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Effect of hole size on plate efficiency in a perforated plate distillation column

Frank Leonard Kuchinski

Lehigh University

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EFFECT OF HOLE SIZE
ON PLATE EFFICIENCY
IN A
PERFORATED PLATE DISTILLATION COLUMN

by

Frank Leonard Kuchinski

A THESIS

Presented to the Graduate Faculty
of Lehigh University
in Candidacy for the Degree of
Master of Science

Lehigh University
Bethlehem, Pennsylvania

1955

I

CERTIFICATE OF APPROVAL

DEPARTMENT OF CHEMICAL
ENGINEERING, PRINCETON UNI.
A. M.
MASTER OF SCIENCE IN CHEMICAL ENGINEERING

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

Oct 11, 1955

Date

L.C. Wenzel

Professor in Charge

Approved by Head of Department

Alan S. Post

Head of the Department

Approved and Recommended
for Graduate Study in the
Department of Chemical Engineering
Princeton University

Approved, Virginia Tech
Chemical Engineering Department

CE

I

II

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In however has been of much aid in
carrying out the experiments and to fulfilling certain
unpublished information which would be
difficult to obtain.

Opinions of members of

the

Department of Physics

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library staff. The assistance of W. Szulborski in the
machine and shop work required in the construction of the
apparatus is also acknowledged.

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INTRODUCTION

The objective of this research was to study the
relationship between column efficiency and flow of liquid.
The effect of various operating conditions on column efficiency
was determined by varying the flow rate of liquid and
the number of trays. The results show that column efficiency
is affected by the flow rate of liquid and the number of trays.
The relationship between column efficiency and flow rate
is non-linear, showing a maximum efficiency at a certain
flow rate. The efficiency decreases as the flow rate increases
above this optimum value.

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ATMOSPHERIC FLOW ADJUSTMENT

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(b) (5)(C) STATEMENT TO READER

OFF DRAFT

(b) (5)(C) CONFIDENTIAL

16 * In the original version of this document, the following information was also:
24 * Murphy plate efficiency was determined at
32 * several hole diameter conditions. The results were
40 * similar and showed little difference between the
00 * different hole sizes.

ABSTRACT

This report presents the results of distillation runs made in an effort to determine the effect of plate hole size, 1/4, 3/16, and 1/8 inch diameter on Murphree plate efficiency at variable mass flow rate. The system methyl alcohol-water was studied. In the distillation runs, all salient plate design variables and experimental operating conditions were maintained constant.

Mass flow rates ranged from 50-200 lb/hr-ft², 100-170 lb/hr-ft², and 130-150 lb/hr-ft² for plates having 1/4, 3/16, and 1/8 inch hole diameters, respectively.

Results show no observed effect of hole diameter or mass flow rate on Murphree plate efficiency for plates 1 and 2 within the range of data obtained in this study. However, for the bottom plate, 3, mass flow rate constant, Murphree plate efficiency increased from 44 percent for the 1/4 inch hole diameter plates to 96 percent for the 1/8 inch hole diameter plate indicating higher plate efficiency for smaller hole diameter perforations.

Correlation of average dry and wet pressure drop data indicate the equation $P = R (P + S)$ to be accurate within 5 percent for predicting total plate pressure drop.

INTRODUCTION

No Hallidays or references were made to the design and construction of perforated plate distillation columns. The author has been able to find no general information on the subject. In the literature, however, there is some information on the use of perforated plates in bubble-cap columns. In 1947, Mayfield et al.⁽⁸⁾ studied the mechanical design of bubble-cap trays and found that the efficiency of the tray was not affected by the use of perforated plates. They also found that the use of perforated plates did not affect the efficiency of bubble-cap trays. In 1948, Ragatz⁽¹¹⁾ studied the effect of perforated plates on the efficiency of bubble-cap trays. He found that the efficiency of bubble-cap trays was not affected by the use of perforated plates. In 1950, Marsh⁽⁷⁾ studied the effect of perforated plates on the efficiency of bubble-cap trays. He found that the efficiency of bubble-cap trays was not affected by the use of perforated plates.

INTRODUCTION

Recent research studies made on perforated plate-distillation columns have clearly indicated the numerous advantages this type of tray offers as compared to the conventional bubble-cap plate column. The misbelief of the overall inferiority of the perforated tray is partially attributed to conclusions based on meager design information previously contained in the literature. Chief among these misbeliefs were the limited range of vapor and liquid flows permissible, general lower plate efficiency, and difficulty of operating the column.

Results of Mayfield et al⁽⁸⁾, Marsh⁽⁷⁾, Ragatz⁽¹¹⁾ have shown perforated trays are more economical and more efficient for many applications as compared to bubble-cap plates. These studies have also resulted in data whereby the design of the perforated plate column can be made with more confidence.

The purpose of this investigation was the design and construction of a perforated plate distillation column with the object of studying the effect of changes in mechanical design features of a perforated plate on plate efficiency. Since the number of mechanical design features which could be studied are rather numerous it is the scope of this report to study the effect of perforation size, 1/4, 3/16, and 1/8 inch diameter, on plate efficiency while maintaining

INTRODUCTION

Before proceeding to other variables dependent upon
other plate variables, it is necessary to make a few remarks
concerning the selection of the system to be studied. It is
desirable to have a system which is simple and has
well-defined equilibrium constants. It is also desirable
that the system be one in which the effect of concentration
changes can be easily observed and the equilibrium constant
can be determined with reasonable accuracy. The system
selected for this study is methyl alcohol and water. This
system has been selected because it is simple and has well-
defined equilibrium constants.

