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FEEDFORWARD CONTROL OF A BINARY

DISTILLATION COLUMN USING THE FEED TRAY

LOCATION AS THE MANIPULATIVE VARIABLE

by

Emmett J. Speicher

A Research Report

Presented to the Graduate Faculty

of Lehigh University

in Candidacy for the Degree of

Master of Science

in

Chemical Engineering

Lehigh University

1969

CERTIFICATE OF APPROVAL

This research report is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering.

Nov. 25, 1969
(Date)

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Professor in Charge

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Engineering

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NOMENCLATURE

B : bottoms rate (lbmoles/hr)

CP_M : specific heat of methanol (Btu/lbmole-°C)

CP_{SOL} : specific heat of the solution (Btu/lbmole-°C)

CP_W : specific heat of water (Btu/lbmole-°C)

D : distillate rate (lbmoles/hr)

d_S : density of steam (grams/cm³)

$d_{SOL(T)}$: density of the solution at temperature T
      (grams/cm³)

$d_{W(T)}$: density of water at temperature T (grams/cm³)

E_{24} : energy of vapor stream leaving the top of the column
      (Btu/hr)

E_B : energy of bottoms stream (Btu/hr)

E_F : energy of feed stream (Btu/hr)

E_R : energy of reflux stream (Btu/hr)

E_S : energy of incoming steam (Btu/hr)

F : feed rate (lbmoles/hr)

FT-1 : spare feed tank

FT-2 : spare feed tank

h_B : enthalpy of the bottoms stream (Btu/lbmole)

h_{BSAT} : enthalpy of saturated bottoms of composition X_B
      (Btu/lbmole)

h_F : enthalpy of the feed stream (Btu/lbmole)

h_{FSAT} : enthalpy of saturated feed of composition X_F
      (Btu/lbmole)

H_i (i=1,2,...24) : enthalpy of vapor leaving the ith tray
            (Btu/lbmole)

H_{iSAT} (i=1,2,...24) : enthalpy of saturated vapor leaving
            the ith tray (Btu/lbmole)

h_R : enthalpy of the reflux (Btu/lbmole)

h_{RSAT} : enthalpy of saturated reflux of composition X_R
        (Btu/lbmole)

H_{VAP} : heat of vaporization (Btu/lb)

MFT : main feed tank

N_F : feed tray

P-1 : feed pump

P-2 : bottoms pump

P_1-P : pressure drop across the orifice of d/p cell

P_2 : pressure upstream from inlet valve (psia)

P_{REB} : calandria steam pressure (psia)

R : reflux rate (lbmoles/hr)

S : steam rate   (lbmoles/hr)

t : time

tau : time constant

T-1 : distillate tank

T-2 : bottoms tank

T_F : temperature of the feed (^{o}C)

T_{FSAT} : temperature of saturated feed of composition X_F (^{o}C)

T_i (i=1,1,...24) : temperature of the vapor leaving the

i^{th} tray (^{o}C)

T_R : temperature of the reflux (^{o}C)

T_{RSAT} : temperature of saturated reflux of composition X_R

(^{o}C)

V-i (i=1,2,...15) : valves throughout the system

V_i (i=1,2,...24) : vapor rate leaving the ith tray

(lbmoles/hr)

$V_{SOL}(T)$: volumetric flowrate of the solution at temperature

T (gal/min)

$V_W(T)$: volumetric flowrate of water at temperature T

(gal/min)

w_S : mass flowrate of the steam (lbs/hr)

X_B : mole fraction of methanol in the bottoms

X_D : mole fraction of methanol in the distillate

X_F : mole fraction of methanol in the feed

X_i (i=1,2,...24) : mole fraction of methanol in the liquid

stream leaving the ith tray

X_R : mole fraction of methanol in the reflux

Y_i (i=1,2,...24) : mole fraction of methanol in the vapor

stream leaving the ith tray

ABSTRACT

Feed tray manipulation was used on a distillation col-
umn in an experimental attempt to control the top product
composition of a methanol-water system subjected to a feed
composition disturbance.

A steady-state model of a 24-tray, 8-inch diameter,
bubble cap column was developed and experimentally veri-
fied. It took into account the heat effects of subcooled
feed and reflux, Murphree efficiencies throughout the col-
umn other than 100%, and nonequimolar overflow. Using this
model, curves of the distillate composition X_D versus the
bottoms composition X_B were produced for various feed tray
locations and feed compositions which theoretically demon-
strated the steady-state feasibility of using the feed tray
as a manipulative variable.

Experiments were performed on the column using a series
of simple, empirical, steady-state and dynamic feedforward
control schemes which did, in fact, give satisfactory re-
sults for negative feed disturbances (a disturbance whose
composition was less than that of the original feed). How-
ever for a positive disturbance, pressure build-up in the
top of the column caused some secondary effects that de-
graded the effectiveness of the scheme. The steady-state
and initial transient results demonstrated the need for
some kind of dynamic controller and therefore several types

were experimentally tested. It was finally determined that
a simple dead time plus a gradual change of feed from one
tray to another gave effective feedforward control.

INTRODUCTION

Throughout the past twenty years interest in distillation dynamics has been largely motivated by developments in the field of automatic control. Industry has made wide use of conventional feedback schemes (1,2,3) but because of the nature of the distillation column engineers are now researching the possibility of using feedforward controls. Basically the distillation column is a distributed, non-linear, multivarible system having large dead times and large time constants. Traditional feedback control has had some difficulty with this type of system since it must wait until perturbations arise in the product streams before it can take the proper corrective action. On the other hand, the feedforward approach senses the disturbance in the input variable before it has time to act on the system and therefore may predict, in advance, its effect on the control variable. It can then manipulate other variables to compensate for this disturbance before any large deviations from the steady-state can develop.

The future role of feedforward control in the chemical industry was qualitatively discussed by Calvert and Coulman (4). There is no question that ideally this approach does offer a more scientific and a more perfect form of control. This has been confirmed by some early theoretical investigations in the field. Rippen and Lamb (5) used the

linear model of Lamb, Pigford, and Rippen (6) to obtain the frequency transfer functions for a binary distillation column and then, using matrix methods, synthesized some feedforward controllers for the system. Luyben and Gerster (7) modified this procedure somewhat by calculating the controllers directly without first calculating the plant transfer functions. The latter also experimentally verified the effectiveness of steady-state feedforward controllers. The practical application of this theory was presented by Lupfer and Parsons (8), Lupfer and Johnson (9), and MacMullan and Shinskey (10). More recently, Cadman, Rothfus, and Kermode (11) extended these linear, binary studies to multicomponent distillation and Distefano, May, and Huckaba (12) designed nonlinear feedforward controllers which theoretically made the size of the disturbance immaterial. In all of the above studies, reflux and vapor boilup have been the only manipulative variables used. However, Luyben (13) introduced a new idea by considering the feed tray location as the manipulative variable to correct for a feed composition disturbance.

Any new proposal must be economically justified. Shinskey (14) reported some economic considerations for the standard manipulative variables, but there are certain advantages offered by feed plate manipulation which might not be offered by reflux or vapor boilup. For example, if the column happens to be part of an interrelated system of proc-

esses, changing the reflux or boilup to compensate for a disturbance might, in itself, be a disturbance to another part of the system. Another possibility is that if the column is being pushed to maximum capacity, any change in the reflux or boilup could cause flooding. Finally, changing to the optimum feed plate might result in a reduction in utility consumption, especially if the feed plate were located in a pinch region. However each system must be examined separately because it is the system alone that dictates if any of the above advantages are applicable.

The work presented by Luyben (13) was a digital simulation study which theoretically demonstrated the dynamic effectiveness of using the feed tray as a manipulative variable. The present study is an experimental attempt to verify some of his results. It consists of very little theoretical work and the experimental testing is, for the most part, trial and error procedures. Even these crude, empirical techniques resulted in effective steady-state and dynamic feedforward control.

GENERAL DESCRIPTION OF THE SYSTEM

The distillation unit used in this study was a 24-tray, 8-inch diameter, bubble cap column. It was equipped with a vertical, thermosiphon reboiler, a feed preheater, both a total and a vent condenser, a distillate cooler, and a reflux drum. Distillate and reflux flows were due to gravity while there were separate pumps for the feed and bottoms. The system also included a main feed tank, a distillate tank, a bottoms tank, and two other tanks used to store and introduce feed having a composition different from that of the original feed. This system is shown in Figure I.

The reflux, distillate, bottoms, and steam flowrates were measured with Foxboro pneumatic flow transmitters and recorded automatically on Moore recording stations. The feed flowrate however, was determined with a calibrated rotameter and recorded by hand. The compositions of both the feed and bottoms were measured by taking discrete samples from their respective lines and then using hydrometers to determine their compositions. This, however, was not done for the distillate composition. Instead, because it was the control variable, it was continuously measured with a Princo Densitrol and continuously recorded on a Moore recording station. This Densitrol sent an electric signal to a Transmation transducer which, in turn, transmitted a pneumatic signal to the recording station. Thermocouples

were used to measure temperatures throughout the system and these values were continuously recorded on a Leeds and Northrup Speedomax.

There were two cascade control loops as shown in Figure II. Each loop was capable of controlling a tray temperature, a flowrate, or a valve position. For my case, the reflux and steam rates were held constant by putting the controllers on automatic control (See Control Loops of Appendix A). Two Moore Nullmatic controllers were used in this cascade system with the master controller having all three modes of corrective action while the slave controller had proportional and reset only.

For specific details on the equipment, see Appendix A.

SCOPE OF EXPERIMENTS

The purpose of this work was to experimentally determine whether the feed plate location could be used to control the distillate composition. The first step in this study was a steady-state analysis of the problem. This involved writing a digital computer program describing the steady-state system and using this program to construct curves of distillate composition versus bottoms composition for various feed tray locations and feed compositions. The experimental data necessary to use this program was:

1) feed rate, composition, and temperature;

2) reflux rate and temperature;

3) steam rate;

4) feed tray location;

5) column heat loss and plate efficiencies.

