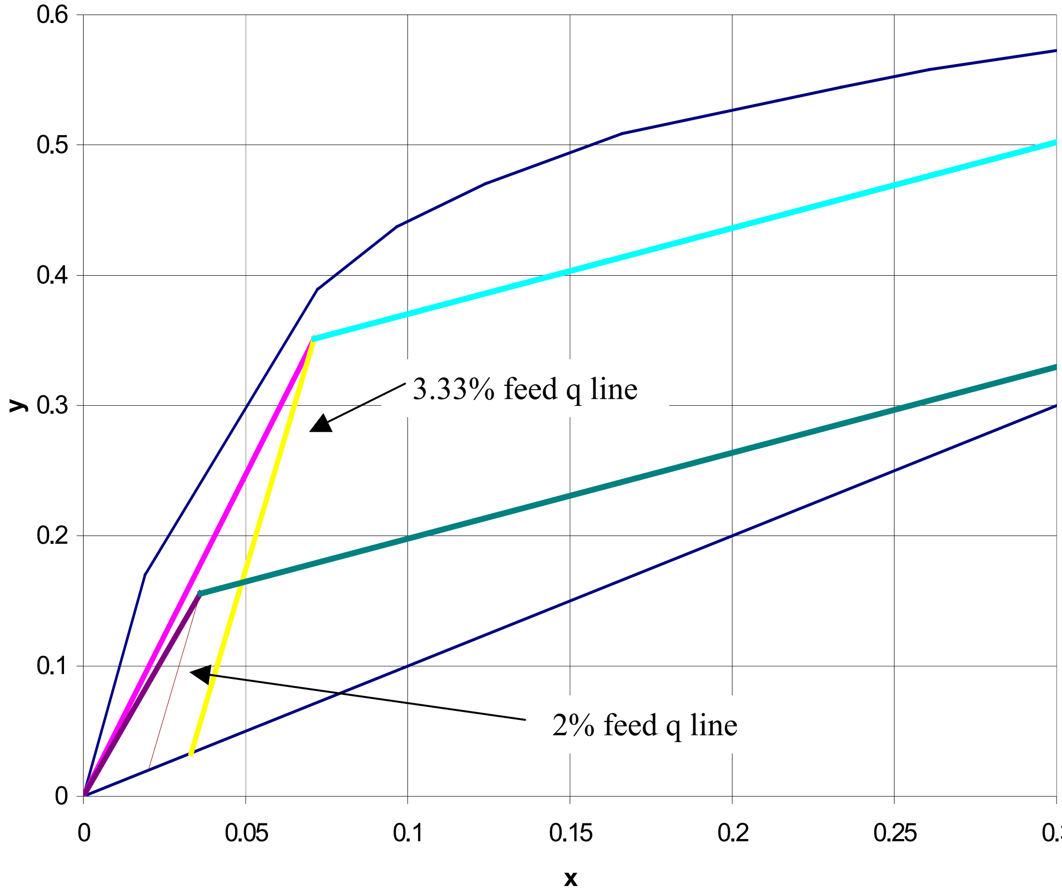

Figure 3.17. McCabe-Thiele operating lines, change in feed composition

3.14 Experiment: System Identification 14

Date Performed: March 26, 2009

3.14.1 Objectives

This experiment was run to examine the effect a change in reboiler heat would have in isolation. The heat value was chosen by selecting the final value reached in Feedback Control 1, the experiment where a feedback control system was approximated manually, 1660W.

3.14.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

Since the column was already warm because of an experiment that had been performed prior to this one, the column only needed to warm up for about 5 minutes. Once tray 1 had heated up, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was operated at these conditions until 90 minutes had elapsed in the experiment. At that time, the reboiler heat was increased to 1660W The column was then run for a further 150 minutes.

3.14.3 Observations

The middle of the column again showed a rapid response to the disturbance. The increase in heat raised the temperatures in all of the trays. The final purity of the distillate was considerably lower than the starting value.

3.14.4 Conclusions

Increasing the heat without also increasing the feed flow rate as in the previous experiments had a much more powerful effect on the column. The effect the heat input has on the boil-up ratio seems to be significant.

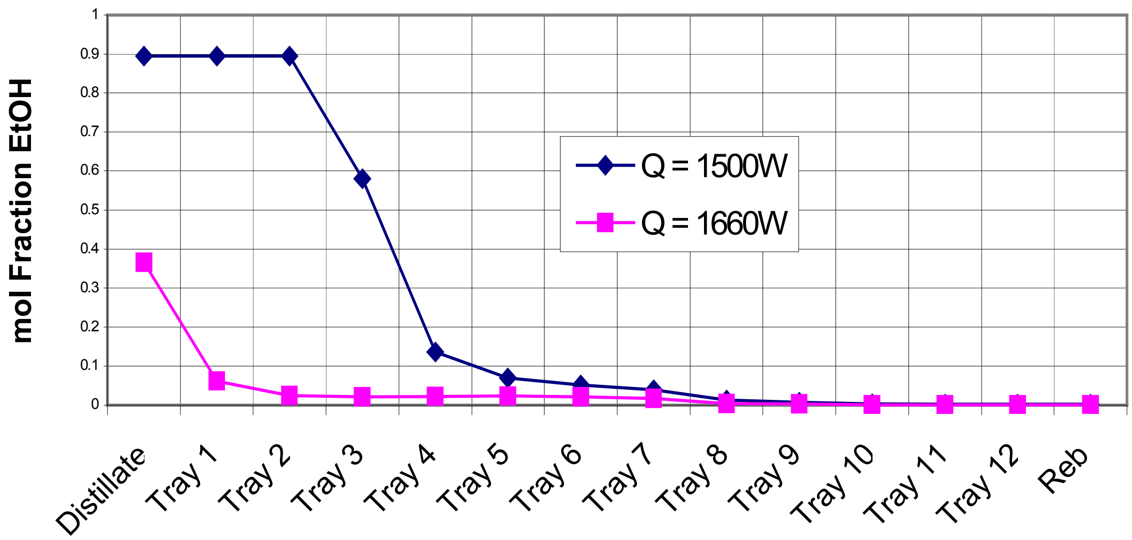

Figure 3.18. Concentration front, change in reboiler heat

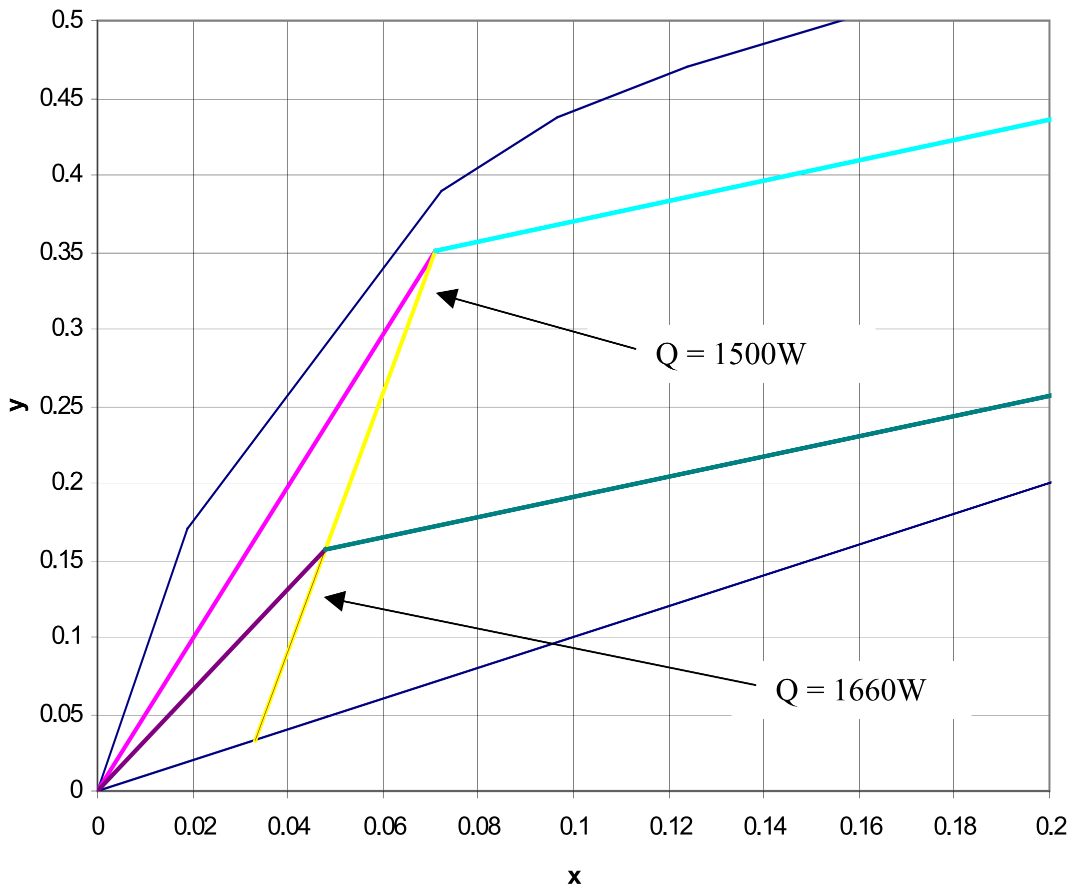

Figure 3.19. McCabe-Thiele operating lines, change in reboiler heat

3.15 Experiment: System Identification 15 and System Identification 16

Dates Performed: March 28, 2009 and April 25, 2009

3.15.1 Objectives

All previous experiments had been run using a purchased ethanol/water mixture that had been denatured using a small amount of methanol. A question had arisen as to how significant the effect of the methanol was. For this reason, two identical experiments were run: one with the purchased mixture, and one where an attempt had been made to remove as much of the methanol as possible. It was decided to use a change to feed setting 3 and an increase in reboiler heat to 1660W. Steady state values for these conditions had been recorded from previous experiments and were available for comparison if needed.

3.15.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to 66%

The column was allowed to operate at these conditions until the system was at steady state (this occurred at different times for the two experiments, since the System Identification 15 run had an issue with the feed supply early on). At that time, the feed pump was changed to setting 3 and the reboiler heat was increased to 1660W. The column was then run for a further 150 minutes.

3.15.3 Observations

A comparison of the two sets of data shows that, while the temperatures are slightly different, the behavior of the column is almost identical. The system responds in the same manner, and the profiles both before and after the disturbance are close enough that the presence of the methanol can be effectively ignored, making the previous data still usable.

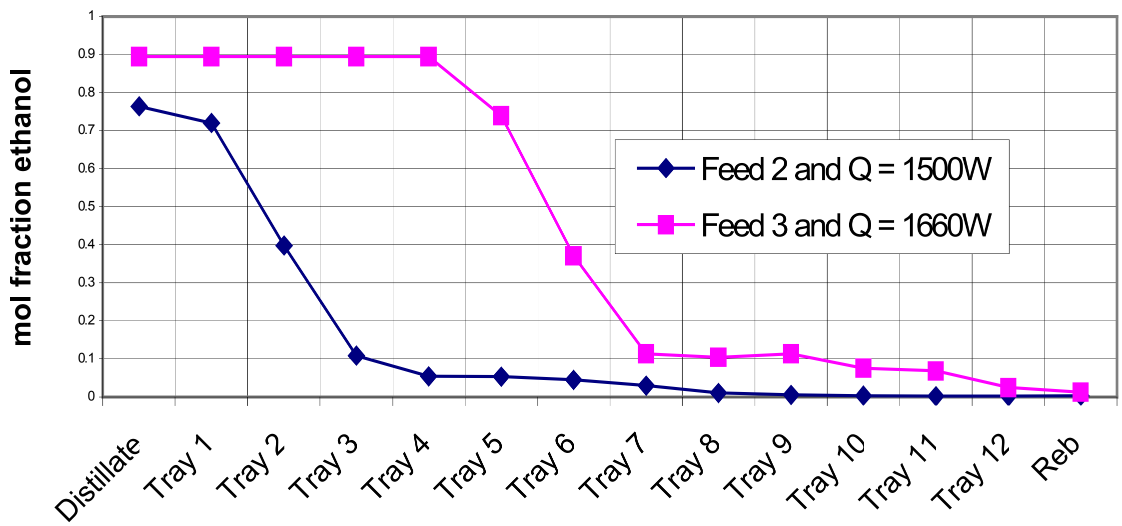

Figure 3.20. Concentration front, feed with methanol

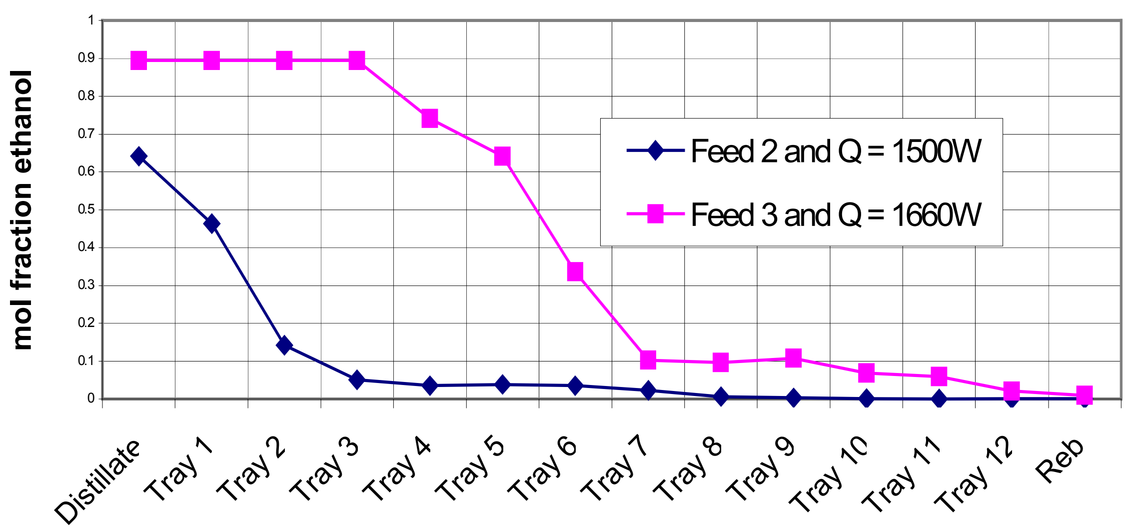

Figure 3.21. Concentration front, feed with methanol removed

3.16 Experiment: Feedback Control 2 and Feedback Control 3

Dates Performed: May 2, 2009 and May 6, 2009

3.16.1 Objectives

These experiments are similar to the Feedback Control 1 experiment simulating a feedback control system, but in this case, the reboiler temperature was being observed instead of the tray 1 temperature. In addition, the reboiler heat was not changed at the time of the step.