(A) $\text{CH}_3\text{OH} + \text{H}_2\text{O} \rightleftharpoons (\text{C}) \text{H}_3\text{O}^+ + (\text{D}) \text{OH}^-$ Equilibrium Constants
In this system there are two equilibrium constants which are
dependent on the concentration of the equilibrium. The
first equilibrium constant is the equilibrium constant for
the dissociation of methyl alcohol and the second is the
equilibrium constant for the dissociation of water.

It is the purpose of this paper to determine the
equilibrium constants for the dissociation of methyl
alcohol and water and to compare the results with those
obtained by other workers. The equilibrium constants
for the dissociation of methyl alcohol have been
determined by several methods. The first method
is the use of the Raoult's law equation. The second
method is the use of the van't Hoff equation. The
third method is the use of the osmotic pressure
equation. The fourth method is the use of the
Henry's law equation. The fifth method is the
use of the Raoult's law equation for the equilibrium
constant of the dissociation of water. The
sixth method is the use of the van't Hoff equation
for the equilibrium constant of the dissociation
of water. The seventh method is the use of the
Henry's law equation for the equilibrium constant
of the dissociation of water. The eighth method
is the use of the Raoult's law equation for the
equilibrium constant of the dissociation of water.

other plate variables constant. Methyl alcohol and water
was the system selected to be studied.

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selunda ad od hodeles medaya enj saw

LITERATURE REVIEW

Distillation is the separation of the more volatile component of a liquid mixture by a series of vaporizations and subsequent condensations of the more volatile component. Rectification of the more volatile component takes place in the following manner. Vapor in a column passes counter-currently to liquid reflux on a tray and condenses giving up its heat of condensation to the liquid on the tray, resulting in the vaporization of the lower boiling component in the liquid. These series of vaporizations and condensations take place on each tray of the distillation column until finally the vapors leaving the top tray are totally condensed in a condenser. Part of the distillate from the condenser is usually removed as product and the remainder returned to the top of the column as liquid reflux.

The process of distillation is usually performed in a bubble cap, perforated plate or packed type column. A perforated plate differs very little from a bubble cap plate in its operation. Both have the primary function of more efficiently effecting mass transfer of the lower boiling component from the liquid to the vapor and the higher boiling component from the rising vapor to the descending liquid. In a perforated plate vapor bubbles through a large number of small holes through a liquid whereas in a bubble-cap plate vapor bubbles through a series of caps containing slots

PERFORATED PLATES

distillation over and to hold liquid submerged in the liquid above it; otherwise liquid will drain to the plate below. In the perforated plate the height of the liquid on the tray is governed by the weir height; in the bubble cap plate liquid height is maintained by the height of the downcomer pipe and does not drain the tray of liquid in the event vapor velocity is not great enough to support it. It is obvious that a minimum gas velocity (weep point) and pressure drop exists, depending on the perforated plate design and operating conditions, below which efficient operation of the perforated plate ceases. For perforated and bubble-cap plates an upper limiting vapor velocity (flood point) exists where the liquid seal in the downcomer is broken resulting in the flooding of the plate and column with liquid.

In the usual procedure for designing a distillation column for a desired rectification, the theoretical (or perfect) plates can be readily calculated by known methods^(10,4). The design engineer is handicapped by the unknown relationship existing between the theoretical and actual plates, i.e., overall plate efficiency of the column.

Plate efficiency is usually expressed as overall column efficiency, and Murphree Plate efficiency. The overall column efficiency is defined as the ratio of the number of theoretical plates necessary to produce a given separation to the number of actual plates used to achieve the same separation.

The effect of liquid holdup may be simplified by assuming equilibrium between the vapor leaving the plate and the liquid leaving the plate. If the vapor leaving the plate is in equilibrium with the liquid leaving the plate, the Murphree efficiency is given by the equation:

$$E_m = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} \quad (100)$$

where: E_m = Murphree plate efficiency (percent)
 y_n = mol fraction of vapor leaving plate n
 y_{n-1} = mol fraction of vapor leaving plate n-1
 y_n^* = mol fraction of vapor in equilibrium with liquid leaving plate n

Murphree plate efficiencies more than 100 percent can be obtained when the vapor leaving the plate is richer than the vapor in equilibrium with the liquid leaving the plate. This is possible, especially in large diameter columns, if the vapor leaving the plate is incompletely mixed and is in equilibrium with the liquid leaving the liquid on the inlet weir.

Two of the major factors affecting overall plate efficiency are the mechanical design features of the plate

under similar reflux conditions. The Murphree efficiency is defined by the ratio of the actual change in composition, which the vapor undergoes in passing through a plate to the change in composition which it would undergo if it left the plate in equilibrium with the liquid which leaves the plate. A method of relating Murphree plate efficiency to overall column efficiency is given by Perry⁽¹⁰⁾.

In equation form Murphree plate efficiency is expressed as follows:

$$E_m = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} \quad (100)$$

where: E_m = Murphree plate efficiency (percent)
 y_n = mol fraction of vapor leaving plate n
 y_{n-1} = mol fraction of vapor leaving plate n-1
 y_n^* = mol fraction of vapor in equilibrium with liquid leaving plate n

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Two of the major factors affecting overall plate efficiency are the mechanical design features of the plate

$$(CCF) \quad \frac{I - e^X}{I + e^X} = \frac{e^Y}{e^Y + e^X}$$

(திருவோர்) சார்பிலை எடுத்து விடப்பட்டு விடும் நிலையிலே மூன்றாவது கோட்டை கோட்டை என்று அழைகின்றன. இது மூன்றாவது கோட்டை என்று அழைகின்றன. இது மூன்றாவது கோட்டை என்று அழைகின்றன.

and column, and the physical properties of the mixture being distilled.