With the exception of the heat loss and efficiencies, all of the above data was recorded when the column was operating at steady-state conditions. The heat loss, however, was calculated from the above data by making an energy balance around the system. Having this information, the efficiencies were then determined by:

1) guessing efficiency values for each tray (see Ap-

pendix C, Description of the Model);

2) putting the data, including the efficiencies, into the steady-state program;

3) comparing the computed values of the distillate composition, of the bottoms composition, and of several temperatures throughout the column with the corresponding experimental values;

4) repeating the entire procedure again, if necessary, until the computed and experimental values are approximately equal.

The steady-state curves indicated that it was possible to control the distillate composition by changing the feed tray. However, initial transient runs showed that a dynamic controller was necessary for better control. Therefore, several empirical, dynamic approaches were tested. These included:

1) delaying the corrective change in feed tray a certain time period following the introduction of the disturbance;

2) changing to an intermediate tray before changing to the final feed tray;

3) changing back and forth between two feed trays;

4) instead of directly changing all of the feed from one tray to another, gradually change it by initially directing only part of the feed to the new

tray and then, after a certain time period, change the remainder of the feed to the new feed tray.

The operating procedures are described in detail in Appendix B.

DISCUSSION OF RESULTS

Using the developed model, steady-state curves of distillate composition X_D versus bottoms composition X_B were produced which held the standard manipulative variables of reflux and heat added to the reboiler constant while varying the feed tray location. These curves, shown in Figure IV, demonstrate the steady-state feasibility of using the feed tray as the manipulative variable. To illustrate this point, suppose that the column were operating at a steady-state condition which resulted from introducing a 40% methanol-water solution onto the 14th tray of the system. This point is designated A on Figure V. Now suppose that suddenly the feed composition dropped to about 31%. If no corrective action were taken, the distillate composition would decrease from a 94% to a 92% solution. However, if the feed plate were also changed to the 8th tray, the distillate composition would remain constant while the bottoms composition changed. The final steady-state condition would be that designated B on Figure V. This, of course, tells nothing about the dynamics or transient period resulting from the disturbance. All it illustrates is that when the system which was disturbed by a change in feed composition and later compensated for by a feed tray change returned to steady-state, the distillate composition would be the same as it was prior to the disturbance. Curve A of Figure

VI gives the experimental result of the system running at steady-state suddenly subjected (at time zero) to a feed composition change of from 40% to 31% methanol. It shows that the response of the distillate composition X_D to this disturbance is a dead time of approximately 6 minutes and a relatively slow, exponential decay. Curve B of Figure VI describes another experimental run which demonstrated that the response of X_D due to a change in feed tray of from the 14th to the 8th tray is faster and has a smaller dead time than that described by Curve A of the same figure. This brings out a point worth noting. To be able to attain perfect feedforward control, it is essential that

1) the dead time of the control variable resulting from a change in the manipulative variable be less than the corresponding dead time caused by a disturbance in an input variable;

2) the response of the control variable resulting from a change in the manipulative variable be faster than the corresponding response caused by a disturbance in an input variable.

Having now established the fact in Figure VI that a dynamic feedforward controller is physically realizable, we then empirically tested several of these simple, dynamic controllers.

The simplest of all controllers is one that takes immediate corrective action, that is, when the disturbance is first noticed in the input variable, there is an instantaneous change in one of the manipulative variables to compensate for it (in this case, the manipulative variable is the feed tray location). Using control symbols, this controller might look something like:

$$F_D \quad = \quad \left[\frac{N_F}{X_F} \right] \quad = \quad K$$

where K is a constant. However, this particular type of controller cannot always be used with satisfactory results. Curve A of Figure VII demonstrates what happened when it was experimentally tested, using the feed composition disturbance described earlier. There are two things to notice here. First of all, the corrective action tended to overcompensate and as a result the control variable deviated noticeably from the steady-state in the opposite direction from the openloop case (i.e. no control). Secondly, because of the large time constants involved, the system took a relatively long time to return to its steady-state. Of course, neither of these points are desired and therefore, some other controllers must be examined.

The next type tested was one in which the corrective action taken was identical to that of the preceding one

with one exception and that being, that the action was
begun after a certain time delay. This can be represented
by

$$F_D = \left[\frac{N_F}{X_F} \right] = K(e^{-(tau)t})$$

Curves B and C of Figure VII illustrate the experimental
effectiveness of this controller on the system which had
been subjected to the same feed composition change of
40% to 31%. Curve B describes the distillate composition
when the change in feed tray was delayed for three minutes
from the time the disturbance was first noticed. It is
evident that the perturbation observed here was not quite
so pronounced as that witnessed in Curve A, nor was the
time required for the system to return to steady-state
quite as long as that with the first controller. Curve C
follows this up by delaying the corrective action six min-
utes instead of three and the result is again better con-
trol. With this controller, before the effect of the feed
tray change became the predominant force, the composition
disturbance was actually noticed in the control variable.
This is evident in Curve C by the negative deviation from
the steady-state. The significant fact here is that both
the positive and negative perturbations were less than the
perturbations witnessed using the preceding controllers.

All controllers considered thus far have consisted of strictly one-step, instantaneous and delayed corrective action. Each of these indicate that a more sophisticated dynamic controller is essential for perfect control and therefore, several other dynamic approaches were tested. However, let me delay my discussion of these controllers, and instead describe, in some detail, the situation resulting from the introduction of a positive disturbance (the mole fraction of methanol was greater in the disturbance than in the original feed) into the system.

We have seen the effectiveness of feedplate manipulation theoretically verified by Luyben (13) and experimentally substantiated by some simple controllers. The arguments have all been favorable and there has been no disagreement between the theoretical and experimental approaches. However, this has not been true for all the disturbances tried, and in particular, when a positive disturbance was introduced. Here's the situation. The system was operating at steady-state with feed composed of 31% methanol being introduced onto the 8th tray. Suddenly the feed composition was changed to 40% and then some unexpected events occurred. The reflux to the top of the column had been placed on automatic control and the vapor rate in the enriching section started to increase slightly. This was due to the fact that there was now more methanol in the system and methanol has a lower heat of vaporization than

water. This, of course, would not be seen if the assumption of constant molal overflow were used.

With this increase in vapor rate, it was expected that the distillate rate would also increase to keep the reflux rate constant. However, this did not occur because of the gravity flow of the reflux. Instead, the reflux rate decreased, the distillate rate remained steady, and the level in the reflux drum began to rise. As the level mounted, the reflux rate began to increase and when the level reached some four inches above the overflow (see Figure III), the rate had returned to normal and the distillate rate had increased as was expected in the first place. It required about two minutes for the level to rise the four inches and during this time the increased vapor to reflux ratio tended to compensate somewhat for the disturbance. Another point to note was that now that the level was above the reflux overflow, the material leaving the condensers had to mix with the material already in the drum and a composite left as reflux. Because of this, the perturbations recorded in the control variable were much too slow to be controlled by one-step, dynamic feedforward controllers.

Let's now look at what might have caused this problem. The pressure in the top of the column before the disturbance was approximately 10 inches of water above atmospheric. The vapor rate was about 9.65 pound-moles per hour. After the disturbance the pressure had risen to 14 inches

of water above atmospheric and the vapor rate had risen to
9.8 pound-moles per hour. This increase in pressure, result-
ing from the increase in material in the enriching section
of the column, is what caused the decrease in reflux and
the subsequent level rise in the reflux drum. This entire
problem could have been avoided by simply raising the drum
up to increase the pressure head or driving force of the
reflux.

Let me now return and discuss some of the other dynam-
ic approaches tested. The next controller involved the use
of an intermediate tray. Instead of changing directly from
the 14th to the 8th tray, the feed was initially introduced
onto the 10th tray some six minutes after the disturbance,
and then after a couple of minutes on that tray, it was fi-
nally directed to the 8th tray. Intuitively, this would
seem to help alleviate the problem of overcompensating, but
unfortunately the experimental results do not show any sig-
nificant improvement over that described by Curve C of Fig-
ure VII. There are a couple of possible explanations. For
one thing, the steady-state effect of changing the feed pl
plate from tray 14 to tray 10 is very similar to the effect
caused by a direct change to tray 8. Perhaps if the feed
were inserted onto the 12th or 13th tray, more conclusive
results might have been obtained. However, this was not
possible because the experimental system only permits the
feed to be introduced on one of five trays, the 2nd, 4th,

8th, 10th, or 14th. Another possible and more probable explanation is that the system with all its variables and controls was not sensitive enough to record the initial difference between two very similar controllers and it required too long a time to allow the steady-state difference, noticed in Figure V, to come into the picture.

The next approach involved switching the feed between two plates, namely the 14th and the 8th. Curve A of Figure VIII describes a situation which is very much like that of Curve C in Figure VII. The column was running at steady-state, a disturbance was introduced, and the feed tray was changed from tray 14 to tray 8. But then, as soon as the effect of the feed tray change began to overtake the perturbation caused by the disturbance (when the control variable started to rise in Curve A), the feed was suddenly switched back to tray 14. After a short period of time, the control variable again reversed direction and the experiment was concluded. Although this approach did not improve the initial perturbations, it did illustrate that changing the tray location does have a significant and a relatively fast effect on the distillate composition.

From the results presented thus far, it became evident that to obtain better control it would be necessary to gradually change the feed tray instead of switching directly from one tray to another. Therefore, the final controller tested was one consisting of a simple dead time coupled

with a gradual change of feed from tray 14 to tray 8. This lag was accomplished by first splitting the feed between t trays 14 and tray 8 for six minutes before changing it all to tray 8. This helped to eliminate some of the perturbations arising from overcompensating and, as shown by Curve B of Figure VIII, gave the best control of any of the controllers tested. More details are given in Section III of Appendix B.