3.16.2 Procedure

The new start-up procedure is still being used. The experiment begins with the same procedure as before:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column was observed until the temperature in tray 1 began to rise, once this happened, the operating conditions were changed as follows:

- the reboiler heat was set to 1500W
- the feed pump was set to a speed setting of 2
- reflux was changed to
  - 66% for Feedback Control 2
  - 75% for Feedback Control 3

The column was allowed to operate at these conditions until steady state had been reached. The temperature in the reboiler was observed after the step had occurred; every minute, the heat was raised by 1W per 0.1 ^{o}C the temperature was below where it was at the step. This was continued until the temperature was less than 0.1 ^{o}C low and the system had reached steady state.

3.16.3 Observations

The behavior of the column during the two experiments was very similar. However, the higher reflux percent caused the temperatures to fall a little farther, and more significantly, to remain down for a longer period of time.

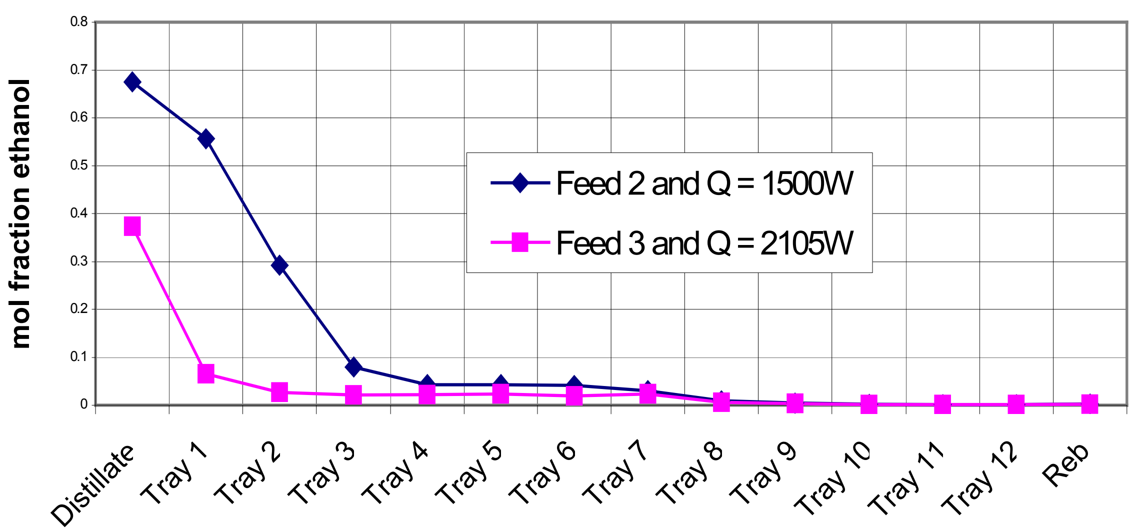

Figure 3.22. Concentration front, 66% reflux

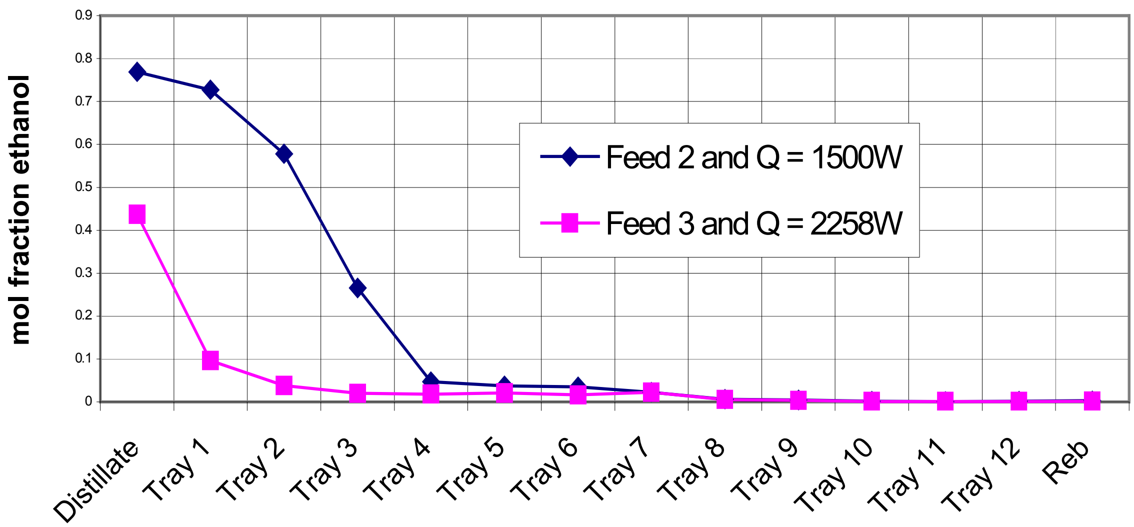

Figure 3.23. Concentration front, 75% reflux

4 Discussion

The experiments performed fell into two major categories:

1. identification of the important system parameters and their behavior.
2. experimental analysis of the response of the system to feedback control.

For the System Identification experiments, analysis of the data had two main goals: understanding how the trays respond to a disturbance, and understanding how the column as a whole responds. For the Feedback Control experiments, the analysis focused on simply understanding the response.

4.1 System Identification: Tray Response

It is important to evaluate the dynamics of the individual trays in order to optimize the response of a control system to a disturbance. Even though the ultimate goal of the separation is the purity of the product streams, simply monitoring the behavior of the composition (via the temperature) of the reboiler and tray 1 is insufficient to be able to efficiently control these values. The effect of a disturbance will most likely not be reflected in the extremes of the column for some time after the disturbance occurs. Experiments were run with each of the four changes occurring in isolation: feed flow rate, feed composition, reboiler heat and reflux percent.

Table 4.1. Experiments with parameters changing in isolation

Experiment	Changing Parameter	Change
System Identification 5	Reflux percent	75% to 70%
System Identification 9	Feed flow rate	Pump setting 2 to pump setting 3
System Identification 13	Feed composition	3.33 mol% to 2 mol%
System Identification 14	Reboiler heat	1500W to 1660W

For each of the experiments, the response times were calculated for each tray and the reboiler. Response time is the amount of time the temperature took to reach 63% of the way from its initial to its final value. The response times were then plotted (Figure 4.1.) The values for System Identification 5 were plotted on the secondary y-axis because of the longer times involved; the illustration of the general trends in the times within each experiment is the most significant point, not a comparison of times between experiments.

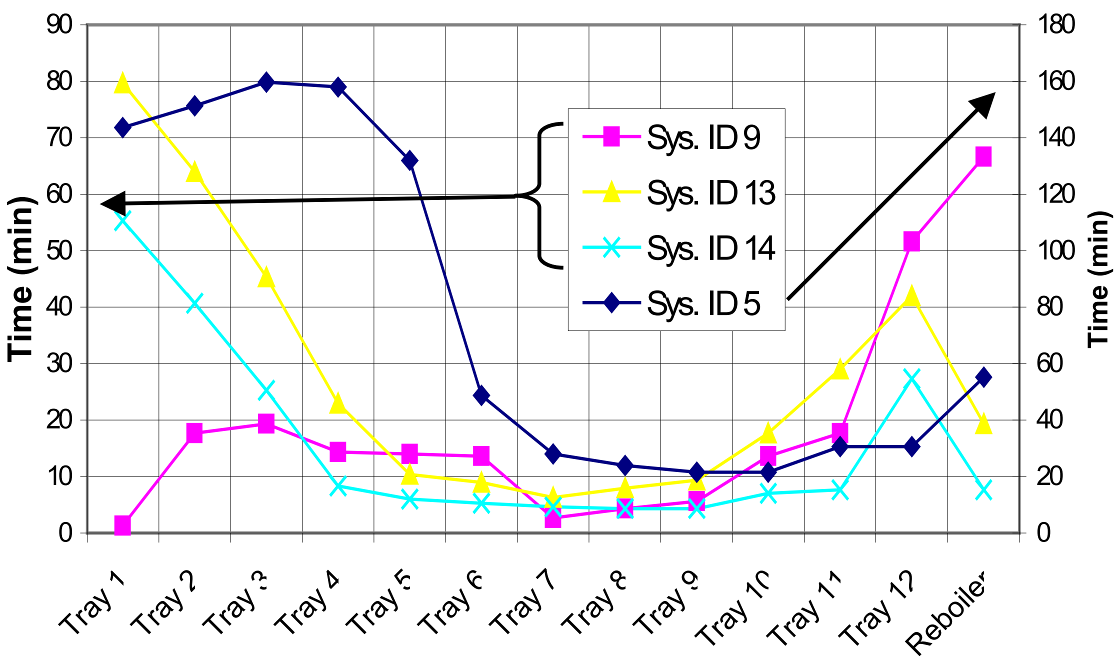

Figure 4.1. Tray response times for singles parameter experiments

In all of the experiments, the central portion of the column responds to the disturbance much more rapidly than the extremes do. The only exceptions that occur are at the very top and bottom of the column. For situations where the observed composition is close to being (or is actually) pure, the temperatures might show little, or even no, response. This is most likely to occur in the reboiler and tray 1, and is illustrated in the artificially low response times for the top and bottom of the column for several of the experiments shown in Figure 4.1.

While the response times are a good indicator of the overall response of the trays, they do not indicate how extreme the immediate response is for each tray. A better illustration of this comes from an examination of the slope of the temperature curves. The slope of each curve was calculated at each point after the change, and the point at which that slope was a maximum was determined; a plot of these times is shown in Figure 3.2. Because of the large differences in scale, experiments 5 and 9 are plotted on the left y-axis, while 13 and 14 are on the right axis.