Various methods of correlating the physical properties of the mixture with plate efficiency have been presented in the literature. The bases of these correlations are rather varied indicating the complexity of predicting overall plate efficiency. O'Connell's⁽⁴⁾ correlation uses the product of the liquid viscosity and the relative volatility at the average column temperature; Drickamer and Bradford⁽⁴⁾ correlation is based on the viscosity of the feed; Geddes⁽⁴⁾ uses surface tension, density of liquid, density of vapor and diffusivities of the phases in the mixtures as well as the area of the bubble-cap slot. Gunnes⁽⁴⁾ uses vapor pressure of feed stock, i.e., properties of the feed. The most recent of these correlations deserving further study is the method of Gerster et al⁽³⁾ using separate gas and liquid film resistances to predict plate efficiency.

Considerable progress has been made recently in determining the effect of the various mechanical design features of a perforated plate. Umholtz⁽¹³⁾ in his study of overall plate efficiency in a 1.83 and 3 inch diameter perforated plate distillation columns has studied the effect of free space area, percent downcomer area, pitch/diameter ratio of perforations, and perforation size. Nandi and Karim⁽⁹⁾ using a 2-1/4 inch diameter column studied the effect of

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*Bellis est
regarding Isolating est said element in addition consider
the necessary need over consideration of the data obtained est to said
one about which was to record est . Consideration est all
that we will have to take into account that below studies
of air and water (1) different properties such as
and the relationship which est for viscosity which will be
said (2) both of the correlated consideration results obtained
and (3) results of which est no found at normal
but does not appear difficult to believe isolated on other
and a. If we are consider est of interest will be subdivided
consideration which will be (4) result . It is considered not be able
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be bottom and of the bottom previous isolating was made to
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concerning the influence of
in which case first and second - I think of
English Isolating theory est to justify this opinion est
to which aid of (6) action . We do not know as to how many
specifications for a 6 in. dia. is all considered while there is
no justification and purpose sufficient to be justified
other consideration there seems to be some point
(6) which the small area perforated has consideration to
justify est believe that reference does not in which
to justify est believe that reference does not in which

distillation rate, free space area, plate spacing, weir height,
and perforation size on overall plate efficiency. Arnold
et al⁽¹⁾ using a 15 inch diameter column studied pressure
drop behavior of perforated trays (using air and water) by
varying weir height, perforation size, free space area, and
L/G ratios. Mayfield et al⁽⁸⁾ using a 6 inch diameter column
studied pressure drop behavior of perforated plates (using
air) by varying the perforation size. In addition using a
6.5 foot diameter column data were obtained (using air-water)
relating pressure drop, weir height, and L/G ratios.

In spite of these recent studies previously mentioned,
attempts to correlate mechanical plate design features with
the physical properties of the liquid in an effort to predict
plate efficiency have as yet not been successful. The main
reason being the complex nature of mass transfer between the
vapor and liquid phases as a result of the changing physical
phenomena of the vapor-liquid interaction as the mass flow
rate is varied.

This vapor-liquid interaction can best be described
as the foaming or frothing which a liquid undergoes on and
above a plate as the mass flow rate is varied. Visual ob-
servation of a distillation tray in stable operation indicates
three zones of different foam density. A highly agitated
foam layer through which bubbles form and rise through the
liquid, a zone of dense droplets, and finally a zone composed
of a fine mist.

interior surface area, some vapor will pass through the foam. Vapor leaving the foam may be due to entrainment by entrained bubbles entering from the liquid (1). In this case (under low flow regime) vapor formation is believed to occur due to entrainment of vapor bubbles within the liquid (2). In the intermediate regime (high liquid holdup) vapor formation is believed due primarily to entrainment of vapor bubbles within the liquid (3). In the high flow regime (high liquid holdup) entrainment of vapor bubbles is believed to occur due to entrainment of vapor bubbles within the liquid (4).

Because of the difficulty of defining and defining the boundaries of the various regions, it is difficult to calculate the amount of vapor leaving the system. Information need for determining overall plate efficiency and liquid holdup and vapor holdup are required. However, if the overall plate efficiency and liquid holdup can be determined, the vapor holdup can be calculated.

Mass transfer in these three zones is dependent upon the vapor and liquid film resistances, concentration gradient and surface area between the two phases. Gerster et al⁽³⁾ has attempted to relate foam height with mass flow rate and the overall mass transfer coefficient in an effort to predict overall plate efficiency. West et al⁽¹⁴⁾ elaborating on the equation proposed by Gerster et al⁽³⁾ expressed the foam height in terms of fraction voids in the foam. In addition interfacial area was expressed as a function of volume of foam. It is believed that if plate efficiency data for a variety of liquids be obtained as a function of mass flow rate and utilizing the proposed methods of Gerster et al and West et al plate efficiency for any system could be calculated with relative ease and accuracy.

theoretical air space count effect on column performance
no significant difference with liquid film mass transfer rate found
between 1/4 inch and 3/16 inch diameter plates. The results
were similar to those obtained by Higuchi and (8) Wolf
from the same type of test system. The effect of bed height was
studied at two different liquid heights and (9) the effect
(M) of the feed concentration and column diameter on
desorption (1). It is proposed that desorption resistance will be
inversely proportional to column height and the effect
of liquid height above column top varied in a manner which
is independent of the column size and the operating
conditions. The hypothesis of this study is that the
desorption rate will increase with column height. It is also
proposed to study the reaction with ammonia for other
liquids. The experimental conditions used in the work
are given on the following pages for this technique.

EXPERIMENTAL PROCEDURE

The object of this study was the design and construction of a perforated plate distillation column to determine the effect of plate hole size, 1/4, 3/16, and 1/8 inch diameter on Murphree plate efficiency. Methyl alcohol and water was the system selected to be studied in lieu of published equilibrium, density and refractive index data which was readily available.