FIGURE I

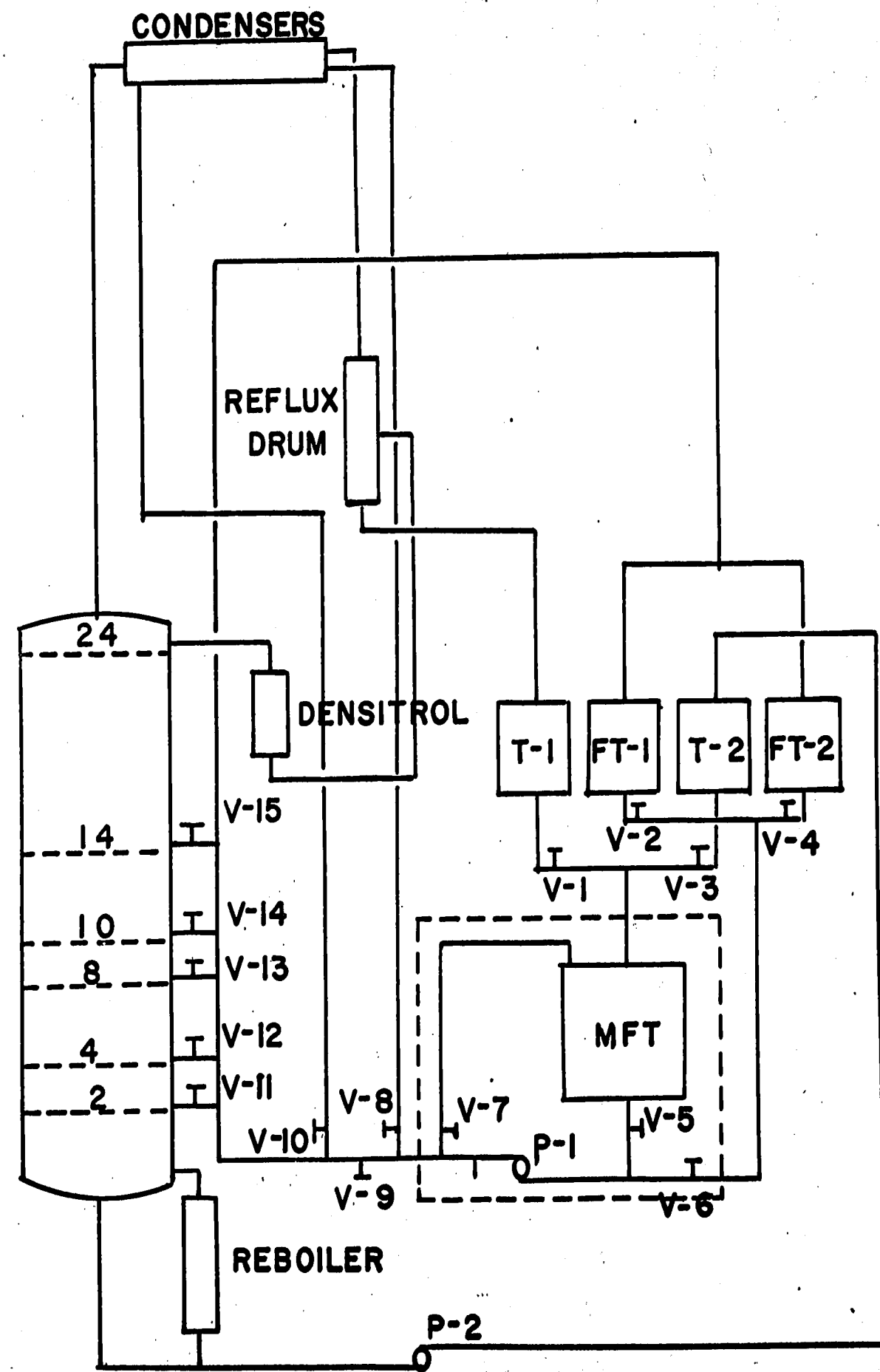## DIAGRAM OF DISTILLATION SYSTEM

FIGURE II
INSTRUMENTATION ON DISTILLATION COLUMN
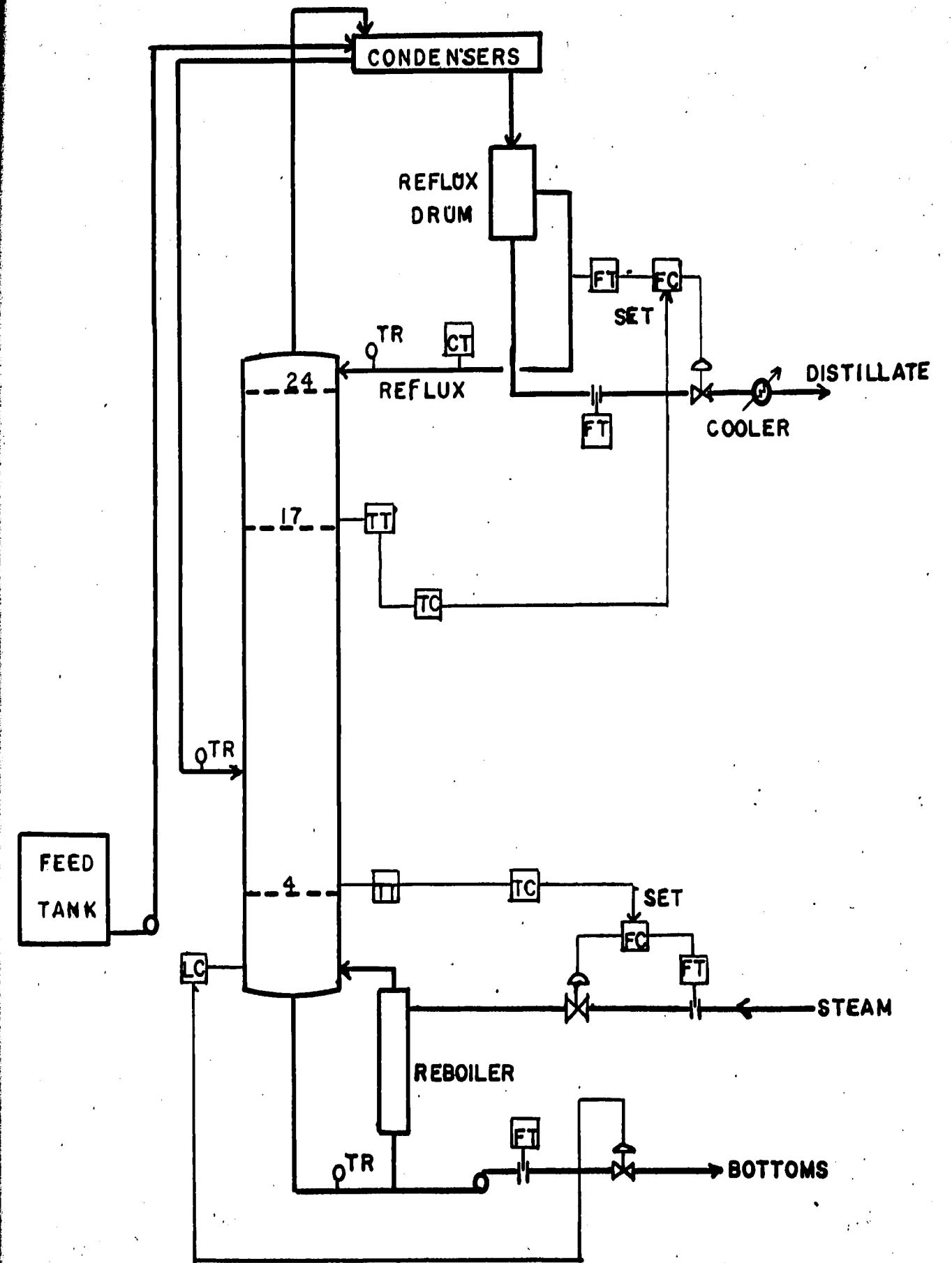
FIGURE III