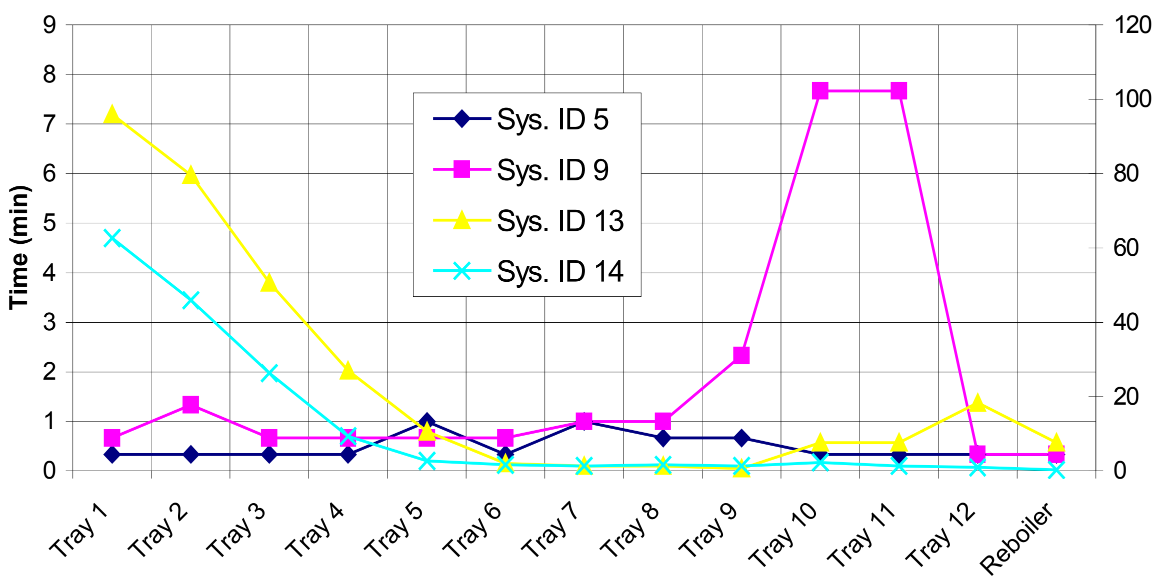

Figure 4.2. Time for tray temperatures to reach maximum slope

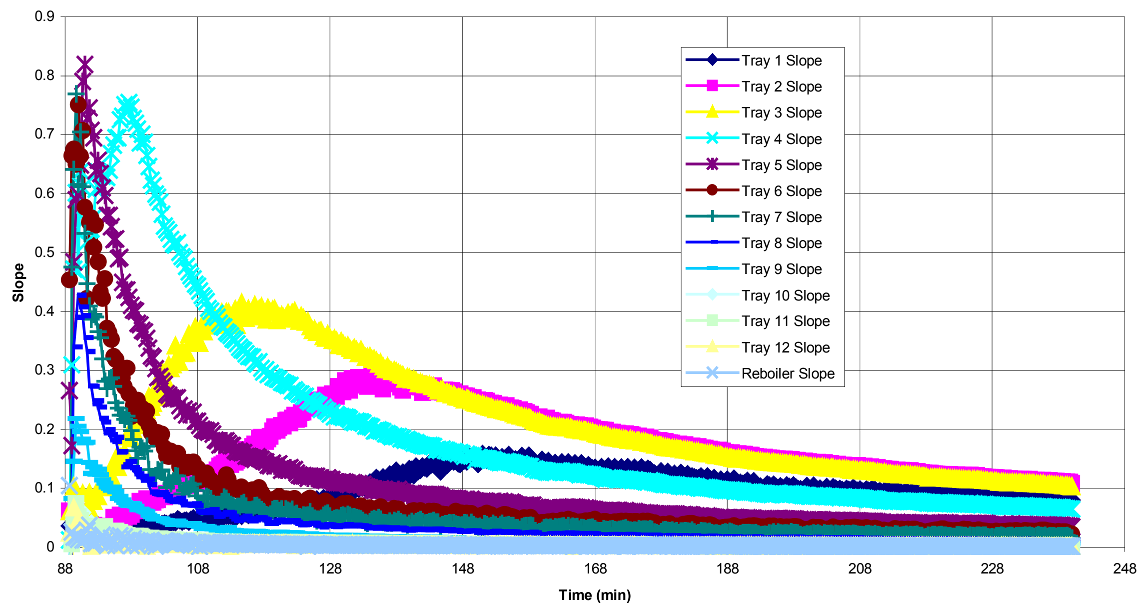

Figure 4.3. Slopes of tray temperatures after change

For some experiments, all the trays showed a rapid initial response, but the general trend showed the center of the column responding quickest. In addition to how quickly the maximum slope occurred, the center portion of the column, especially tray 7, also showed the largest slopes. An example of this from experiment System Identification 14 is shown in **Figure 4.3**.

4.2 System Identification: Column Response

There are two methods that were used to analyze the changes wrought on the operation of the column. One, the concentration front, serves as a useful tool for visualizing the overall effect of the change. The other, the McCabe-Thiele diagram, is useful for understanding what needs to be changed to compensate for the effect of the disturbance.

4.2.1 Concentration Fronts

The concentration front is a plot of composition versus tray, and is a good way to graphically summarize the performance of a distillation column. When a disturbance occurs in the operation of the column, plotting the concentration fronts of the column at important points in time—primarily the steady state portions before and after the disturbance—is a convenient way to summarize the changes that a disturbance has. **Figure 4.4** shows the concentration fronts for the two steady state portions of the experiment Feedback Control 2. In this experiment, the combined effect of the flow rate and reboiler heat changes served to shift the concentration front towards the top of the column.

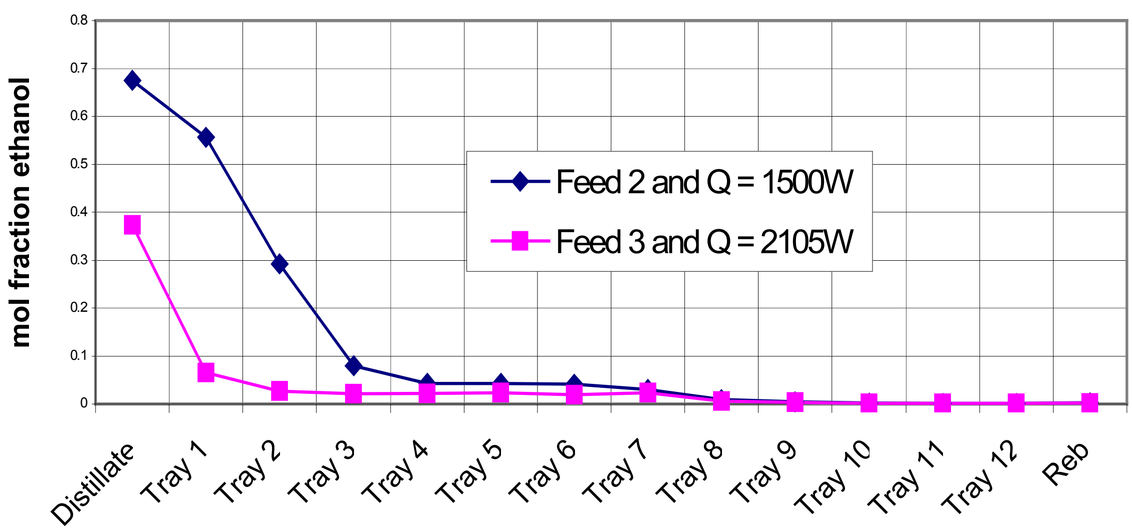

Figure 4.4. Concentration fronts, experiment Feedback Control 2

4.2.2 McCabe-Thiele Diagram[1]

At its most basic, the operation of a distillation column can be reduced to two main factors: the boil-up ratio and the reflux ratio. A good illustration of the effect boil-up and reflux ratios have on the behavior of the column can be had by examining the McCabe-Thiele diagram for the steady state portions of an experiment before and after a disturbance. Figure 4.5 displays the operating and q lines for both steady state sections of a representative experiment (Feedback Control 2, performed on 5/2/2009).

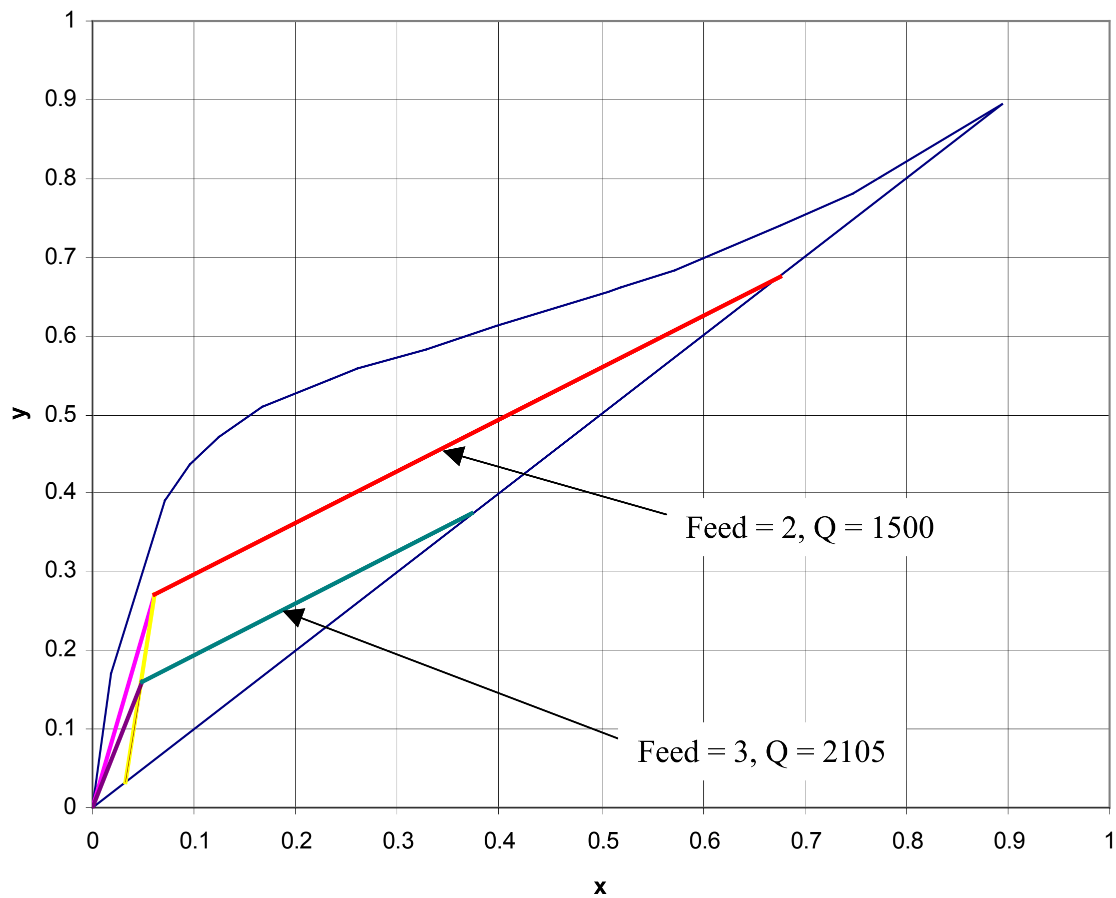

Figure 4.5. McCabe-Thiele operating lines, experiment Feedback Control 2

[1] All McCabe-Thiele equations use the notation presented in *Perry's Chemical Engineers' Handbook*.

The q line is determined by the composition and thermal condition of the feed, and has an equation of:

$$y = \frac{q}{q-1}x - \frac{x_F}{q-1}$$

Where $q = \frac{energy\ to\ convert\ 1 mol\ of\ feed\ to\ saturated\ vapor}{molar\ heat\ of\ vaporization}$

$$= \frac{\left(\left(x_F MW_E c_{p,E}\right) + \left((1-x_F)MW_W c_{p,W}\right)\right)\Delta T + x_F \Delta H_{vap,E} + (1-x_F)\Delta H_{vap,W}}{x_F \Delta H_{vap,E} + (1-x_F)\Delta H_{vap,W}}$$

x_F = mol fraction in the feed

MW_E = molecular weight of ethanol

$C_{p,E}$ = specific heat of ethanol

MW_W = molecular weight of water

$C_{p,W}$ = specific heat of water

$\Delta H_{vap,E}$ = molar heat of vaporization of ethanol

$\Delta H_{vap,W}$ = molar heat of vaporization of water

ΔT = temperature change to heat the feed to the boiling point

In all cases for these experiments, the feed entered the column as a sub cooled liquid, being at room temperature (which was reasonably consistent for the experiments and assumed to always be 22 ^{o}C.) The majority of experiments were run with a feed composition of 3.33 mol%. The following values can then be used to find q:

Table 4.2. Values for use in calculating q[2]

$\Delta H_{V,W}$	40.65	kJ/mol
$\Delta H_{V,E}$	38.6	kJ/mol
$c_{p,W}$	4.186	J/(g*K)
$c_{p,E}$	2.45	J/(g*K)
BP_F	93.05	^oC
ΔT	71.05	^oC
x_F	0.0333	

Substituting these into equation 3.2 gives a q value (for most experiments) of 1.135. This is then used to find the equation for the q line:

$$y = \frac{1.135}{1.135-1}x - \frac{0.0333}{1.135-1} = 8.407x - 0.247$$

The slopes and intercepts of the two operating lines are dependant on the operating conditions of the column. The equations can be determined by performing a mass balance on the top and bottom halves of the column, and have the following equations (the subscripts *n* and *m* are used to identify trays above and below the feed tray, respectively):

Top operating line: $y_n = \frac{L}{V}x_{n+1} + \frac{D}{V}x_D$

Where y_n = the vapor composition for on a given tray, n

L = the molar flow rate of the liquid in the top section

V = the molar flow rate of the vapor in the top section

x_{n+1} = the liquid composition from the tray just above tray n

D = the molar flow rate of the distillate

x_D = the composition of the distillate

Bottom operating line: $y_m = \frac{L'}{V'}x_{m+1} - \frac{B}{V'}x_B$

[2] Values from *CRC Handbook*.

Where y_m = the vapor composition for on a given tray, m

L' = the molar flow rate of the liquid in the bottom section

V' = the molar flow rate of the vapor in the bottom section

x_{m+1} = the liquid composition from the tray just above tray m

B = the molar flow rate of the bottoms product

x_B = the composition of the bottoms

It is not possible to determine many of the values in these equations, so simply plugging in the appropriate numbers and doing some minor arithmetic cannot determine the equations. However, the lines can be plotted if either two points or one point and the slope are known. It is only possible to determine one point for the bottom operating line from the available data: the bottoms composition, x_B. For the top operating line, one known point is the distillate composition, x_D. These two compositions are easily determined from the boiling points of the reboiler and tray 1, and the ethanol-water equilibrium data. These two points lie on the diagonal formed by the equilibrium data, so the y value is the same as the x value for both.

It is also possible to determine the slope of the top operating line, since the term $\frac{L}{V}$ (called the internal reflux ratio), can be determined from the external reflux ratio, R, using the following relationship:

$$\frac{L}{V} = \frac{R}{1+R} = \text{reflux percent in decimal form.}$$

As an illustration, and continuing to use the data from experiment Feedback Control 2, the following procedure shows the calculation of the operating lines for the steady state period before the step occurred.