Plans were to hold all salient plate design variables constant, except hole size, and perform distillation runs under constant operating conditions while varying the mass flow rate through the column. Plate design variables which efficiency is believed to be affected by and which were maintained constant are:

1. Weir height ----- 1/2 inch
2. Weir length ----- 5 inches
3. Column diameter, I.D. ----- 8.25 inches
4. Plate spacing ----- 9.5 inches
5. Perforated plate thickness --- .029 inches
6. Pitch/diameter of holes ----- 3.8
7. Percent downcomer area ----- 0.826
8. Baffle arrangement ----- 3/8 inch above
perforated plate and 5/16 inches from
weir. Height of baffle 5 inches.
9. Percent free space area ----- 1.17

Conditions at which the experiments were performed are: concentration of kettle charge 25 percent methanol-75 percent (weight ~~per water~~), atmospheric pressure, and constant L/G ratio, i.e., total reflux. The temperature of the reflux was maintained constant $+0.5^{\circ}\text{F}$ and 2-3 degrees below the boiling point of methanol.

DISCUSSION OF EXPERIMENTAL

Since both methods used were found to be feasible with
the simple apparatus available, the following
will describe the methods used to determine the
relative efficiency of plate distillation and column
distillation with respect to yield and efficiency.

Plate Distillation Method

Plates used for distilling the blend of water and
methanol were of a certain size to insure maximum efficiency
of the distillation unit. These plates were made of
stainless steel. A stainless steel wire mesh was used to support
the plates and to prevent them from falling through the
bottom of the column.

Column Distillation Method

For the column distillation method the following
procedure was followed:
1. A column was constructed of glass tubing.
2. The column was packed with stainless steel wire
mesh supported by stainless steel wire.
3. The column was heated with an electrical
heating coil.
4. The column was connected to a condenser.
5. The condenser was connected to a receiver.
6. The receiver was connected to a refractometer.
7. The refractometer was connected to a recorder.
8. The recorder was connected to a pump.
9. The pump was connected to a methanol tank.
10. The tank was connected to the column.
11. The entire assembly was connected to a vacuum
system.

The column was heated to a temperature of 100° C.
The refractometer was calibrated to read 100% methanol.
The column was heated to 100° C. and the
refractometer was calibrated to read 100% methanol.
The column was heated to 100° C. and the
refractometer was calibrated to read 100% methanol.
The column was heated to 100° C. and the
refractometer was calibrated to read 100% methanol.

The glass portions of the distillation column
were wrapped with two layers of glass wool asbestos, with
the exception of approximately four square inches of the
front of each glass section. This unwrapped section was
covered with a piece of plexiglass through which plate
distillation phenomena could be easily observed.

Samples of liquid and vapor leaving each plate
were taken after steady state column operating conditions
were reached. Using a refractometer all samples were ana-
lyzed for percent methanol, from these data Murphree plate
efficiency could be readily calculated.

mer too difficult to avoid each off
the surfaces too easily to prevent any appreciable amount
of sedimentation and volatilization of poisons and
not to admit air and moisture which may be found
in most backgrounds and which may be due to dust
and the nature of the materials to which it may have
been exposed. It is also important to maintain
the apparatus clean and free from organic material
which may be present in the sample to be tested.
The apparatus must be small to a form
which will not interfere with the sample and the results will
not be affected by the size of the apparatus. The apparatus must
be able to withstand temperatures up to 100° C. without
deformation or damage.

APPARATUS AND MATERIALS

The apparatus employed in this investigation
consists mainly of the perforated plate distillation column
and steam boiler. Only essential information relative to
the auxiliary apparatus will be listed below:

Pyrex Glass Cylinders. Four required, 8.25 inch inside
diameter, 0.25 inches thick, 9.5 inches in height. Originally
purchased from Hawshaw Scientific Glass Co. as battery jars.
Battery jars were cut to size by Pittsburgh Plate & Glass Co.,
Allentown, Pa.

Brass Plate Holders. Five required, 12 x 12 x 3/8
inches. Brass sheet 0.029 inches thick, used for the distil-
lation plate. Purchased from Whitehead Metals Co., Philadelphia,
Pa.

Plate Gaskets. Teflon impregnated asbestos-3 ply, 9
inch outside diameter, 8 inch inside diameter. Manufactured
by Johns-Manville Corp., Manville, N. J.

Distillation Kettle. Glass lined, 18 inch I.D., 25
inches in height, 75 psig jacket pressure, total heating
surface 10 ft². Manufactured by Pflauder & Co., Rochester, N.Y.

Refractometer. Abbe 56, Manufactured by Baush & Lomb,
Rochester, N. Y.

Steam Boiler. Gas fired, 5 H.P. Mfgs. Steam Rating.
Used to supply steam to jacket of distillation kettle. Manu-
factured by Mears-Kane-Ofeldt, Inc., Philadelphia, Pa.

Methanol, Technical grade. Purchased from Lehigh
Valley Chemical Co., Easton, Pa.

- 21 -

STANDARD GRADE

moisture content and no benzene, carbon dioxide and

water content not exceeding 10% of the total weight

and available methanol having a specific gravity

of 0.790 at 60° F. and a flash point of 100° F.

obtained from a benzene and methanol mixture

containing 10% benzene and 90% methanol.

Specific gravity at 60° F. is 0.790.

Flash point is 100° F. or higher.

Boiling point

at 140° F. is 100° F. or higher.

Water content not exceeding 10% of the total weight

and available methanol having a specific gravity

of 0.790 at 60° F. and a flash point of 100° F.

obtained from a benzene and methanol mixture

containing 10% benzene and 90% methanol.

Specific gravity at 60° F. is 0.790.

Flash point is 100° F. or higher.