CONDENSER AND REFLUX DRUM ARRANGEMENT
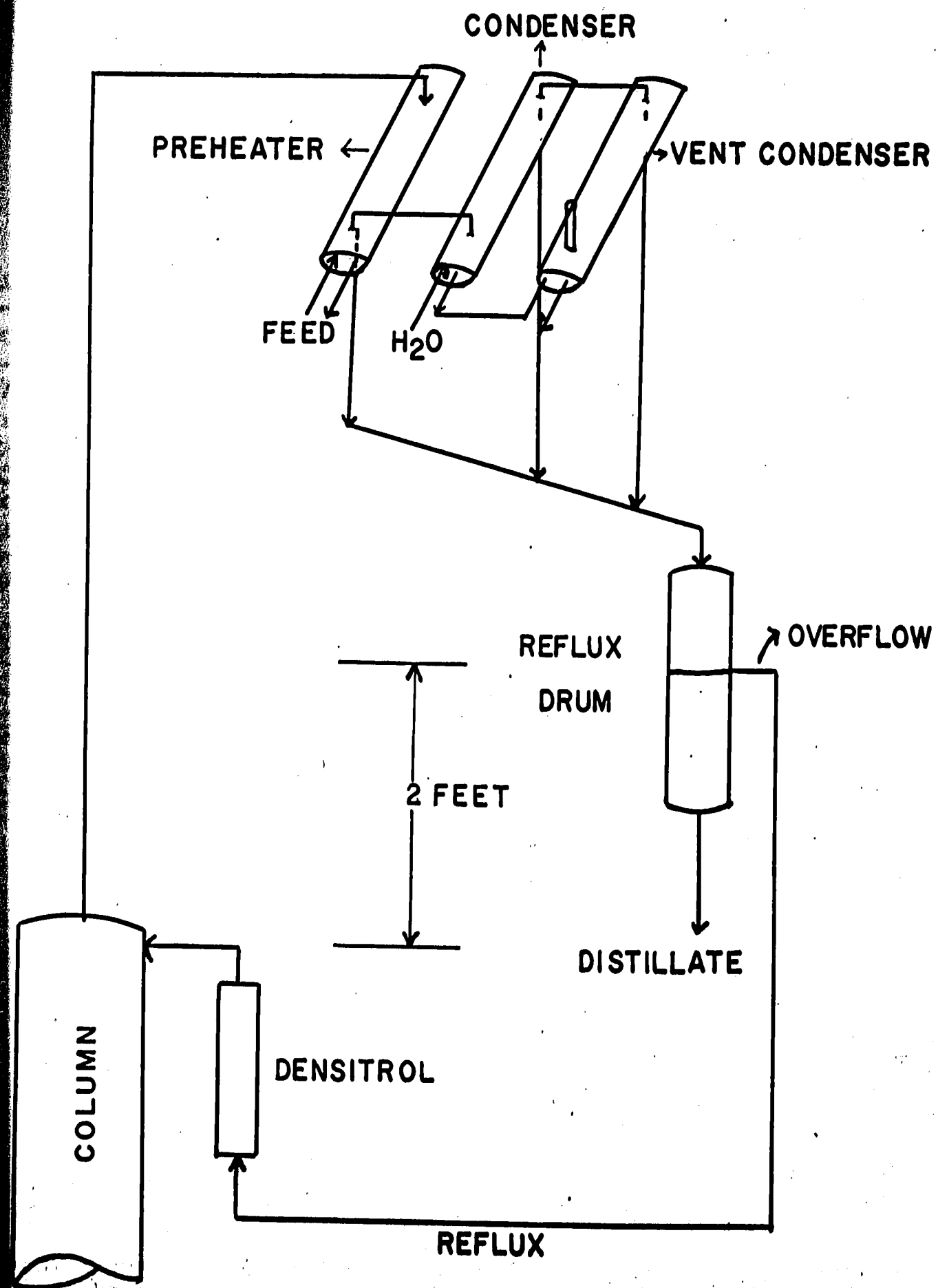
FIGURE IV

OPENLOOP STEADY-STATE

23.
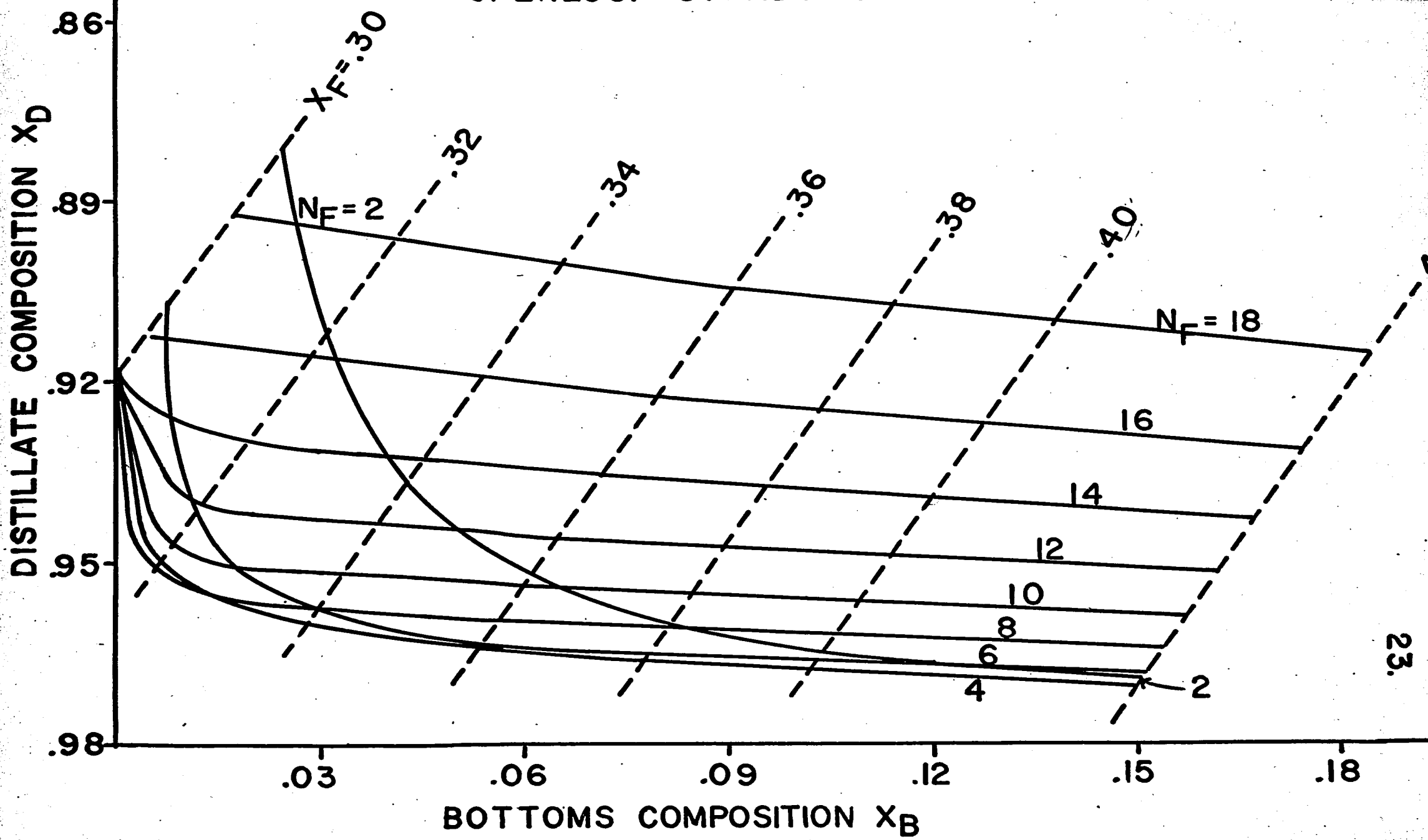
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PAGES