The reflux percent is 66%, so the slope of the top operating line is: $\frac{L}{V} = 0.66$. It intersects the equilibrium diagonal at $y = x_D = 0.6754$. The intercept term in the top operating line equation can then be solved for:

$$\frac{D}{V}x_D = y_n - \frac{L}{V}x_{n+1}$$

$$\frac{D}{V}x_D = 0.6754 - 0.66 * 0.6754$$

$$\frac{D}{V}x_D = 0.2296$$

This gives the top operating line the equation of

$$y_n = 0.66x_{n+1} + 0.2296$$

The top operating line and the q line equations can be used to find the point of intersection between the two,

$$\frac{q}{q-1}x - \frac{x_F}{q-1} = \frac{L}{V}x + \frac{D}{V}x_D$$

$$8.407x - 0.247 = 0.66x + 0.2296$$

$$x = 0.0616$$

$$y = 0.2703$$

Since the two operating lines must intersect the q line at the same point, this also provides a second point on the bottom operating line, thus allowing the third equation to be determined. The slope is determined from the two known points:

$$\frac{L'}{V'} = \frac{y_2 - y_1}{x_2 - x_1}$$

$$\frac{L'}{V'} = \frac{0.2703 - 0}{0.0616 - 0}$$

$$\frac{L'}{V'} = 4.388$$

In this case the bottoms product is essentially pure water, so the intercept is 0, making the equation:

$$y_m = 4.388x_{m+1}$$

Following these same steps for the steady state conditions after the disturbance, the equations for the second set of three lines can also be found.

	Before change:	After change:
q line:	$y = 8.407x - 0.247$	$y = 8.407x - 0.247$
top op. line:	$y_n = 0.66x_{n+1} + 0.2296$	$y_n = 0.66x_{n+1} + 0.1268$
bottom op. line:	$y_m = 4.388x_{m+1}$	$y_m = 3.293x_{m+1}$

In the normal use for a McCabe-Thiele diagram, these equations can then be used to plot the concentration data for the individual trays (see **Figure 5.3**, page 82 for an example). Since the focus is the overall effect on the column, this will be omitted. Once the operating line equations are understood, they can be used to illustrate the effect of disturbances on the column and understand what steps should be taken to account for them (in the case of a change in feed flow rate or feed composition), or how they can be used to counter the disturbance (in the case of a change in reflux percent or reboiler heat.)

4.2.3 A Change in Reflux Percent

The experiment illustrated in Figure 4.6 and Figure 4.7, System Identification 5, involved decreasing the reflux percent heat from 75% to 70% while leaving the other parameters unchanged.

The direct result of a change in reflux percent is a change in the magnitude of the flow rate of liquid down the column. A relatively small change in the reflux percent can have a relatively large effect on the concentration front; the 5% change in reflux percent in this experiment shifted it to such an extent that instead of the top 4 trays in the column having azeotrope, only tray 1 did after the change.

The change in reflux percent is directly represented in the McCabe-Thiele operating lines as a change in the slope of the top operating line. A decrease in reflux percent also decreases the slope of the operating line, shifting it more towards horizontal. An increase in reflux percent moves the operating line closer to the diagonal.

The gain and time constant values for tray 1, tray 7 and the reboiler are summarized below.

Table 4.3. Control data for System Identification 5

	Gain	Time Constant
Tray 1	-0.121914927	143.66
Tray 7	-1.111331503	28
Reboiler	-0.013557488	55.33
	^{O}C/reflux %	minutes

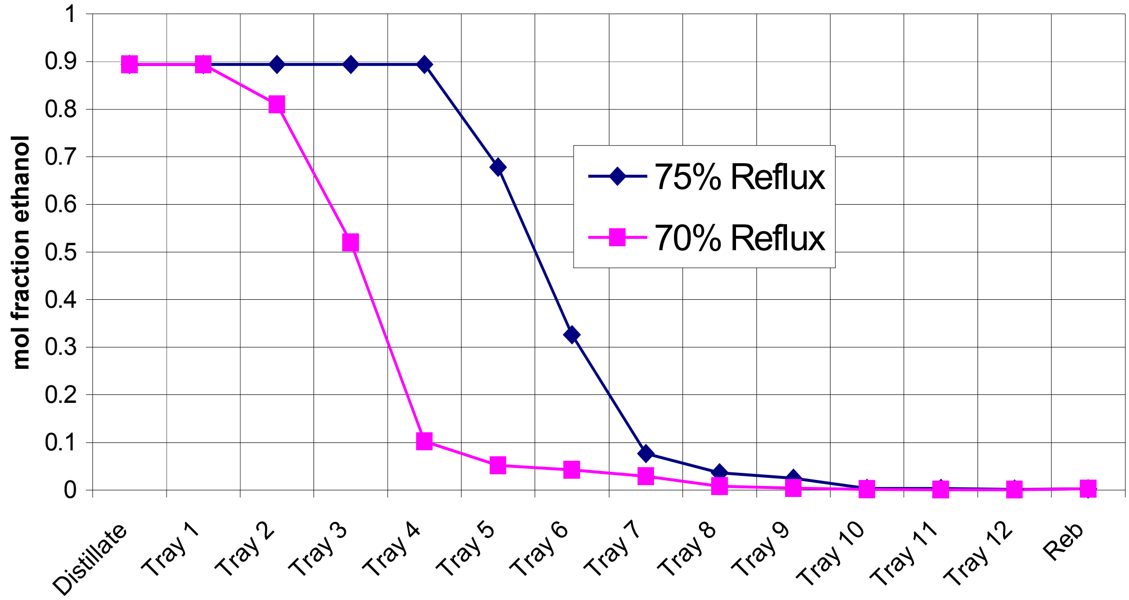

Figure 4.6. Concentration fronts, change in reflux percent

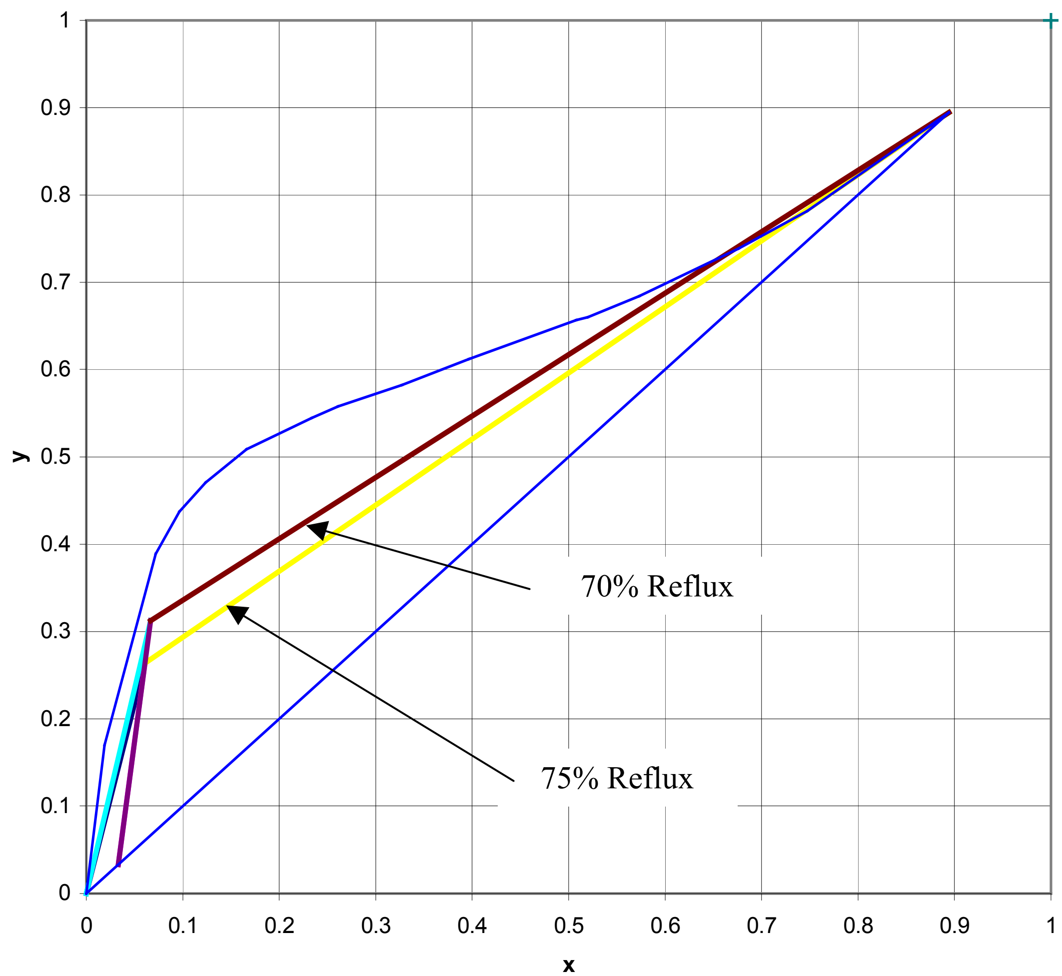

Figure 4.7. McCabe-Thiele operating lines, change in reflux percent

4.2.4 A Change in Reboiler Heat Input

The experiment illustrated in Figure 4.8 and Figure 4.9, System Identification 14, involved increasing the reboiler heat from 1500W to 1660W while leaving the other parameters unchanged.

A change in reboiler heat acts in many ways as a change in reflux percent does, just on the bottom of the column instead of the top. Increasing heat increases the boil-up percent, while decreasing heat decreases the boil-up percent. The heat increase of slightly more than 10% resulted in a significant reduction in distillate purity: tray 1 dropped from the azeotrope to less than 7 mol%. Analyzing the change on the McCabe-Thiele diagram can best give an explanation of this effect. Because of the relatively low purity of the feed, the q line and the bottom operating line are very close. This means that any change in the slope of the bottom operating line can have a major change on the point where the bottom operating line and the q line intersect. Since the top and bottom operating lines both intersect the q line at the same point, this will consequently also have a major effect on the final composition of the distillate.

The gain and time constant values for tray 1, tray 7 and the reboiler are summarized below.

Table 4.4. Control data for System Identification 14

	Gain	Time Constant
Tray 1	0.0774	55.33
Tray 7	0.0193	4.66
Reboiler	0.0017	7.66
	^{O}C/W	minutes