Water content not exceeding 10% of the total weight

and available methanol having a specific gravity

of 0.790 at 60° F. and a flash point of 100° F.

obtained from a benzene and methanol mixture

containing 10% benzene and 90%

water content not exceeding 10% of the total weight

and available methanol having a specific gravity

Digested work breakdown: Primary Investigation → Perforated Plate Column Design → Flow Diagram

PERFORATED PLATE COLUMN DESIGN

The ideal column for this experimental study should be simple in construction to allow frequent changes in plate design, small enough to obtain reasonable boilup rates, permit close observation of the distillation process, and yet not be too costly. In Figure I, page 15, is shown a flow diagram and Figure II, page 16, a photograph of the perforated plate column and apparatus used in the experimental study.

SELECTION OF COLUMN SIZE

A four-plate column (2), 8-1/4 inch I.D. pyrex glass cylinders, 9-1/4 inches in height was selected and found to be commensurate with the boilup expected using a steam jacketed glass-lined kettle and heat available from the steam main.

CONDENSER SPECIFICATIONS

On the basis of the boilup of methanol expected from a 5 HP steam boiler and using a steam jacketed glass-lined kettle a condenser was designed. The specifications of this condenser are as follows: horizontal, single pass, counterflow, with vapor condensing in the shell and water flowing through the copper tubes. Tubes, eight 5/8 inch O.D., shell I.D. 2-3/4 inches, tube length 32 inches, total tube surface area 3.50 ft².

REFLUX TEMPERATURE CONTROL SYSTEM

An automatic reflux temperature control system was

MELTING EQUIMENT AND OPERATING

Before starting up equipment, turn on electric power switch.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

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Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

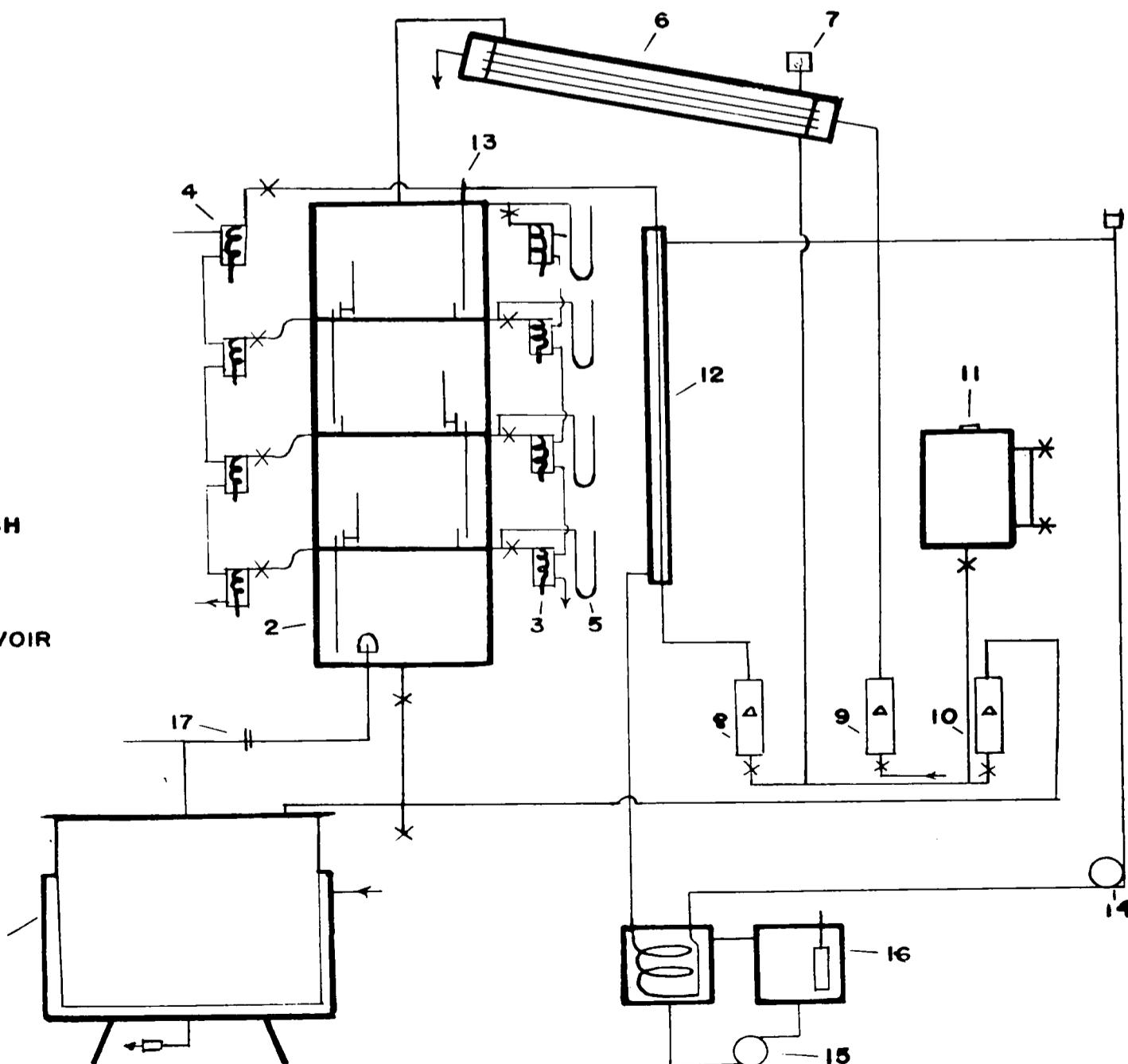
Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