FIGURE VI
OPENLOOP RESPONSES

FIGURE VII
RESPONSES OF CONTROLLED VARIABLE

FIGURE VIII

RESPONSES OF CONTROLLED XD

A - DIRECT CHANGE IN N_F

B - GRADUAL CHANGE IN N_F

FIGURE VI
OPENLOOP RESPONSES

A - ΔN_F

B - ΔX_F

FIGURE VII
RESPONSES OF CONTROLLED VARIABLE

DELAY IN CONTROL
A - NO DELAY
B - 3 MIN. DELAY
C - 6 MIN. DELAY

FIGURE VIII

RESPONSES OF CONTROLLED XD

A - DIRECT CHANGE IN N_F

B - GRADUAL CHANGE IN N_F
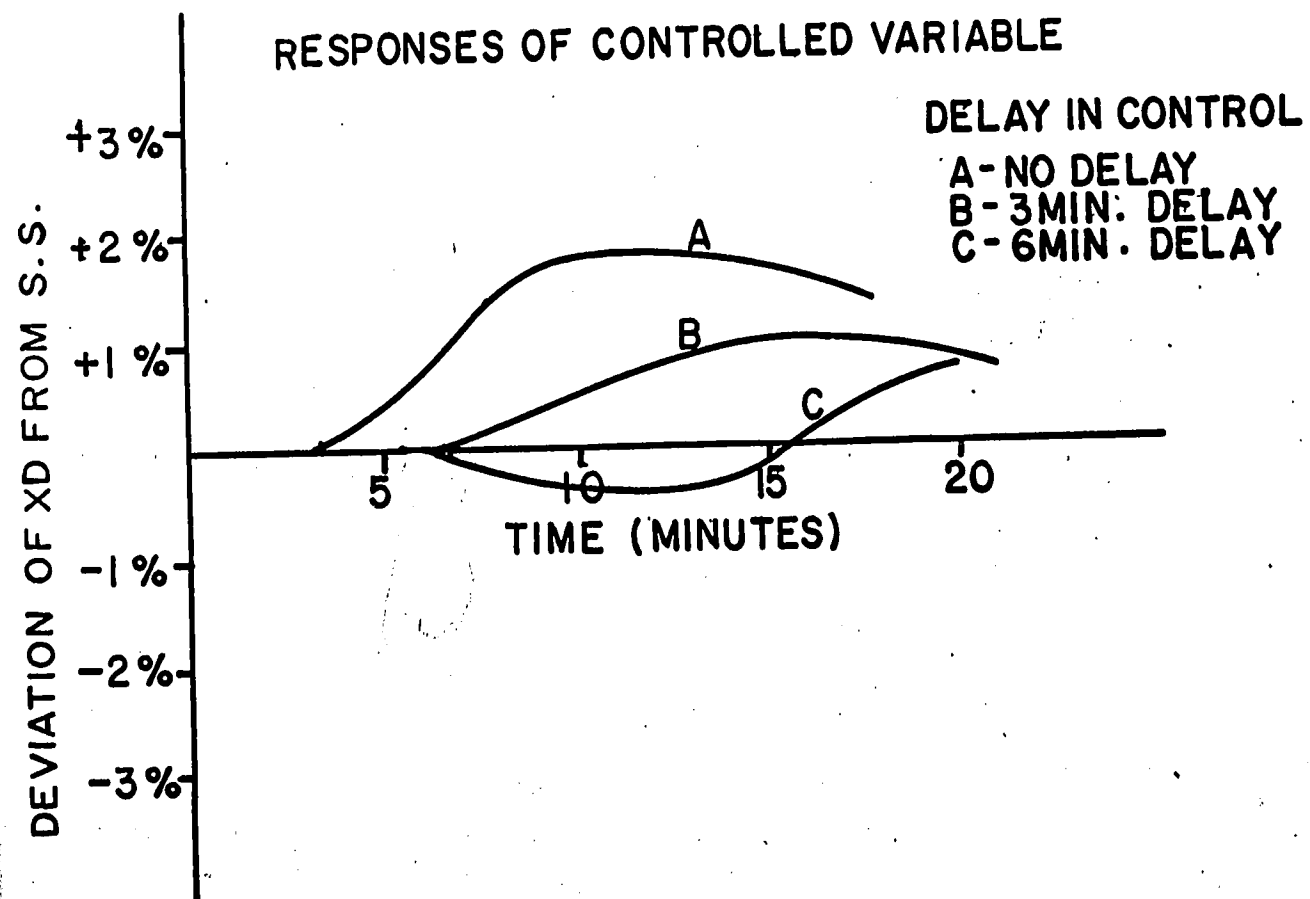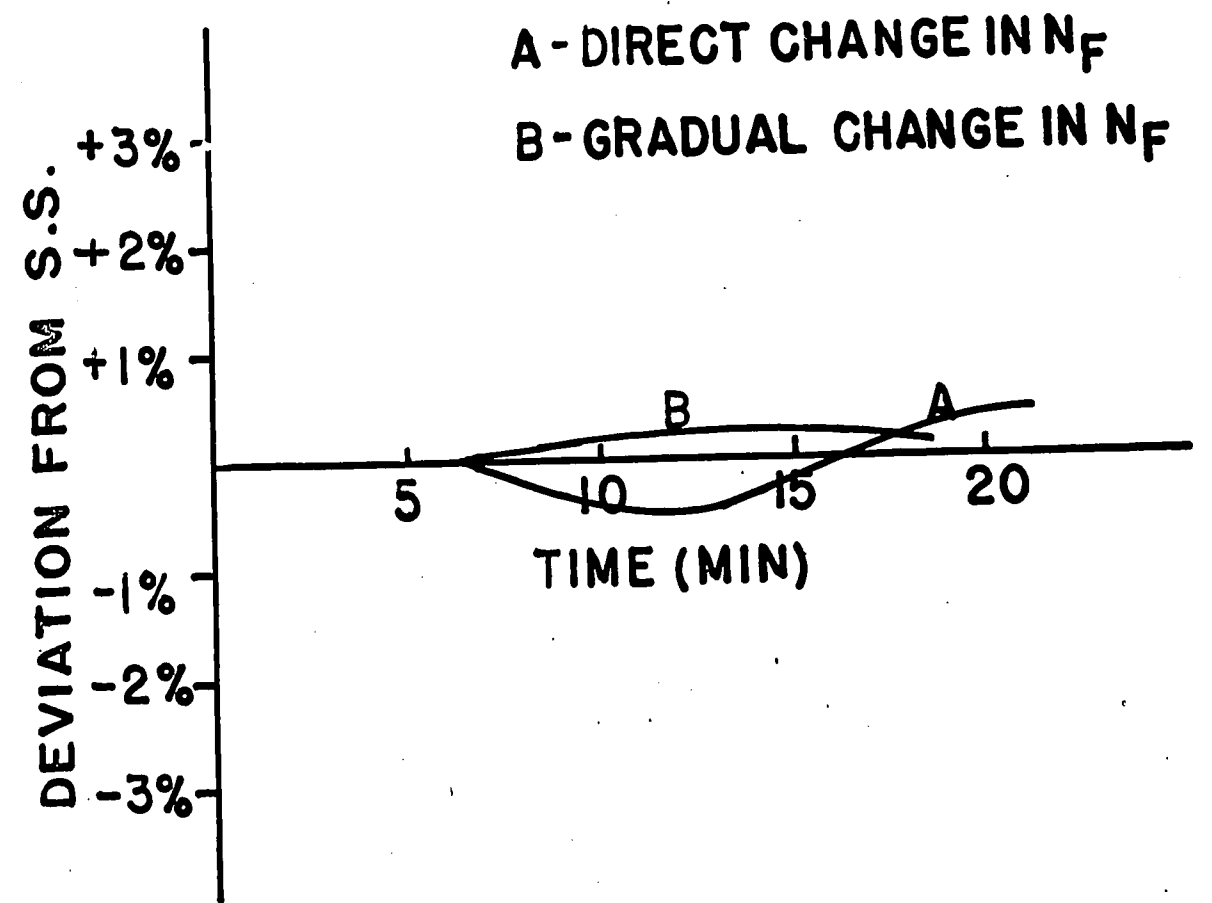
CONCLUSIONS

The effect of changing the feed plate to compensate for a feed composition disturbance has been demonstrated in this report. It was shown that for a feed composition change from 40% to 31% methanol, the distillate composition could be controlled reasonably well by delaying the feed tray change from the 14th to the 8th tray for approximately six minutes. It was also demonstrated that better control would be achieved by first splitting the feed between the two trays for six minutes before finally changing all of the feed to the new tray.

APPENDIX

APPENDIX A

Column and Controls

I Description of Column

Tower

- 8-inch inside diameter seamless copper pipe

- twenty-four bubble cap trays spaced 6 inches apart

- two 3-inch outside diameter bubble cap assemblies
  per tray with 5.54 square inches total slot area

- one downpipe with 3.3 square inches cross sectional
  area

- effective length of overflow weir is 6.5 inches

- feed can be introduced on trays 2, 4, 8, 10, 14

Condensers (preheater, condenser, and vent condenser)

- 4-inch outside diameter seamless copper shell with
  cupro-nickel tube sheets

- ten 4-foot long tubes, each having an outside diam-
  eter of 0.625 inches and 0.065 inches thick, with
  double pass arrangement

- outside heat transfer area of 6.54 square feet

Calandria (vertical thermosiphon) and Distillate Cooler

- 4-inch outside diameter seamless copper shell with
  cupro-nickel tube sheets

- fourteen 4-foot long tubes, each having an outside

diameter of 0.625 inches and 0.065 inches thick, with single pass arrangement

- outside heat transfer area of 9.15 square feet

Reflux Drum (see Figure III)

- 6-inch outside diameter seamless copper pipe, 24 inches long with reflux overflow 12 inches from the bottom

Main Feed Tank

- 26-inch outside diameter by 22 inches high copper drum

- operating capacity of 50 gallons

- internal steam coil with 5.3 square feet of heat transfer area and sparger provided for direct steam

Receiving Tanks (distillate, bottoms, and two feed tanks)

- 16-inch outside diameter by 24 inches high copper drum

- operating capacity of 20 gallons

Feed Pump

- turbine pump with 5-horsepower motor

Bottoms Pump

- turbine pump with 1-horsepower motor

II Instrumentation

Flowrates

Feed

- measured by a rotameter having a range of 0 to 1.2 gallons per minute

Bottoms

- measured by a Foxboro 13A1 pneumatic flow transmitter with an integral orifice (0.159 inches) attachment having a range of 0 to $(0.99805/d_s(T))^{\frac{1}{2}}$ gallons per minute (See Appendix E, Section I)
- recorded on a Moore 3-pen recording station

Distillate

- measured by a Foxboro 13A pneumatic flow transmitter with an integral orifice (0.159 inches) attachment having a range of 0 to $0.8(0.79924/d_s(T))^{\frac{1}{2}}$ gallons per minute (See Appendix E, Section I for sample calculation)
- recorded on a Moore 3-pen recording station

Reflux

- same as Distillate except for its range which is 0 to $(0.79924/d_s(T))^{\frac{1}{2}}$ gallons per minute

Steam

- measured by a Foxboro 13A pneumatic flow transmitter having a range of 0 to $3(p2/44.7)^{\frac{1}{2}}$ (See Appendix E, Section II)
- recorded on a Moore 3-pen recording station

Compositions

Feed

- measured with a hydrometer with its smallest
increment being 0.001

Bottoms

- measured with a hydrometer with its smallest
increment being 0.001

Distillate

- measured continuously with a Princo Densitrol
Pipe Line Model having a density range of 0.7675
to 0.7875 @ 50°C

Temperatures

- measured with iron-constantan thermocouples in-
serted into each of the following: cooling water
to condensers, reflux line, reboiler, steam in,
liquid leaving tray 8, and vapor leaving trays
2, 10, 14, 20, 24

- recorded on a Leeds and Northrup Speedomax
12-point recorder

- two Moore Nullmatic temperature transmitters
inserted into trays 4 and 17 with the tempera-
tures being recorded on the control station it-
self (See Control Loops for further description)

Pressures

- there are three pressure gauges, one in the

reboiler, one in the steam line into the reboil-
er, and one in the top of the column

Levels

  - there is a liquid level controller to control
    the level in the bottom of the column

III <u>Control Loops</u>

  Upper Control Loop

    Manual

      - controls distillate valve directly

    Automatic

      -controls reflux flow by manually changing the
        reflux set point

    Cascade

      - controls the 17th tray temperature by automati-
        cally changing the reflux set point

  Lower Control Loop

    Manual

      - controls steam valve directly

    Automatic

      - controls steam flow by manually changing the
        steam set point

    Cascade

      - controls the 4th tray temperature by automati-
        cally changing the steam set point

APPENDIX B

Operating Procedure

I Feed Preparation

For the first experimental run, approximately 15 gallons of water and 15 gallons of methanol were loaded into the main feed tank, MFT. This material was then mixed by hand for three minutes and circulated by the feed pump, P-1, around the system denoted by the dashed lines in Figure I and constructed by closing valves V-6, V-8, and V-9 and opening valves V-5 and V-7. A sample was withdrawn and immediately analysed with the hydrometer to determine its composition. If the solution differed by more than ± 1% from the desired value of 31%, either methanol or water, whichever was appropriate, was added to the system. The entire procedure, starting with the mixing by hand, was then repeated. While the feed was being circulated, samples were taken and analysed every ten minutes. When three successive samples indicated approximately the same composition, about 20 gallons of the material was pumped into tanks FT-1 and FT-2 to be used as the feed disturbance. This was accomplished by opening valve V-9 and closing valves V-6, V-7, and V-10 through V-15. Whenever a sample other than the first correct one, differed by the ± 1%, the next sample was withdrawn five minutes later instead of the usual ten. If this again

indicated the same error, the pump was shut off, the water
or methanol added, and the procedure begun again. If how-
ever, the second sample did not confirm the disagreement,
the procedure was again repeated, but this time, without
adding any new material.