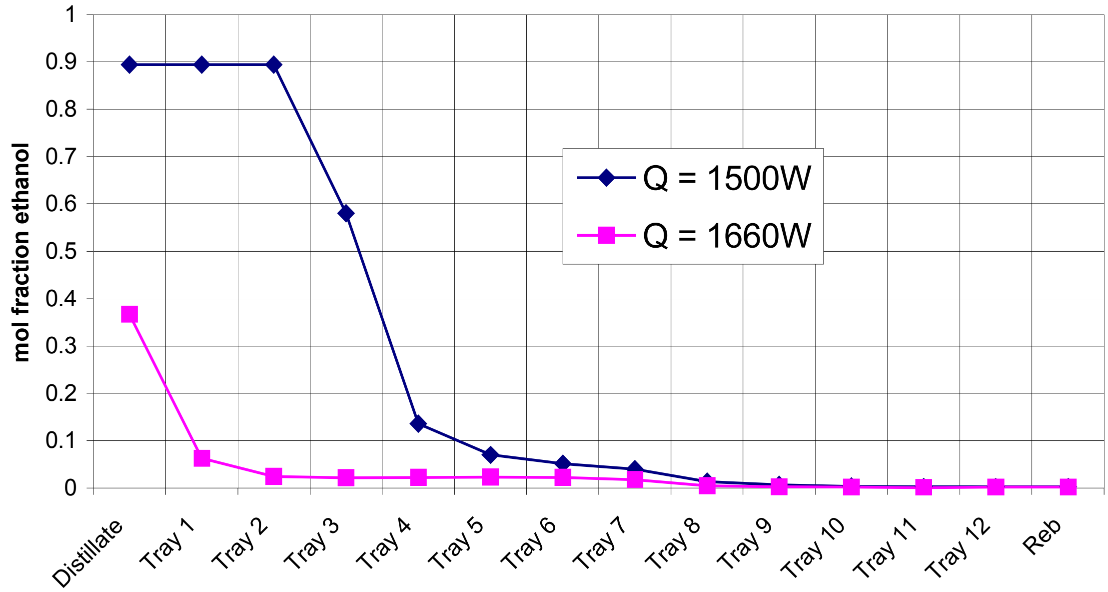

Figure 4.8. Concentration fronts, change in reboiler heat

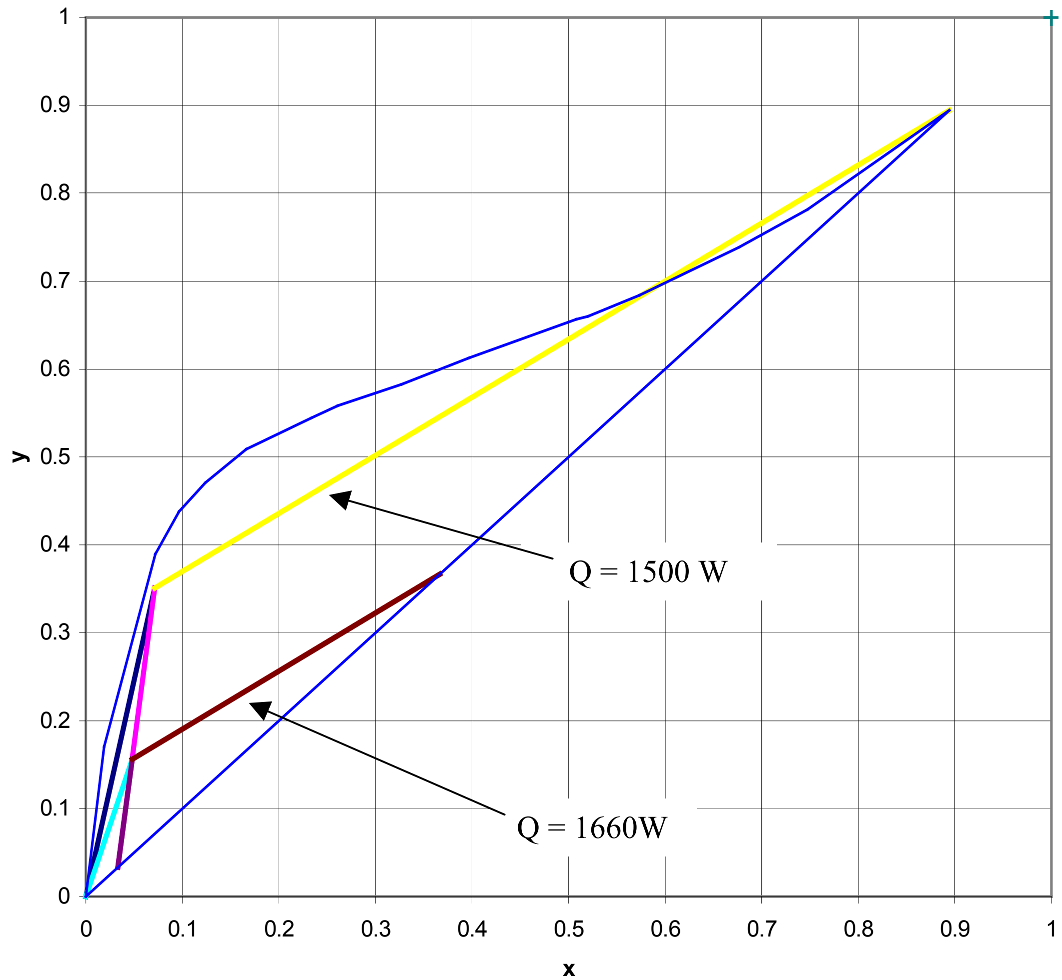

Figure 4.9. McCabe-Thiele operating lines, change in reboiler heat

4.2.5 A Change in Feed Flow Rate

The experiment illustrated in Figure 4.10 and Figure 4.11, System Identification 9, involved increasing the feed flow rate from pump setting 2 to pump setting 3 while leaving the other parameters unchanged.

The increased flow of feed entering the column shifted the concentration front down the column, resulting in higher concentrations in the top of the column while also slightly increasing the reboiler concentration, thus resulting in more ethanol leaving the column in the bottoms stream.

Since the structure of the operating lines on the McCabe-Thiele diagram depends on the ratios of the vapor and liquid flow rates in the column, a change in the feed flow rate won't necessarily have a significant effect on them. This is the case for this experiment; the increased feed has only a minimal effect on the slope of the bottom operating line (increasing it from 4.82 to 4.93), which only slightly changes the location of the top operating line.

The gain and time constant values for tray 1, tray 7 and the reboiler are summarized below.

Table 4.5. Control data for System Identification 9

	Gain	Time Constant
Tray 1	-0.318628565	1.33
Tray 7	-7.39950144	2.67
Reboiler	-3.493470188	66.67
	^{O}C/pump step	minutes

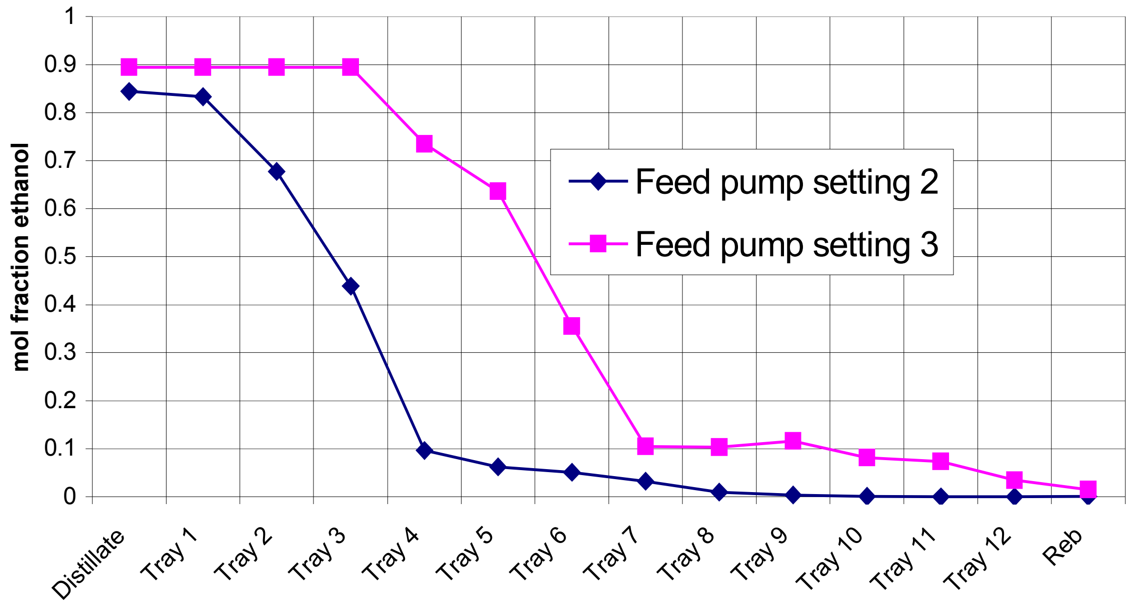

Figure 4.10. Concentration fronts, change in feed flow rate

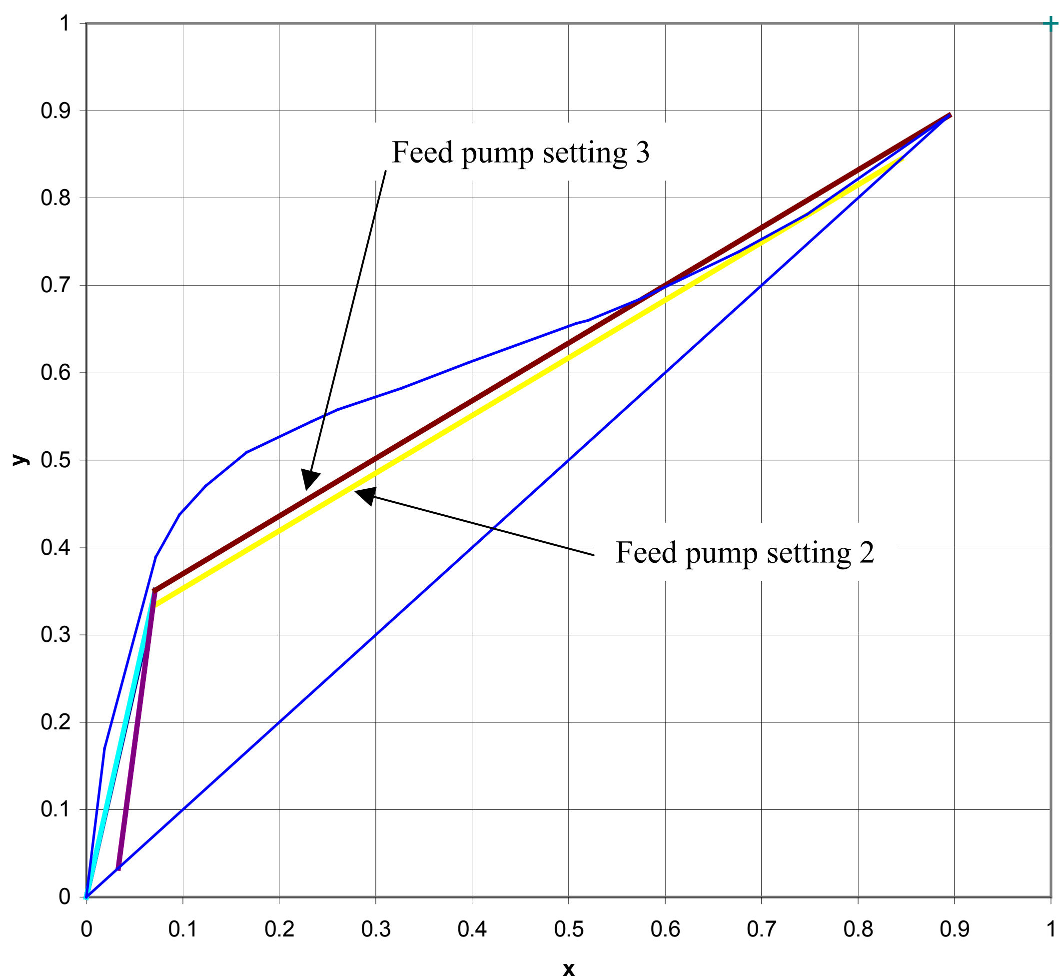

Figure 4.11. McCabe-Thiele operating lines, change in feed flow rate

4.2.6 A Change in Feed Composition

The experiment illustrated in Figure 4.12 and Figure 4.13, System Identification 13, involved decreasing the feed flow composition from 3.33 mol% to 2 mol% while leaving the other parameters unchanged.

The appearance of both the concentration front chart and the McCabe-Thiele chart for the change in feed concentration appear very similar to those from the experiment where the reboiler heat was changed. The concentration front shifted to the left and resulted in a significant drop in the distillate concentration.

The change in feed composition is the only one of the four factors that has changed the q line. While the lowered concentration does change the value of q slightly, and therefore the slope of the q line, the most significant effect comes from the change in where the q line intersects the diagonal. The q line moved closer to the bottom operating line, meaning that they intersect at a lower point, therefore also reducing the intersection of the top operating line and the distillation composition.

The gain and time constant values for tray 1, tray 7 and the reboiler are summarized below.

Table 4.6. Control data for System Identification 13

	Gain	Time Constant
Tray 1	-8.54875	79.67
Tray 7	-2.23006	6.34
Reboiler	-0.16022	19.34
	^{O}C/mol%	minutes