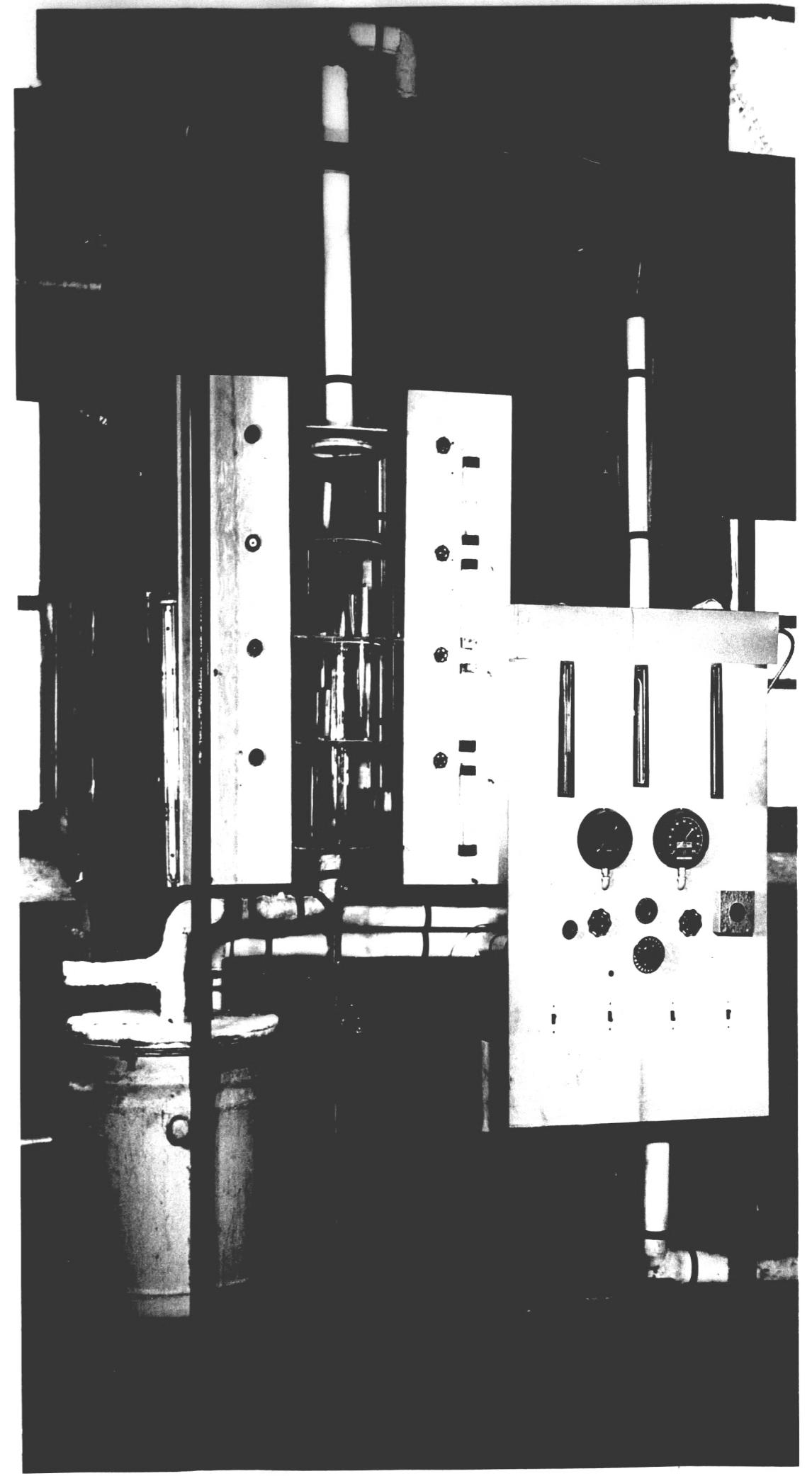
Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

Turn on electric power switch to 110 volt, 60 cycle, 50° C. or less.

FIGURE I. DIAGRAM OF EQUIPMENT





designed to maintain the reflux temperature $\pm 0.5^{\circ}\text{F}$ and to within $2\text{--}5^{\circ}\text{F}$ of the boiling point of methanol. Temperature of the reflux was controlled by a fenwall thermoswitch located at the top of the column. By means of the fenwall thermoswitch (13) and a relay, a pump and an immersion heater (1000 watts) were turned on. The reservoir pump (15) recycled hot water from the reflux heater reservoir to a constant temperature reservoir in which was located a heat exchanger. Another pump (14) recycled water through the heat exchanger located in the constant temperature reservoir to the heat exchanger through which the reflux flowed. When the temperature of the reflux was up to the desired temperature, the fenwall thermoswitch through the relay turned the reservoir pump (15) and immersion heater off, the recycle pump being in continual operation throughout the distillation run.

PLATE HOLDER DESIGN

Selection of the 12 x 12 x 3/8 inch brass plate was made on the basis of the ease at which brass could be machined. In the layout of the plate holders, calculations were based on obtaining maximum perforation area. With cross flow of liquid and using weirs, 58.2 percent perforation area was obtained. Figures III and IV, pages 18 and 19, show the details of construction of the five plate holders. Plate holder four is identical to plate holder two and is not shown in the drawings.