With a few noted exceptions the same technique was em-
ployed in preparing the feed as in preparing the feed dis-
turbance. Therefore, instead of repeating it, let me sim-
ply list the differences and discuss those that need it.

1) The desired composition of the feed was 40% instead
   of 31%;
2) More material was needed - approximately 45 gallons
   instead of 30;
3) It was necessary to collect four successive samples
   instead of three.

Looking at the second point, more material was necessary
because the system had to reach steady-state before the
disturbance could be introduced and this required anywhere
from 45 minutes to an hour. With a feedrate of 0.7 gallons
per minute, it was necessary to have at least 42 gallons of
feed on hand to operate for one hour. It was also essen-
tial to collect four instead of three samples because of
this extra material. The feed was circulated at a rate of
about 1.8 gallons per minute and at this rate it took 25
minutes to circulate the entire load of 45 gallons. There-

fore, it was necessary to collect the four samples because this guaranteed 30 minutes of circulation instead of the 20 when only three samples were taken.

II Steady-State Model

A methanol-water solution consisting of approximately 36% methanol was fed into the column on the 8th tray at a rate of 14 pound-moles per hour. This was accomplished by opening valves V-5, V-8, V-10, and V-13, by closing the remaining valves listed in Figure I, and then using P-1 to pump the material to the feed tray. With this arrangement of valves, the feed was preheated before it entered the column. Steam was introduced into the reboiler and after a short period of time the level in the reflux drum started to rise. Once this level reached the overflow, the distillate valve was gradually opened to keep the reflux flow at the desired rate (see Figure III). When the system finally reached steady-state, both the temperature readings on the multipoint recorder and the signal from the densitrol were holding constant. Samples were taken from the feed, the distillate, the bottoms, and trays 3, 7, and 13. Along with this, the feed, reflux, distillate, bottoms, and steam rates, the reboiler pressure and temperatures of the feed, reflux, and several trays throughout the column were also recorded. Part D, Section II of Appendix C lists

and compares the experimental data from this part of the study with the computed results from the steady-state model.

III <u>Feedforward Controllers</u>

Fourteen pound-moles per hour of feed consisting of 40% methanol was introduced onto the 14th tray of the column. Once again, valves were arranged so that the feed was preheated before entering the column. 175 pounds of steam was then inserted into the reboiler every hour and the reflux rate was set at 5 pound-moles per hour. Valves V-1 and V-3 were closed to keep the distillate and bottoms products from returning immediately to the main feed tank. This return to the feed was undesirable because it would have changed the feed composition during the experiment. The distillate flow was by gravity into tank T-1 and the bottoms was pumped into tank T-2. The column was then run until it reached steady-state at which time the steam and reflux flows were placed on automatic control to keep them constant (see Figure II and Control Loops). After the system had remained at steady-state for a couple of minutes, the disturbance was introduced by manually opening valve V-6 and manually closing valve V-5. Because of the limited size of the tanks T-1 and T-2, it was sometimes necessary to open valves V-1 and/or V-3 for a couple of minutes once

the disturbance was introduced. This prevented any build-up of distillate or bottoms products while, at the same time, did not change the feed composition because the feed was being introduced from the space feed tanks FT-1 and FT-2. In order to change the feed to the 8th tray for the first controller tested, valve V-13 was opened and valve V-15 was closed immediately after the disturbance was introduced. The total operation of introducing the disturbance and changing the feed tray took approximately 15 seconds and therefore this first controller is considered to give instantaneous corrective action. For the remaining controllers tested, the valves were again manually opened and closed according to their individual demands. Finally, the feed, distillate, reflux, and steam rates were recorded, along with the reboiler pressure. There was also a chart recording the distillate composition starting immediately before the disturbance in order to follow the control variable through the entire transient period following the change in feed.

In order to test the final controller which gradually changed the feed from tray 14 to tray 8 it was necessary to insert another rotameter between the two trays as shown in Figure A on the next page. This enabled me to change the feed to tray 8 in the following manner:

1) Delaying the corrective action for four minutes

FIGURE A

following the disturbance;
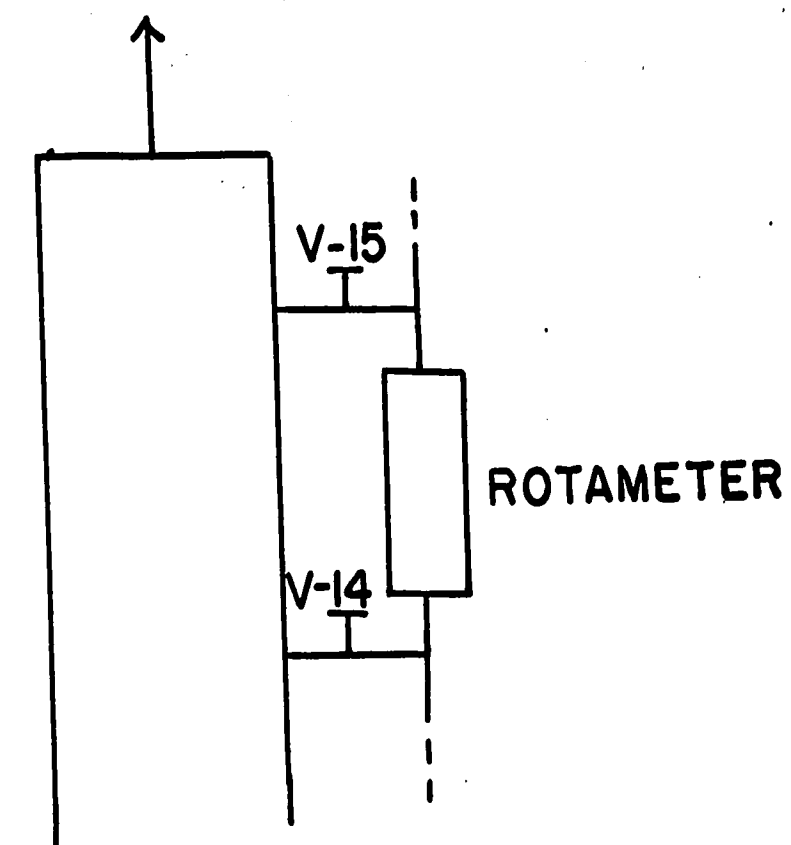
2) Opening valve V-14 until the rotameter indicated
that the feed was evenly split between the 14th and
8th trays;

3) Maintaining this valve position for six minutes
before opening it all the way;

4) Closing valve V-15 so that little or no vapor es-
capes from the column through one of the feed lines.

The results of this part of the experiment are shown
in Figures VI through VIII.

APPENDIX C

The Steady-State Model

I Description of the Model

Before any experimental investigations using feedfor-
ward controllers were performed on the column, it was first
necessary to develop a steady-state model of the system.
This model had to describe a binary, nonlinear, nonequimo-
lar overflow system and had to account for subcooled feed
and reflux, overall heat losses throughout the column, and
Murphree efficiencies other than 100%. The equations used
to describe this system were the standard mass and energy
equations and therefore the development of the model essen-
tially involved the experimental determination of the heat
losses and efficiencies throughout the column. Section II
is a detailed description of the experimental data and cal-
culations involved and Part C of this section shows that
there were essentially no heat losses through the walls of
the column. There are a couple of points worth discussing
here. First of all, the column is entirely insulated and
it seems reasonable that any heat loss which might develop
would be negligible. Secondly, the distillate and bottoms
flowrates used in the total energy balance are calculated
from the experimental values of the feed flowrate and of
the feed, distillate, and bottoms compositions. These flow-

rates differ slightly from the experimental values but are used in order to make the material and component balances in the model exact. (See page 44)

With the overall heat loss specified, the only task remaining was to determine the efficiencies throughout the column. Floyd and Hipken (16) discuss the difficulty encountered when each tray is considered separately and recommend splitting the column into sections with constant efficiencies. This is precisely what was done by giving each tray in the enriching section one efficiency and each tray in the stripping another. The reboiler was also given a separate value because experimental results indicate that its efficiency is much higher than in either of the two sections. There are a number of ways to assign these values and the one chosen for this report was simply to guess the efficiencies, put them into a steady-state program, and compare the computed values with the experimental. If they are sufficiently close, it is assumed that the efficiencies chosen were correct. If not, new values were assigned and the procedure repeated. There was a problem however, in measuring compositions on intermittent trays and so thermocouples were spaced throughout the column to allow a comparison of temperature profiles which is an approximate method of comparing compositions. Part D of Section II lists the results for the efficiencies finally chosen. However, another run was made with the feed introduced on the

14th tray to determine if the efficiencies changed with a change in the feed plate. The results of the run are given in Section III and the agreement between the computed and the experimental values was again quite reasonable. Therefore the efficiencies chosen remain 90% for the reboiler, 55% for the stripping section, and 40% for the enriching.