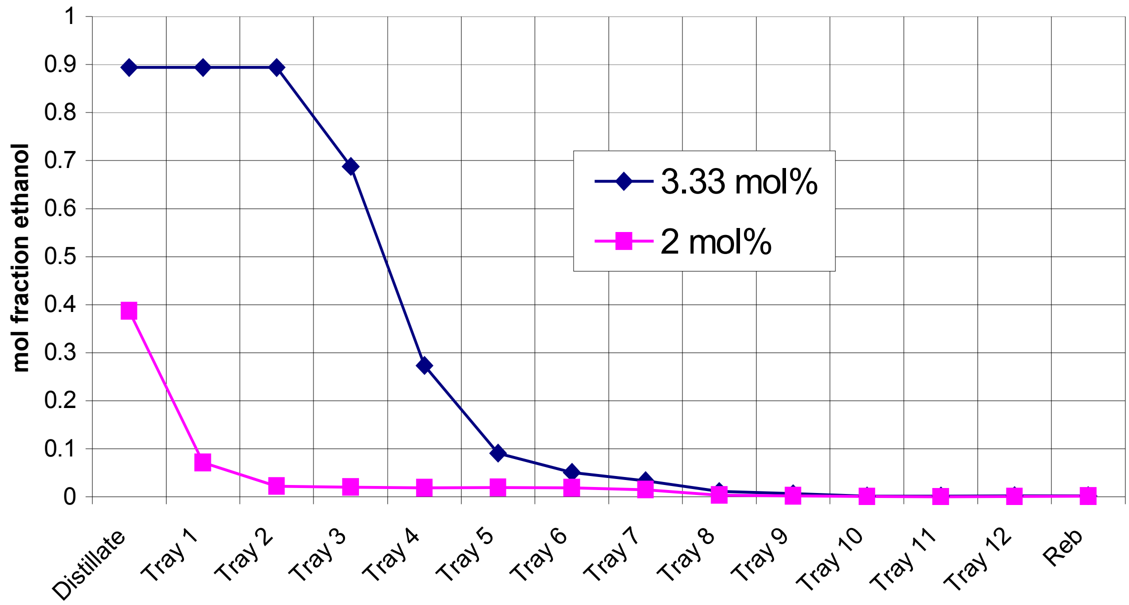

Figure 4.12. Concentration fronts, change in feed composition

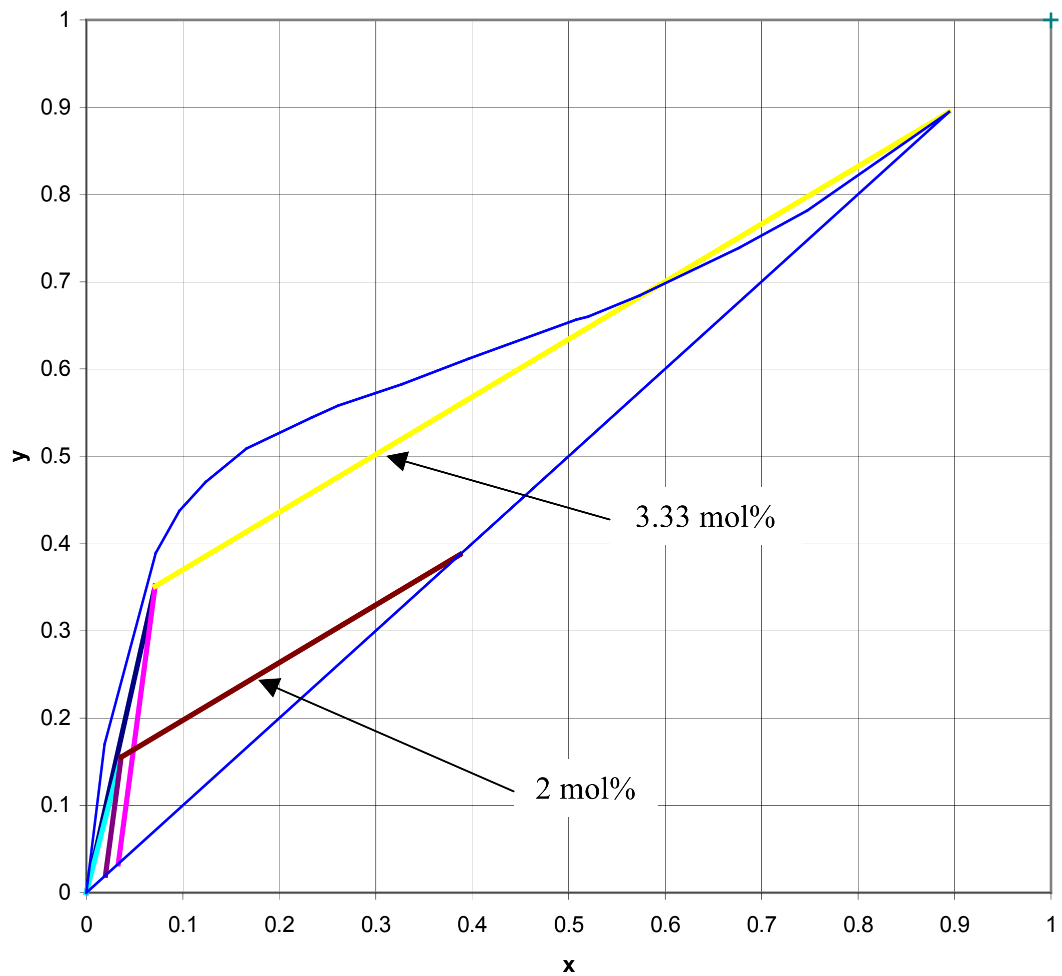

Figure 4.13. McCabe-Thiele operating lines, change in feed composition

4.2.7 System Identification Summary

While the understanding of the effect a disturbance in each parameter can have is useful, it is important to note that these experiments cannot be extrapolated to allow understanding of similar changes with different column operating conditions.

4.3 Feedback Control

The three experiments where manual feed back control was attempted all focused on solely manipulating the reboiler heat in response to an increase in feed flow rate. Anincrease in the amount of feed entering the column proved to be the most complex disturbance to respond to. A change in feed composition results in a distinct change in the McCabe-Thiele diagram, while the feed flow rate change altered the McCabe-Thiele little while having a significant affect on the concentration front.

If the primary goal of the experiment is purity of the distillate, little needs to be done in response to an increase in feed flow rate, because the standard starting conditions for most of the experiments resulted in distillate product that was already as pure as could be achieved using the distillation column. However, with no response to the disturbance, the increased flow results in more of the ethanol leaving the column in the bottoms product. In a situation where recovery of the ethanol is important, this is obviously not desirable and needs to be corrected.

4.3.1 Feedback Control 1

Experiment Feedback Control 1 was performed in response to the results of experiment System Identification 12. That experiment attempted to correct for the increased feed flow by increasing the reboiler heat by 800 watts, an amount calculated to increase the temperature of the additional material from room temperature to the column conditions. The result of the increased heat was a significant reduction in purity in the top half of the column (Figure 4.14) while the lower half remained steady.

A manual feedback control scheme was devised to attempt to maintain tray 1 at the same point after the disturbance as before. Feedback Control 1 would follow the same procedure as System Identification 12 until the point where the temperature of tray 1 began to rise after the disturbance. When tray 1 had risen by one degree, the reboiler heat would be lowered by 10W per degree of increase per minute until the difference was again within one degree. The reboiler heat dropped to 1660W before the temperature of tray 1 had dropped back to within one degree of its initial value. The end result was a distillate purity that was still high, but the concentration front had shifted well to the right, meaning that the bottoms product contained ethanol.

4.3.2 Feedback Control 2 and 3

Two further feedback control experiments were performed to analyze the effect of control schemes in which the reboiler temperature was monitored instead of the tray 1 temperature. The procedure of these experiments differed slightly from the previous one. At the time of the increase in the feed flow rate, the reboiler heat was not increased. When the temperature of the reboiler had decreased by 0.1 degree, the control scheme was implemented. The reboiler heat was increased by 1W per 0.1 degree per minute. This was continued until the reboiler was again within 0.1 degree of its starting value. The only difference between the two experiments was the reflux percent.

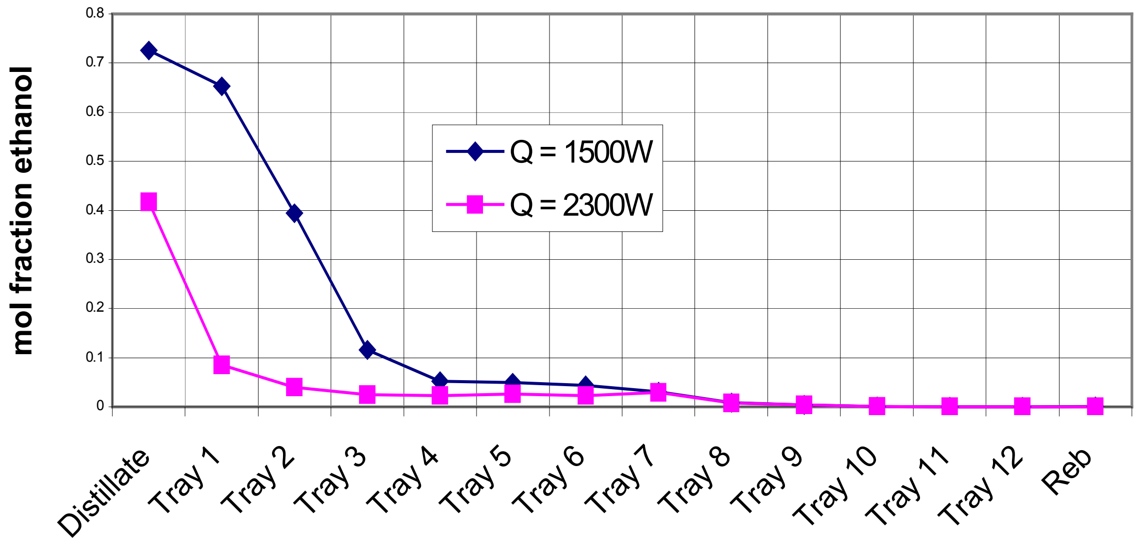

Figure 4.14. Concentration fronts, System Identification 12

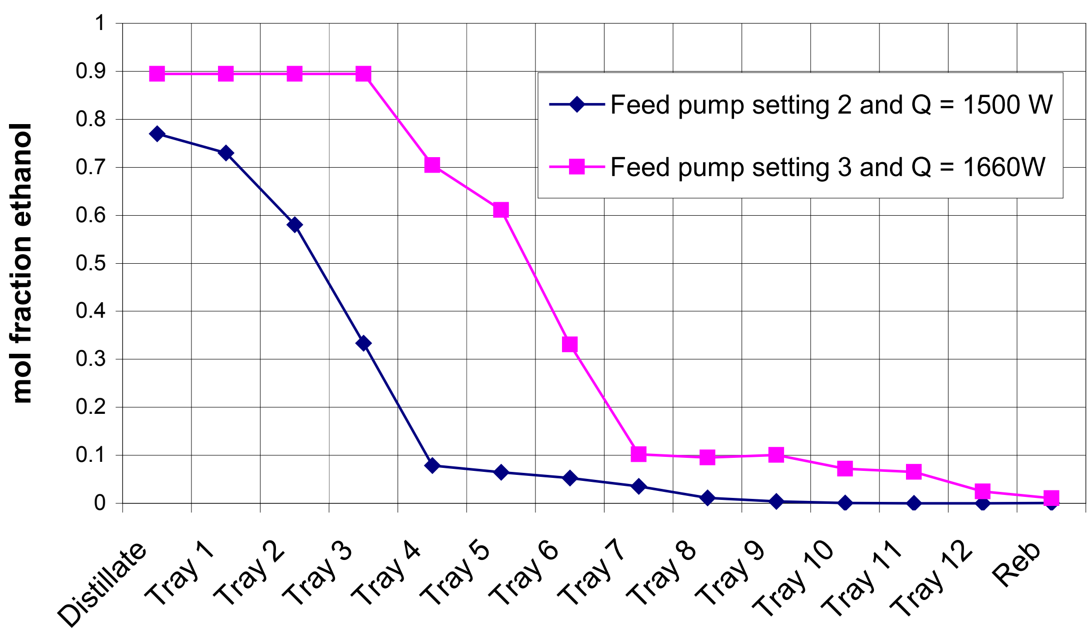

Figure 4.15. Concentration fronts, Feedback Control 1

In both cases, maintaining the reboiler temperature resulted in a significant decrease in the purity of the distillate, as the concentration fronts were shifted to the left (Figure 4.16 and Figure 4.17.)

4.3.3 Feedback Control Summary

The feedback control experiments proved that a change in a single parameter is not sufficient to compensate for the feed flow rate disturbance. It is impossible to maintain both product compositions after a disturbance while manipulating only one variable. The understanding of the general behavior of the column in response to disturbances gained from the system identification experiments needs to be used to plan a combined change in both reflux percent and reboiler heat.

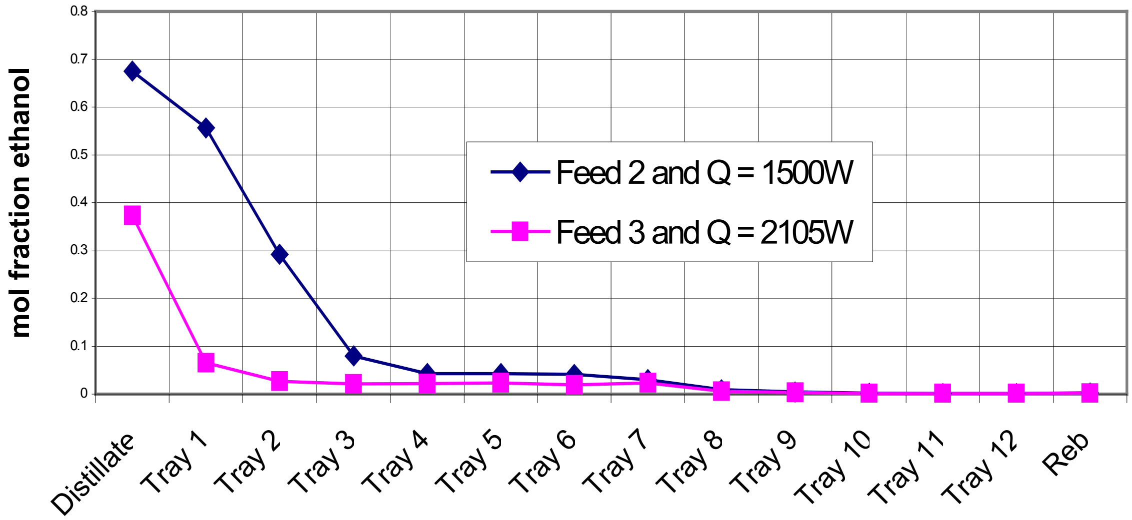

Figure 4.16. Concentration fronts, Feedback Control 2

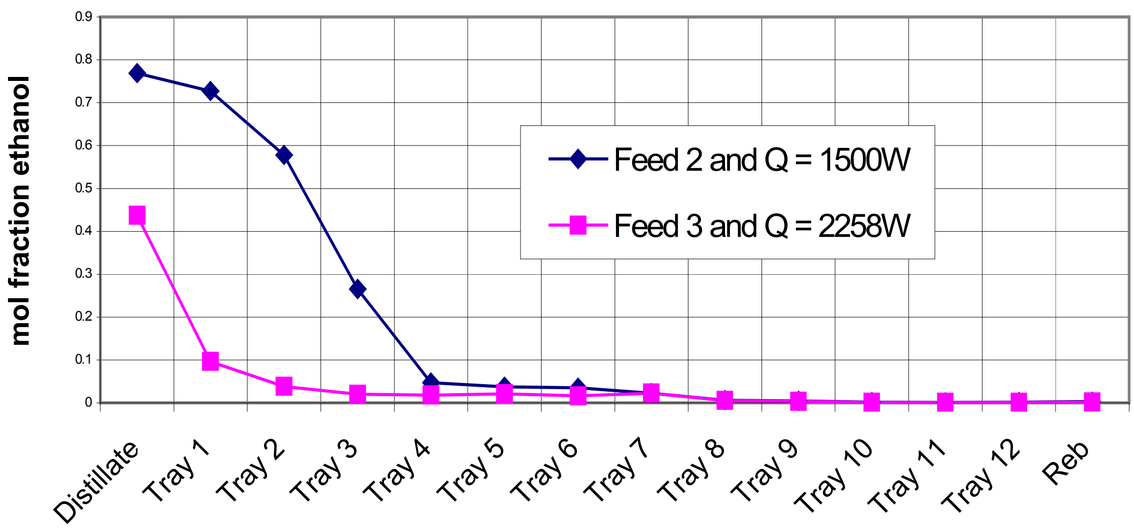

Figure 4.17. Concentration fronts, Feedback Control 3

5 Conclusions

5.1 Column Operation

The early experiments served to show that attempting multiple step changes in one experiment was unfeasible because of the long time constants involved in the dynamic periods. Because of this and because of the need for repeatability in the experiments, it was necessary to develop a standard procedure. The experiments that had already been performed at that point were analyzed to determine how much time would be required for the steps to reach steady state. The majority of the experiments had response times (τ) of 25 minutes or less, meaning that 150 minutes ($6 * \tau$) would be sufficient in most circumstances. The following procedure was then settled on:

The experiments begin with the following conditions:

- the reboiler heater at 3200 W
- the feed pump off
- reflux set to 100%

The column is observed until the temperature in tray 1 begins to rise, once this happens, the operating conditions are changed as follows:

- the reboiler heat is set to 1500W
- the feed pump was is to a speed setting of 2
- reflux is changed to 66%

At this point, the column is allowed to operate until 150 minutes had elapsed in the experiment. In most cases this is sufficient to reach steady state; in those case where it is not, the experiment can be allowed to run longer. Once at steady state, the column parameters are then changed as desired for the experiment.

While this procedure proved to be reliable for the experiments performed for this report, the operating conditions desired for an experiment could have a significant effect on the time required for reaching steady state. As System Identification 6 and 7 showed (Figure 3.7), this is especially true for experiments where ethanol is being removed from the column. It is advisable to determine how much time would be necessary to reach steady state for a given set of operating conditions, and then develop and adhere to a standard procedure to minimize wasted time and effort, and to maximize the chances of obtaining usable data from an experiment.

5.2 Challenges encountered

Numerous questions and challenges arose during the analysis of the distillation column. Most of these issues were dealt with, but some still need to be evaluated and should be investigated further.

5.2.1 Accuracy of the temperature data

During the evaluation of the data, a question arose as to the validity of the control data because of the response of the RTD (resistive thermal device) probes measuring the column temperatures. An evaluation of the temperatures recorded during one of the 66% reflux experiments shows that the RTD response times are fast enough to show the change in temperature that occurs because of the changing liquid flow down the column when the reflux switches from the column to the distillate. The chart below (Figure 5.1) shows a small sample of one of the 66% reflux runs. As each minute begins, the reflux valve directs the condensed liquid back down the column. This continues for 0.66

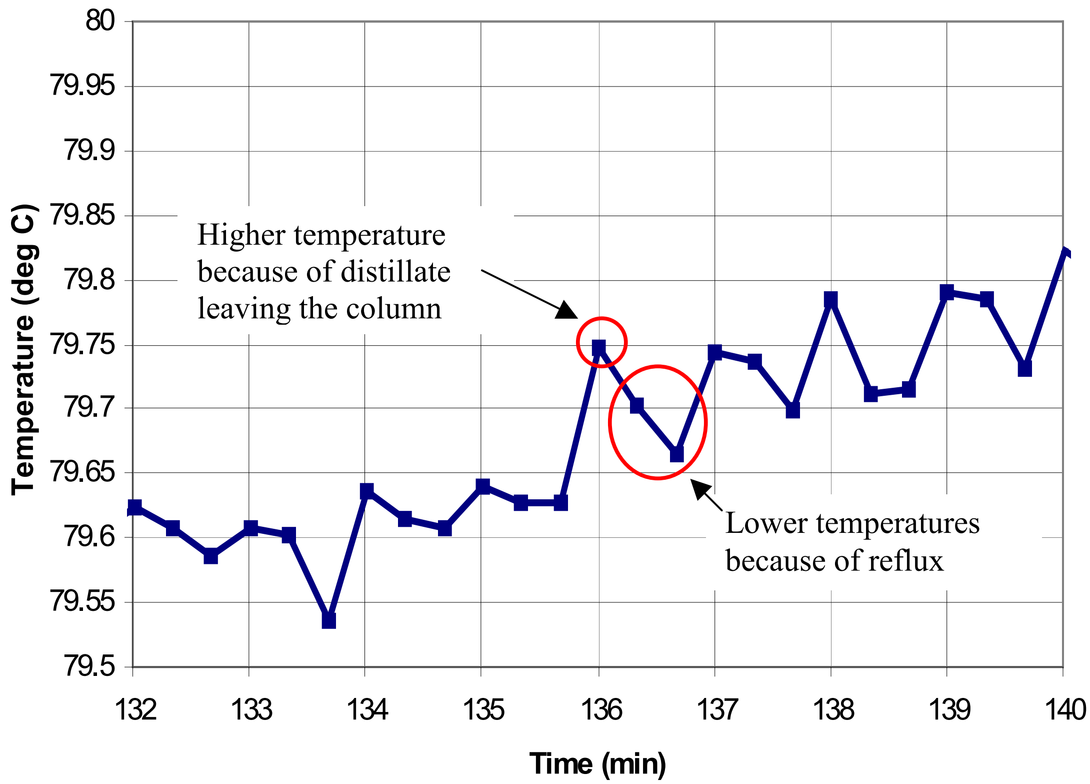

Figure 5.1. Temperature probe response

minutes, at which time the valve switches the flow to a tube that leaves the column and eventually drains to the distillate receiver. The first 0.66 minutes result in cooler temperatures as the condensed liquid absorbs some of the heat in the column; the temperatures recorded at 20 seconds and 40 seconds show a general downward trend because of this. The last 0.34 minutes result in higher temperatures, which is shown by the temperatures recorded at each minute.

5.2.2 Mass Balance Problems

During the analysis of the flow rate data, a mass balance of the ethanol and water in the column was performed. A disturbing problem was revealed by this data: there is a significant amount of unaccounted for material (Figure 5.2.) In all cases, the vast

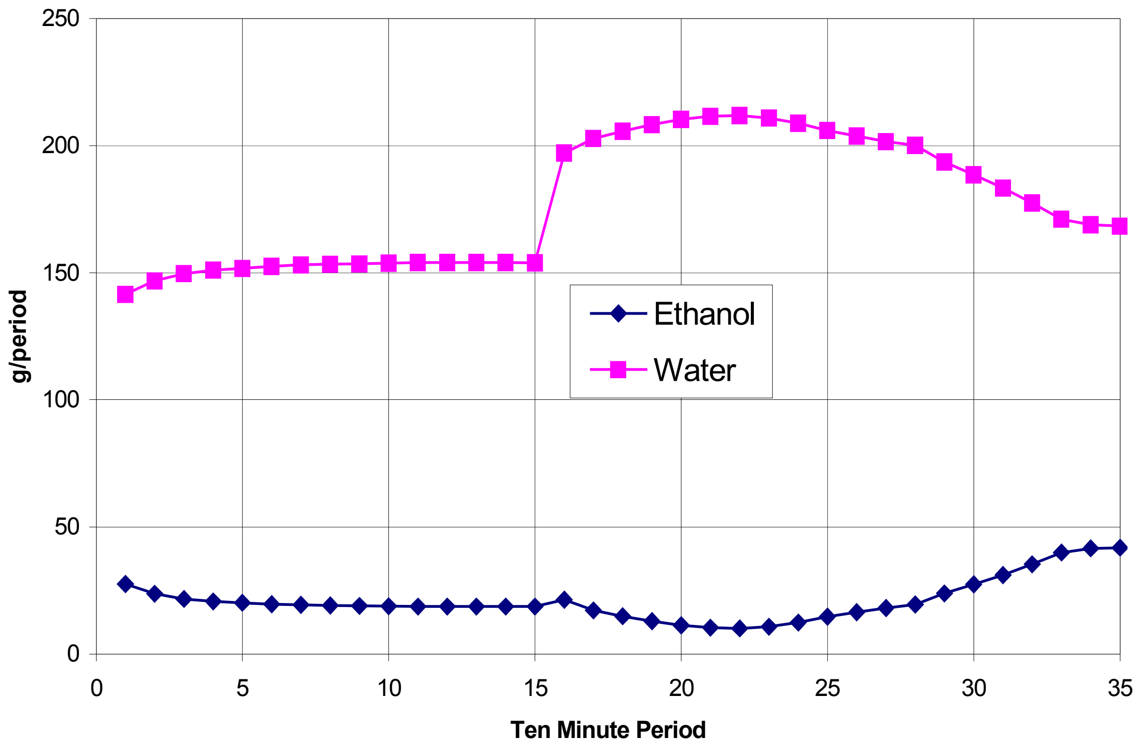

Figure 5.2. Unaccounted for material in mass balance

majority of this unaccounted for material is water, averaging from 2-4 grams of ethanol per minute and 10-20 grams of water per minute. Two primary factors could explain this effect:

5.2.2.1 One or more of the pump flow rates are inaccurate

An error in any of the flow rates could have a significant effect on the mass balance. The flow rates (shown in Appendix A2) were checked on three occasions, and seemed to be consistent each time. However, the last time they were checked the distillate pump flow rate for the 20-second cycle was almost 25% lower than was observed previously (20.4 mL vs. 27.6 mL.) This is troubling and calls into question the reliability of the pump flow rate data.

Because the missing material is mostly water, the most likely pump to have the noticed effect on the mass balance is the reboiler pump. A problem with its flow rate was never noted, but the change in the distillate flow rate raises some doubts. A more thorough analysis of the pumps would be advisable.

5.2.2.2 There are one or more leaks in the column

On observation of the column during several of the runs, small leaks were noted at several points in the column. These included:

1. the joint between the main body of the reboiler and the module housing the resistance heaters
2. the joint between the reboiler and the bottom section of the column
3. the joint between the bottom section of the column and the feed section

None of these leaks appeared very large, but there was no way to capture the liquid in order to accurately measure it, nor to account for the rapid evaporation of the liquid because of the high temperatures involved. The leak at the joint between the reboiler and the column is an especially problematic one. The leak was significant enough to leave a trail down the side of the reboiler, but the heat from the reboiler caused it to completely evaporate before it was able to make it all the way down.

An additional leak was eventually discovered in the reboiler pump. There is some physical defect in the pump head that allows liquid to drain past it when the pump is not running. It does not always occur, so it’s effect on the data before the problem was discovered is unknown. The problem exists because the pump and the drain it flows to are lower than the reboiler. Raising the drain slightly and adding a T-fitting to the line at the highest point to create a siphon break solved the issue.

5.2.3 Tray Efficiency

Analyzing the performance of the column using the McCabe-Thiele method proved to be problematic. On many occasions, the steps plotted to show the effect of the stages showed tray efficiencies over 100% (represented by the steps moving above the equilibrium line in places, see **Figure 5.3**.)