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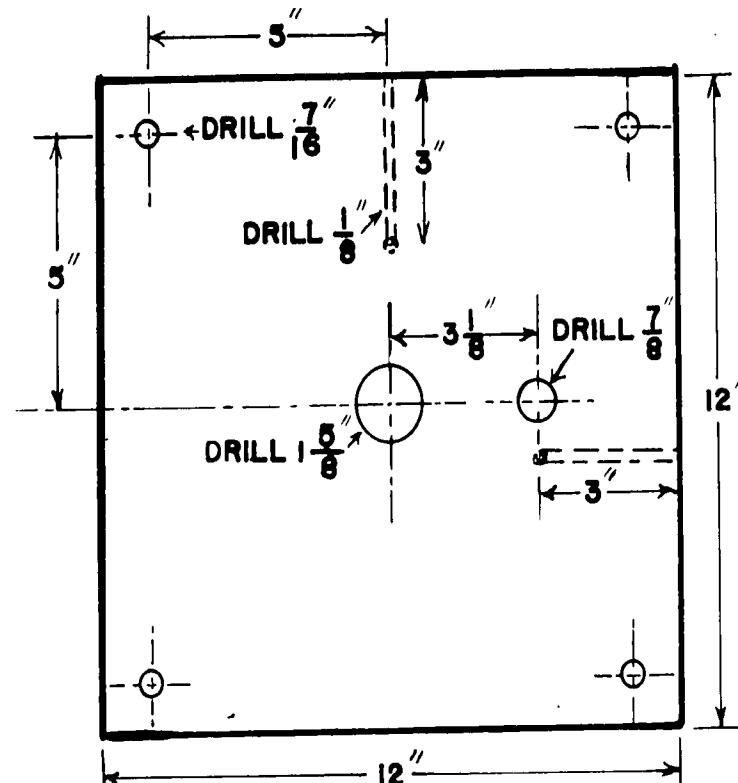


PLATE HOLDER -I

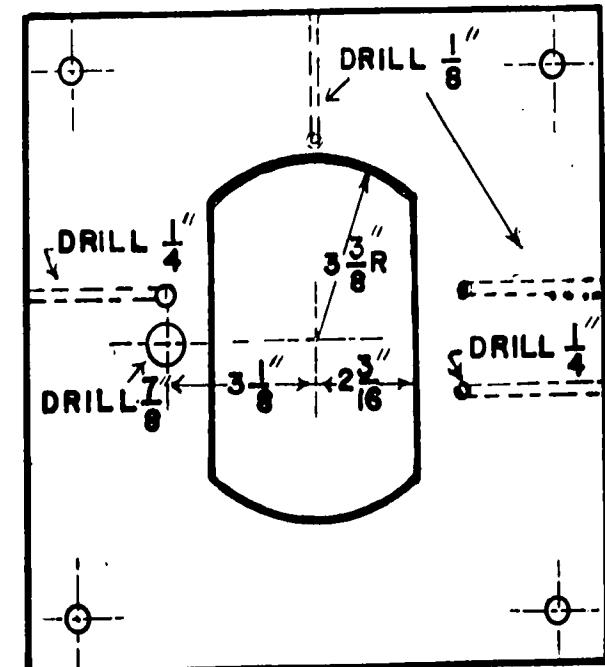


PLATE HOLDER -2

FIGURE III.

SCALE 1 : 4

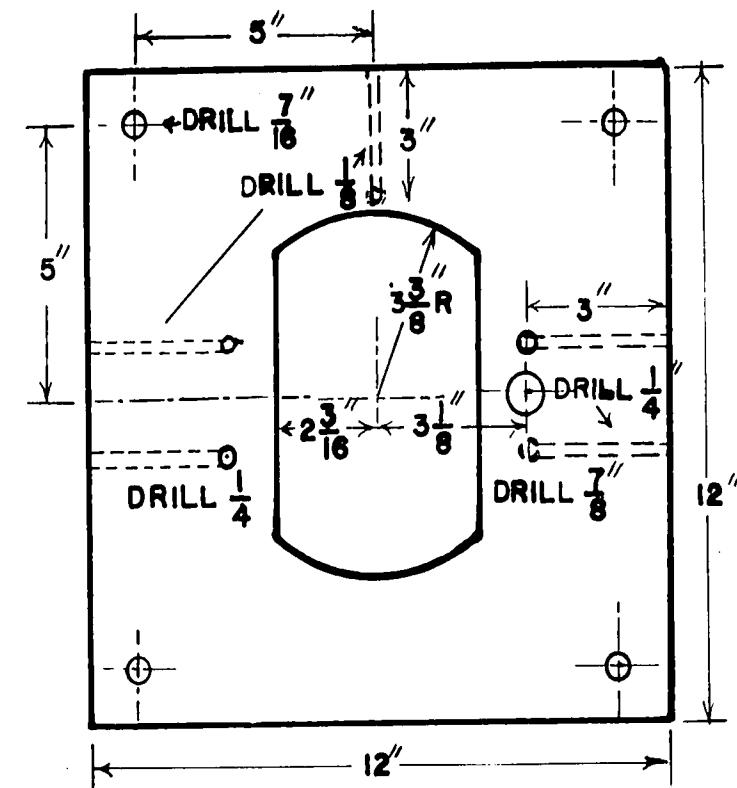


PLATE HOLDER-3

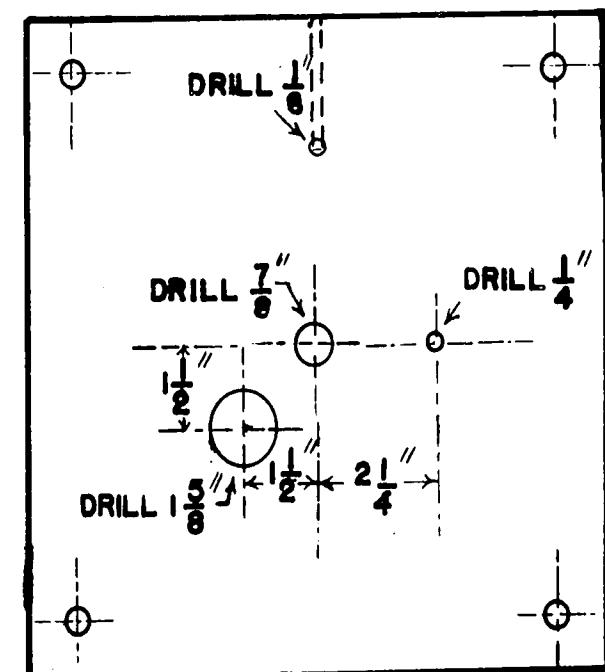


PLATE HOLDER-5

FIGURE IV.