II <u>Steady-State Calculations</u>

A. <u>Experimental Data</u>

$$Feed\ Rate\ (F) = 0.700\ gallons\ per\ minute$$
$$= 5.340\ pounds\ per\ minute$$
$$= 0.233\ pound\text{-}moles\ per\ minute$$
$$= 14.000\ pound\text{-}moles\ per\ hour$$

$$Reflux\ Rate\ (R) = 0.3950\ gallons\ per\ minute$$
$$= 2.6200\ pounds\ per\ minute$$
$$= 0.0833\ pound\text{-}moles\ per\ minute$$
$$= 5.0000\ pound\text{-}moles\ per\ hour$$

$$Distillate\ Rate\ (D) = 0.38\ gallons\ per\ minute$$
$$= 2.52\ pounds\ per\ minute$$
$$= 0.08\ pound\text{-}moles\ per\ minute$$
$$= 4.80\ pound\text{-}moles\ per\ hour$$

$$Bottoms\ Rate\ (B) = 0.358\ gallons\ per\ minute$$
$$= 2.940\ pounds\ per\ minute$$
$$= 0.158\ pound\text{-}moles\ per\ minute$$
$$= 9.500\ pound\text{-}moles\ per\ hour$$

$$Steam\ Rate\ (S) = 29.17\ pounds\ per\ minute$$
$$= 175.00\ pounds\ per\ hour$$

Feed Composition (X_F) = 35.52% Methanol

Distillate Composition (X_D) = 95.90% Methanol

Bottoms Composition (X_B) = 5.25% Methanol

Feed Temperature (T_F) = 58.0 degrees Centigrade

Reflux Temperature (T_R) = 60.0 degrees Centigrade

Pressure Before Steam Valve = 75.0 pounds per square inch guage

Calandria Steam Pressure = 18.0 pounds per square inch guage

Feed Tray (N_F) = 8

B. Material Balances

Total - At steady-state, the total amount of materi-
al entering the column as feed should equal the total
amount of material leaving the column as bottoms and
distillate.

$$B = 9.5 \text{ lbmoles/hr}$$
$$D = 4.8 \text{ lbmoles/hr}$$
$$F = 14.0 \text{ lbmoles/hr}$$
$$B + D = 14.3 \text{ lbmoles/hr}$$

Component - At steady-state, the amount of methanol
entering the column in the feed stream should equal
the amount of methanol leaving the column in the bot-
toms and distillate streams.

$$B(X_B) = 9.5(0.0525) = 0.49875 \text{ lbmoles/hr}$$
$$D(X_D) = 4.8(0.9590) = 5.10195 \text{ lbmoles/hr}$$
$$F(X_F) = 14.0(0.3552) = 4.9728 \text{ lbmoles/hr}$$
$$B(X_B) + D(X_D) = 5.6007 \text{ lbmoles/hr}$$

From the above results, it is evident that the meas-
ured values are approximately correct. However, to
make these balances exact for the mathematical model,
it was assumed that the compositions and the feed-
rate were the most accurate of the measured varia-
bles. Because of this, these values were then used
to calculate the bottoms and distillate flowrates.

$$F(X_F) = B(X_B) + D(X_D)$$

$$B = F - D$$

$$F(X_F) = (F - D)(X_B) + D(X_D)$$

$$F(X_F) = F(X_B) - D(X_B) + D(X_D)$$

$$F(X_F - X_B) = D(X_D - X_B)$$

$$D = F(X_F - X_B) / (X_D - X_B)$$

$$D = \frac{14.0(0.3552 - 0.0525)}{0.9590 - 0.0525}$$

$$D = 4.67 \text{ lbmoles/hr}$$

$$B = 9.33 \text{ lbmoles/hr}$$

C. <u>Calculation of Column Heat Loss</u>

Energy of the Incoming and Outgoing Streams
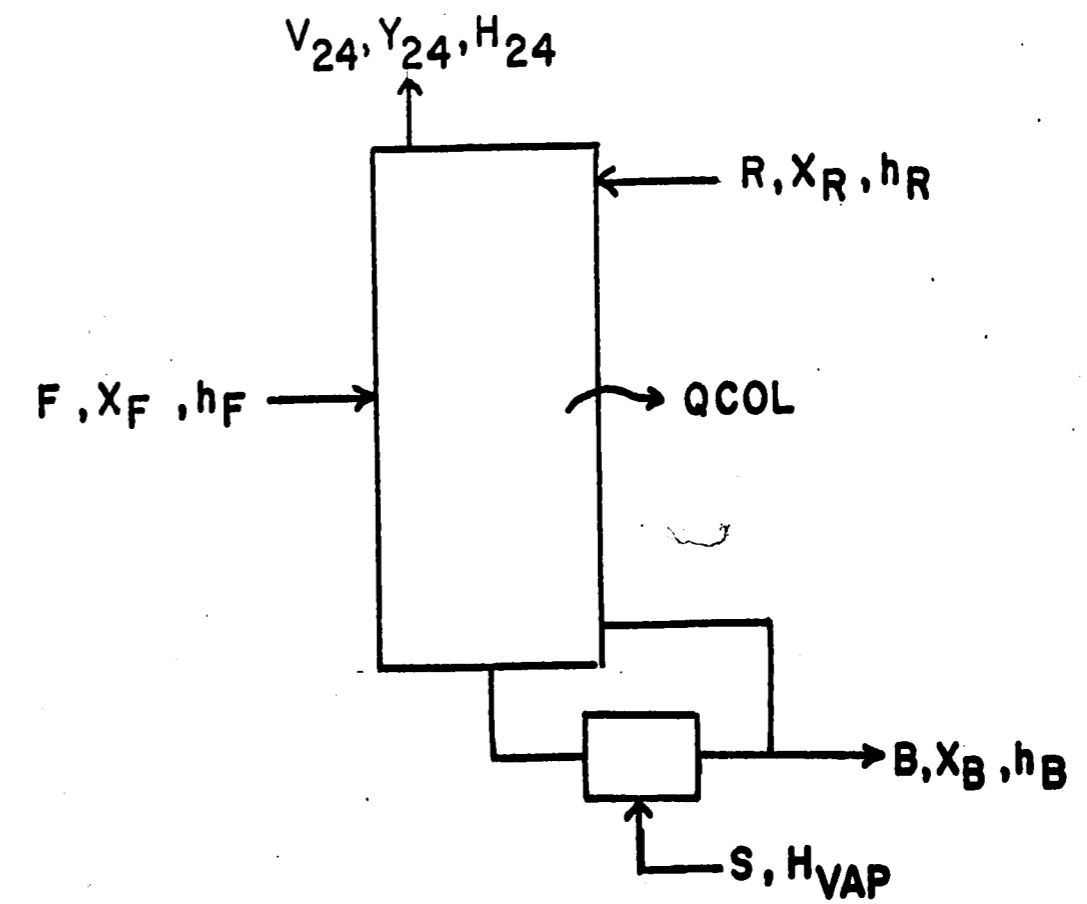
FIGURE B

Feed

- Experimental Data

    $X_F = 0.3552$

    $F = 14.0$ lbmoles/hr

    $T_F = 58.0$ °C

- Saturated Enthalpy and Temperature

    h_{FSAT} (@$X_F = 0.3552$) = 2035.47 Btu/lbmole

    T_{FSAT} (@$X_F = 0.3552$) = 76.51 °C

- Specific Heat of Solution

    $CP_{SOL} = X_F(CP_M) + (1 - X_F)(CP_W)$

    $CP_{SOL} = 0.3552(35.958) + 0.6448(32.436)$

    $CP_{SOL} = 33.687$ Btu/lbmole-°C

- Enthalpy of Solution

    $h_F = h_{FSAT} - CP_{SOL}(T_{FSAT} - T_F)$

    $h_F = 2035.47 - 33.687(76.51-58.0)$

    $h_F = 1411.93$ Btu/lbmole

- Energy into Column

    $E_F = F(h_F)$

    $E_F = 14.0(1411.93) = 19767$ Btu/hr

Reflux

- Experimental Data

    $X_R = 0.959$

    $R = 5.0$ lbmoles/hr

    $T_R = 60.0$ °C

- Saturated Enthalpy and Temperature

    h_{RSAT} (@$X_R = 0.959$) = 2216.25 Btu/lbmole

$$T_{RSAT}(@X_R = 0.959) = 2216.25 \text{ Btu/lbmole}$$

- Specific Heat of Solution

$$CP_{SOL} = X_R(CP_M) + (1 - X_R)(CP_W)$$

$$CP_{SOL} = 0.959(35.958) + 0.041(32.436)$$

$$CP_{SOL} = 35.812 \text{ Btu/lbmole-}^\circ C$$

- Enthalpy of Solution

$$h_R = h_{RSAT} - CP_{SOL}(T_{RSAT} - T_R)$$

$$h_R = 2216.25 - 35.812(65.95 - 60.0)$$

$$h_R = 2003.17 \text{ Btu/lbmole}$$

- Energy into Column

$$E_R = R(h_R)$$

$$E_R = 5.0(2003.17) = 10015.8 \text{ Btu/hr}$$

Steam

- Experimental Data

$$P_{REB} = 1^8 \text{ psig} = 32.7 \text{ psia}$$

$$S = 175.0 \text{ lb/hr}$$

- Heat of Vaporization

$$H_{VAP} (@P_{REB} = 32.7) = 941 \text{ Btu/lb}$$

- Energy into Column

$$E_S = S(H_{VAP})$$

$$E_S = 175.0(941) = 164500 \text{ Btu/hr}$$

Bottoms

- Experimental Data

$$X_B = 0.0525$$

$$B = 9.33 \text{ lbmoles/hr}$$

- Saturated Enthalpy

$$h_{BSAT} \ (@X_B = 0.0525) = 3088.23 \ Btu/lbmole$$

- Enthalpy of Solution

$$h_B = h_{BSAT} = 3088.23 \ Btu/lbmole$$

- Energy out of Column

$$E_B = B(h_B)$$

$$E_B = 9.33(3088.23) = 28800 \ Btu/hr$$

Vapor Leaving Top of Column

- Experimental Data

$$Y_{24} = X_D = 0.959$$

$$V_{24} = R + D = 5.0 + 4.67 = 9.67 \ lbmoles/hr$$

- Saturated Enthalpy

$$H_{VSAT} = 20596.87 - YN(3641.29)$$

$$H_{24SAT} = 20596.87 - 0.959(3641.29)$$

$$H_{24SAT} = 17104.88 \ Btu/lbmole$$

- Enthalpy of Solution

$$H_{24} = H_{24SAT} = 17104.88 \ Btu/lbmole$$

- Energy out of Column

$$E_{24} = V_{24}(H_{24})$$

$$E_{24} = 9.67(17104.88) = 165400 \ Btu/hr$$

Figure B shows that there is one more stream leaving the column and this, of course, is the total heat loss from the column. It can be calculated by simply making an energy balance around the column plus reboiler, and this is precisely what is done on the following page.