As this is impossible, questions arose as to the accuracy of the work being done. Further research into the method revealed the cause: one of the assumptions McCabe-Thiele makes about the column is that it is adiabatic. This is obviously unrealistic in real life, and is especially inaccurate for the UTC column. While the double walled construction of the column sections does eliminated much of the heat loss on the trays, the joints between the column sections do not benefit from the same construction, as they are solid glass. Observation of the column during operation revealed that the joints had surface temperatures ranging between 60^{o}C and 70^{o}C (**Figure 5.4**.) Assuming a convection heat transfer coefficient of $50\frac{W}{m^2K}$ (roughly the middle of the 10-100 $\frac{W}{m^2K}$ range for air), the four joints for the column give an approximate heat transfer of

$$q = hA\Delta T$$
$$q = 4*(50\frac{W}{m^2K})(2in*14in*\frac{0.00064516m^2}{1in^2})(65^OC - 22^OC)$$
$$q = 155W$$

This is a significant source of heat loss (approximately 10% of the standard heat input of 1500W) and results in internal reflux, the condensation of some of the vapor within the column. This effectively increases the separation efficiency of the column. This may seem desirable, but it comes at the cost of lower throughput for the column and increased energy inefficiency. It is also an uncontrollable characteristic of the column and cannot

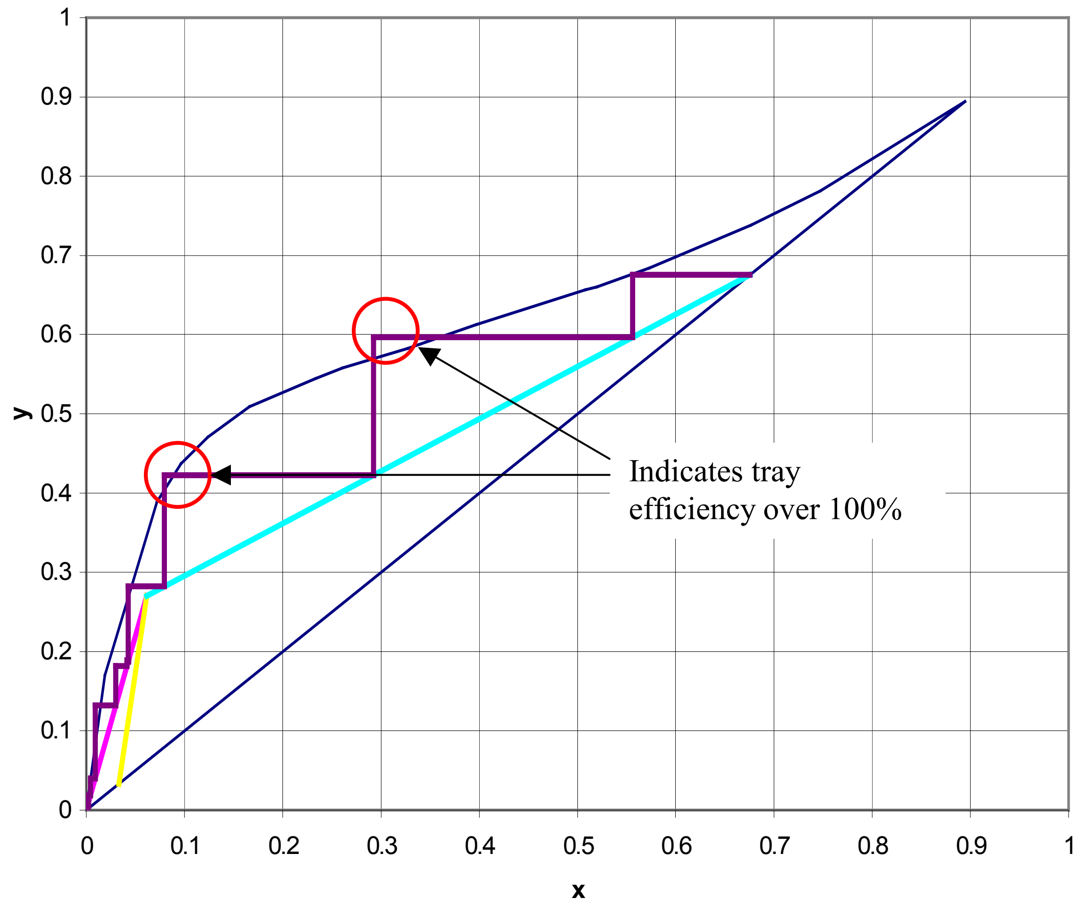

Figure 5.3. McCabe-Thiele diagram, including steps representing tray performance

be eliminated without insulating the column joints. Since the column serves primarily as a teaching tool, and observation of the column internals is an important factor in its use, anything that would impede view of the column is not desirable; any insulation on the column could only cover the small sections of the joints and must not lap over the rest of the column.

5.3 Next Steps

The analysis performed for this report is just a first step towards developing a feedback control system for the UTC distillation column; further analysis is needed.

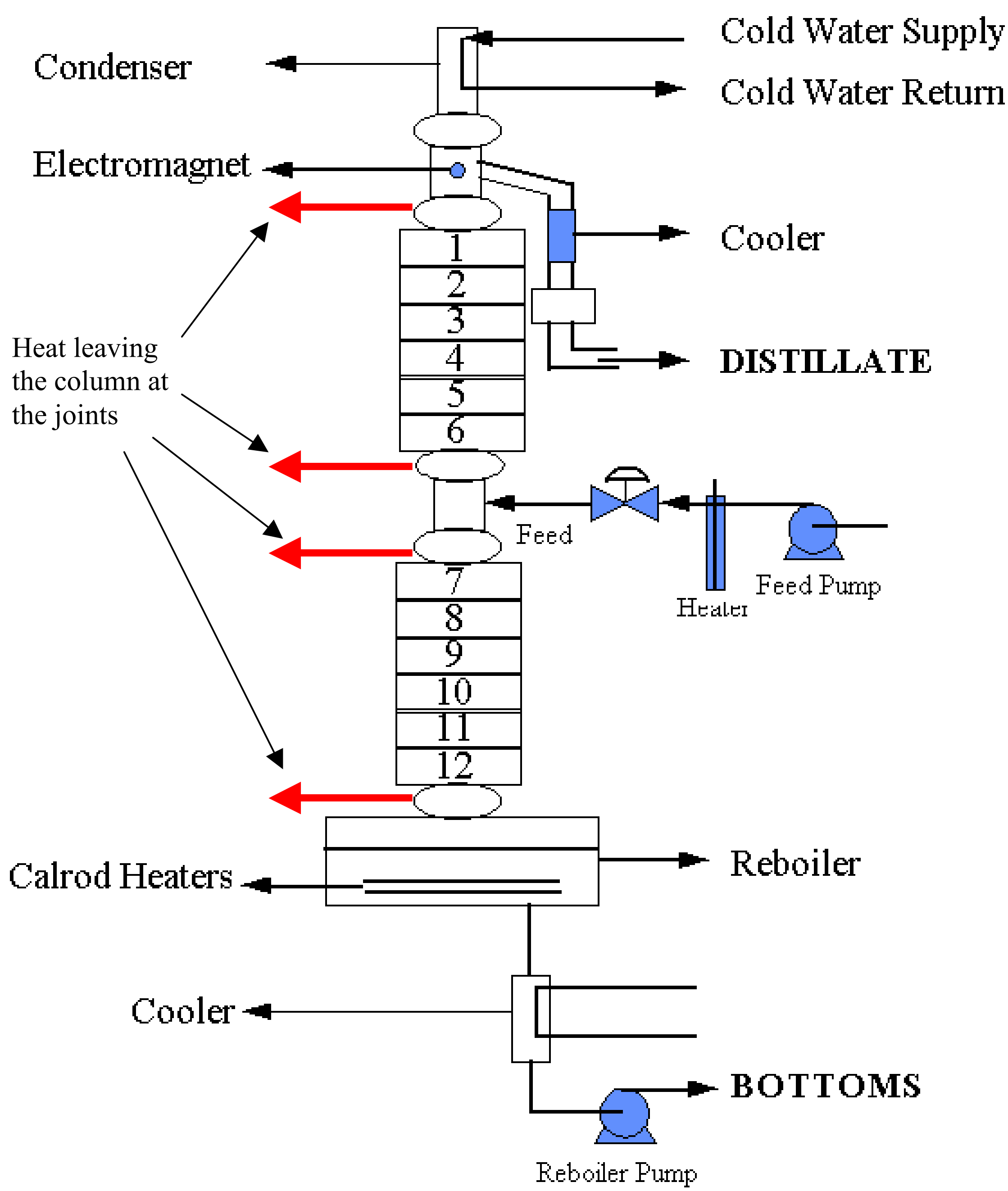

Figure 5.4. Column diagram showing heat loss

5.3.1 More Experiments

Further experiments, especially ones focusing on changes in reflux percent, should be performed to increase understanding of the column. The feedback control experiments were especially useful for this, as they provided evidence of how the column responds to adaptive change.

5.3.2 Fix the Leaks

The leaks in the column are troublesome as they cannot be quantified, and their effect on the column can therefore not be estimated. Finding a way to fix the leaks should be a high priority, as it will probably help improve consistency in column operation.

5.3.3 Resolve Flowrate Problem

The inconsistent flow rates measured for the distillate pump call into question the flow rates for the reboiler and feed pumps, also. If the difference was the result of a one-time change in the structure of the flow path, it is not that significant (but still problematic). However, if it is caused by some other factor, or is representative of a regular variation, then the method used for measuring the pump flow rates is not sufficient and may require reconsidering. Long-term evaluation of the pumps is needed to see if there is any further evidence of variation in the flow rates.

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APPENDIX

A1. Ethanol/Water Vapor-Liquid Equilibrium Data

Table A1.1. Ethanol/Water T, x, y Data

T (deg C)	x	y
100	0	0
95.5	0.019	0.17
89	0.0721	0.3891
86.7	0.0966	0.4375
85.3	0.1238	0.4704
84.1	0.1661	0.5089
82.7	0.2337	0.5445
82.3	0.2608	0.558
81.5	0.3273	0.5826
80.7	0.3965	0.6122
79.8	0.5079	0.6564
79.7	0.5198	0.6599
79.3	0.5732	0.6841
78.74	0.6763	0.7385
78.41	0.7472	0.7815
78.15	0.8943	0.8943

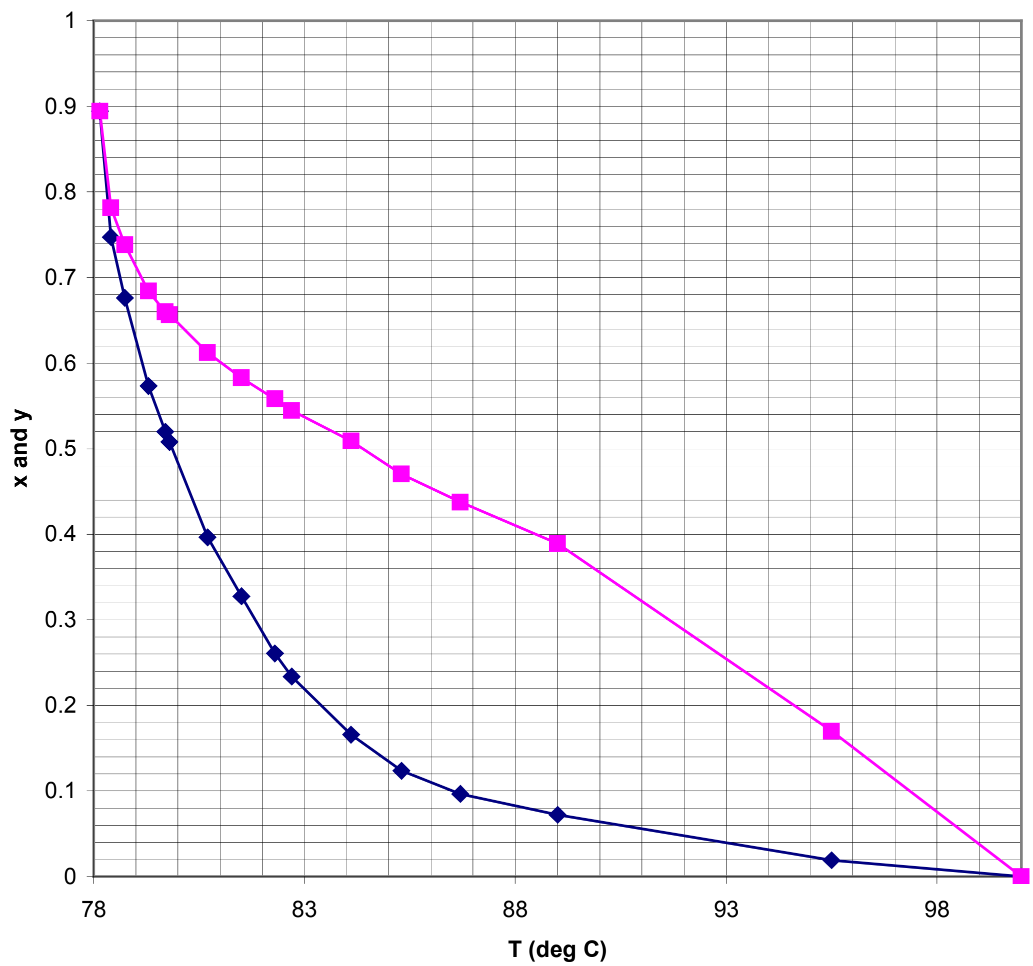

Figure A1.1. Ethanol/Water T, x, y Chart

A2. Pump flow rates

Table A2.1. Feed pump flow rates

Feed Pump		
Setting 2	Setting 3	Setting 4
79.4	138.9	207
mL/min	mL/min	mL/min

Table A2.2. Reboiler pump flow rates

Reboiler Pump					
Setting 2*			Setting 3*		
20 Sec. Cycle	40 Sec. Cycle	60 Sec. Cycle	20 Sec. Cycle	40 Sec. Cycle	60 Sec. Cycle
99.2	339	600	165.6	543.3	990
mL/cycle	mL/cycle	mL/cycle	mL/cycle	mL/cycle	mL/cycle

*The speed of the reboiler pump varies with the speed of the feed pump. When the proximity sensor that monitors the reboiler level triggers the pump, it comes on for 20 seconds at a setting equal to the feed pump. If the sensor it still being triggered at the end of 20 seconds, the pump setting increases to twice the feed pump setting for the next 20 seconds. If the sensor is still active, the reboiler pump increases to three times the feed pump for a further 20 seconds. A pump "cycle" measures the volume pumped for the entire period the pump is on.

Table A2.3. Distillate pump flow rates

Distillate Pump	
27.6	20.1
mL/min	mL/min

The flow rate measured from the distillate pump yielded two different results at two different times. This is discussed in section 5.2.2.1, page 79.

VITA

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