Energy Balance

Incoming

Feed	-	19767	Btu/hr
Reflux	-	10016	Btu/hr
Steam	-	164500	Btu/hr
		194283	Btu/hr

Outgoing

Bottoms	-	28800	Btu/hr
Vapor	-	165400	Btu/hr
		194200	Btu/hr

Incoming - Outgoing = 0

Therefore there is no heat loss from the column

D. Comparison of Computed with Experimental Values

The efficiences finally chosen for the model were:

Reboiler -- 0.90

Stripping -- 0.55

Enriching -- 0.40

Variable	Experimental	Model
T_2	$81.0^{\circ}C$	$80.20^{\circ}C$
T_4	$76.2^{\circ}C$	$76.87^{\circ}C$
T_8	$75.0^{\circ}C$	$75.46^{\circ}C$
T_{10}	$72.5^{\circ}C$	$73.30^{\circ}C$
T_{14}	$69.8^{\circ}C$	$70.58^{\circ}C$
T_{17}	$68.0^{\circ}C$	$68.42^{\circ}C$

Variable	Experimental	Model
T_{20}	$67.6^{\circ}C$	$66.53^{\circ}C$
T_{24}	$65.6^{\circ}C$	$65.06^{\circ}C$
X_3	0.334	0.281
X_7	0.348	0.376
X_{13}	0.645	0.615
X_D	0.959	0.959
X_B	0.0525	0.050

III Comparison of Model with Experimental Values for $N_p=14$

Data

Feed Rate = 13.4 pound-moles per hour

Reflux Rate = 5.0 pound-moles per hour

Steam Rate = 175.0 pounds per hour

Feed Composition = 35.05% Methanol

Feed Temperature = 58.0 degrees Centigrade

Reflux Temperature = 60.0 degrees Centigrade

Pressure Before Steam Valve = 75.0 pounds per square
inch guage

Calandria Steam Pressure = 18.0 pounds per square
inch guage

Feed Tray = 14

Variable	Experimental	Model
T_2	82.5°C	81.81°C
T_4	76.8°C	77.27°C
T_8	76.0°C	75.40°C
T_{10}	75.5°C	75.21°C
T_{14}	74.8°C	75.11°C
T_{17}	72.5°C	71.34°C
T_{20}	69.5°C	68.52°C
T_{24}	66.2°C	65.52°C
X_D	0.9313	0.93400
X_B	0.0212	0.03437

APPENDIX D

Data

)

Specific Heats (16)

$$CP_W = 32.436 \text{ Btu/lbmole-}^oC$$

$$CP_M = 35.958 \text{ Btu/lbmole-}^oC$$

Vapor - Liquid Equilibrium (17)

Vapor Mole% MeOH	Liquid Mole% MeOH	Temp. ^oC
0.0	0.0	100.0
13.4	2.0	96.4
23.0	4.0	93.5
30.4	6.0	91.2
36.5	8.0	89.3
41.8	10.0	87.7
51.7	15.0	84.4
57.9	20.0	81.7
66.5	30.0	78.0
72.9	40.0	75.3
77.9	50.0	73.1
82.5	60.0	71.2
87.0	70.0	69.3
91.5	80.0	67.6
95.8	90.0	66.0
97.9	95.0	65.0
100.0	100.0	64.5

Enthalpy - Concentration (18)

Composition Mole% MeOH	Liquid Enthalpy Btu/lbmole
0.000	3243.69
2.875	3166.60
5.882	3069.78
9.029	2950.74
12.327	2816.10
15.788	2658.67
19.422	2501.61

Composition Mole% MeOH	Liquid Enthalpy Btu/lbmole
23.245	2347.06
27.270	2212.72
31.515	2140.62
35.997	2022.95
40.737	1955.46
45.759	1923.07
51.088	1926.47
56.753	1940.47
62.787	1968.79
69.228	2010.12
83.503	2116.57
100.000	2249.21

The vapor enthalpy is given by the following equation:

$$ENTV = 20596.87 - YN(3641.29) \quad (Btu/lbmole)$$

APPENDIX E-1

The following calculations correct the bottoms d/p cell, which was calibrated for pure water at 60.0°F, to operating conditions. This essentially involves calculating the flow of water through the cell at any temperature for the differential pressure specified and then using this to calculate the corresponding flow of a particular solution for any temperature. Finally, these calculations will also serve as a sample for the reflux and distillate d/p cells.

Maximum Flow of Water at any Temperature

Because the cell is calibrated for 60.0°F or 15.56°C

$$V_{W(15.56)} = k(-(P_1-P)/d_{W(15.56)})^{\frac{1}{2}} \qquad (19)$$

where k is a constant independent of temperature or density. Similarly, the flow of water at a temperature T is equal to

$$V_{W(T)} = k(-(P_1-P)/d_{W(T)})^{\frac{1}{2}}$$

Because the term $(-(P_1-P))$, which is the differential pressure, is the same for both equations, it can be eliminated as follows:

$$\frac{V_{W(T)}}{V_{W(15.56)}} = \frac{k(-(P_1-P)/d_{W(T)})^{\frac{1}{2}}}{k(-(P_1-P)/d_{W(15.56)})^{\frac{1}{2}}}$$

$$\frac{V_{W(T)}}{V_{W(15.56)}} = \frac{(1/d_{W(T)})^{\frac{1}{2}}}{(1/d_{W(15.56)})^{\frac{1}{2}}} = \left[\frac{d_{W(15.56)}}{d_{W(T)}}\right]^{\frac{1}{2}}$$

The cell had been calibrated for

$$V_{W(15.56)} = 1.0 \text{ gallons per minute}$$

$$d_{W(15.56)} = 0.99905 \text{ grams per cubic centimeter}$$

By substituting these values into the above equation, the
maximum flow of water through the cell at any temperature
is then defined as

$$V_{W(T)} = 1.0(0.99905/d_{W(T)})^{\frac{1}{2}}$$

Maximum Flow of a Solution at any Temperature

Once again, the flow of water at a temperature T is

$$V_{W(T)} = k(-(P_1-P)/d_{W(T)})^{\frac{1}{2}}$$

and similarly, the flow of a solution is

$$V_{SOL(T)} = k(-(P_1-P)/d_{SOL(T)})^{\frac{1}{2}}$$

The next step is to eliminate the differential pressure
term and then substitute into this equation the value for
the maximum flow of water defined above.

$$\frac{V_{SOL(T)}}{V_{W(T)}} = \frac{k(-(P_1-P)/d_{SOL(T)})^{\frac{1}{2}}}{k(-(P_1-P)/d_{W(T)})^{\frac{1}{2}}} = \left[\frac{d_{W(T)}}{d_{SOL(T)}}\right]^{\frac{1}{2}}$$

After substituting for $V_{W(T)}$

$$V_{SOL(T)} = \left[\frac{0.99905}{d_{W(T)}}\right]^{\frac{1}{2}} \left[\frac{d_{W(T)}}{d_{SOL(T)}}\right]^{\frac{1}{2}}$$

$$V_{SOL(T)} = \left[\frac{0.99905}{d_{SOL(T)}}\right]^{\frac{1}{2}}$$

APPENDIX E-2

The steam d/p cell was calibrated for steam entering at 30.0 psig with a maximum flow of 3.0 pounds per minute. However, when the steam entering has a different pressure, the maximum flow changes and must be accounted for.

The mass flowrate through the orifice is given by the following equation, which is applicable both to incompressible fluids and to ideal gases:

$$w_S = k(2g_c(P-P_1)d_S)^{\frac{1}{2}}$$

$$w_S^2 = 2g_c k^2(P-P_1)d_S$$

The differential pressure term $(P-P_1)$ does not change and therefore

$$P-P_1 = \left[\frac{w_S^2}{2g_c k^2 d_S}\right]_{30\ \text{psig}} = \left[\frac{w_S^2}{2g_c k^2 d_S}\right]_{P_2}$$

The factor $(1/2g_c k^2)$ is independent of the pressure and can thus be canceled from each term.

$$P-P_1 = \left[\frac{w_S^2}{d_S}\right]_{30\ \text{psig}} = \left[\frac{w_S^2}{d_S}\right]_{P_2}$$

Now, by assuming that the steam is an ideal gas,

$$PV = nRT$$

$$PV = (m/M)RT$$

$$d = m/V = PM/RT$$

Substituting this into the equation

$$\left[\frac{w_S^2}{PM/RT}\right]_{30\text{ psig}} = \left[\frac{w_S^2}{PM/RT}\right]_{P_2}$$

Once again, the factor $(1/(M/RT))$ is independent of the pressure and can be canceled. Therefore

$$\left[\frac{w_S^2}{P}\right]_{30\text{ psig}} = \left[\frac{w_S^2}{P}\right]_{P_2}$$

Originally the cell was calibrated for

$$w_S = 3.0 \text{ pounds per minute}$$

$$P = 30.0 \text{ psig} = 44.7 \text{ psia}$$

Finally

$$\frac{w_S^2}{P_2} = \frac{(3.0)^2}{44.7}$$

and

$$w_S = 3.0(P_2/44.7)^{\frac{1}{2}}$$

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