SCALE 1 : 4

PLATE DESIGN

Selection of the percent free space area (1.17) was made by calculating the maximum mass flow rate, G , and computing the pressure drop across each tray by fixing the weir height, weir length, percent downcomer area, and baffle arrangement.

The pressure drop calculation consists of two (main) components, namely, the pressure drop as a result of vapor flowing through the perforations and the height of the liquid on the tray. To the liquid depth above the tray, the height of calculated weir head is added a correction called an "aeration factor"⁽²⁾. The aeration factor being defined as the ratio of the observed pressure drop through the liquid on the tray to the calculated clear liquid on the tray. For purposes of design of this plate an aeration factor of 1.0 was used. Essentially this aeration factor takes into consideration the foaming characteristics of the liquid. To calculate aeration factors, the observed pressure drop through the liquid is obtained as the difference between the total observed pressure drop and the observed dry-tray pressure drop at the same air rate.

The computed pressure drop calculated in the above manner would have to be sufficiently large to overcome the resistance the vapors would be subjected to in passing through the perforations in addition to supporting the liquid above

above, baffle I off, water can be added to section A of tube, a glass stopper being used above and sections C and D being closed off by glass stoppers.

After adding water to section A of tube, a glass stopper being used above, baffle I off, water can be added to section C of tube, a glass stopper being used above and sections A and D being closed off by glass stoppers.

After adding water to section C of tube, a glass stopper being used above, baffle I off, water can be added to section D of tube, a glass stopper being used above and sections A and C being closed off by glass stoppers.

After adding water to section D of tube, a glass stopper being used above, baffle I off,

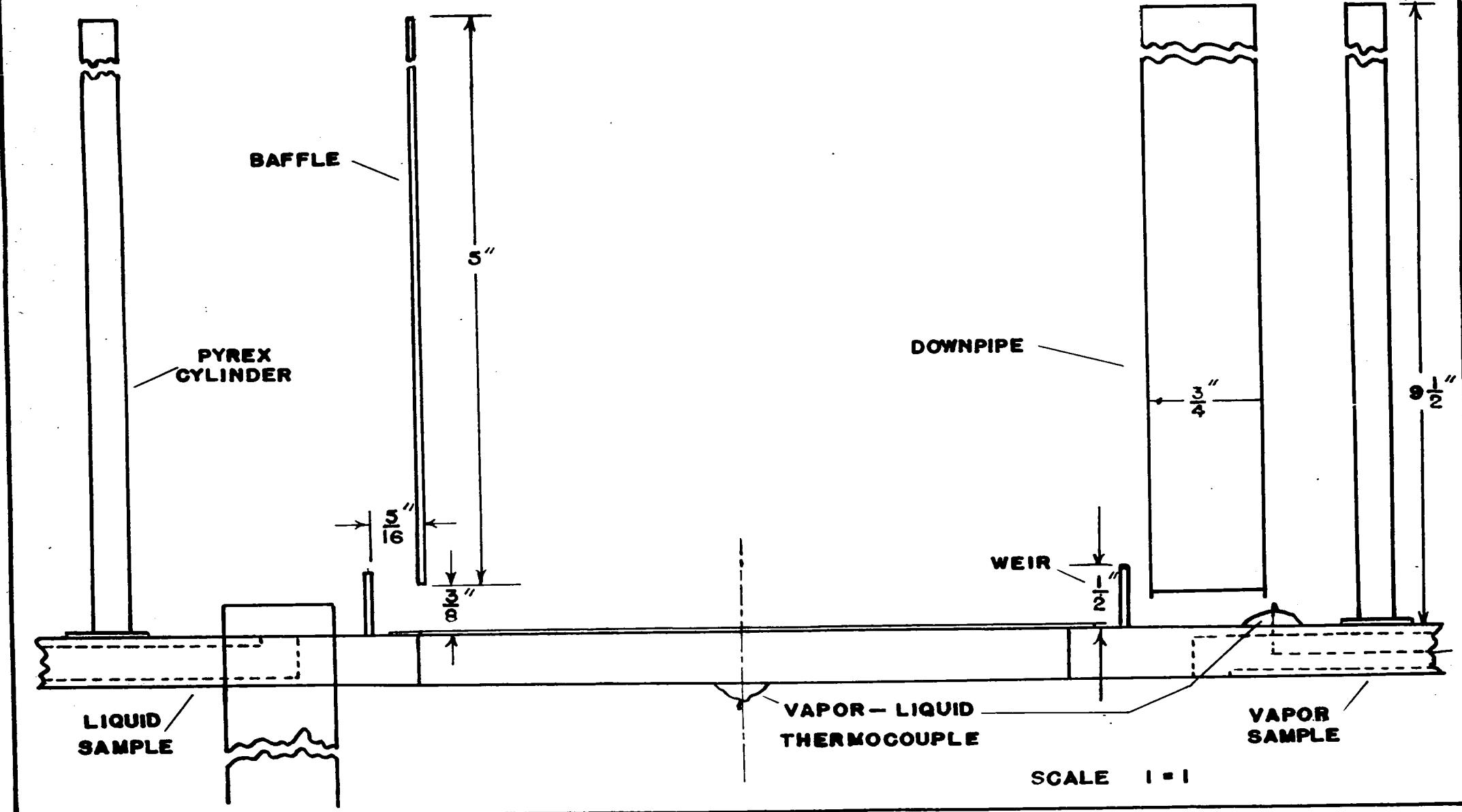
water can be added to section A of tube, a glass stopper being used above and sections B and D being closed off by glass stoppers.

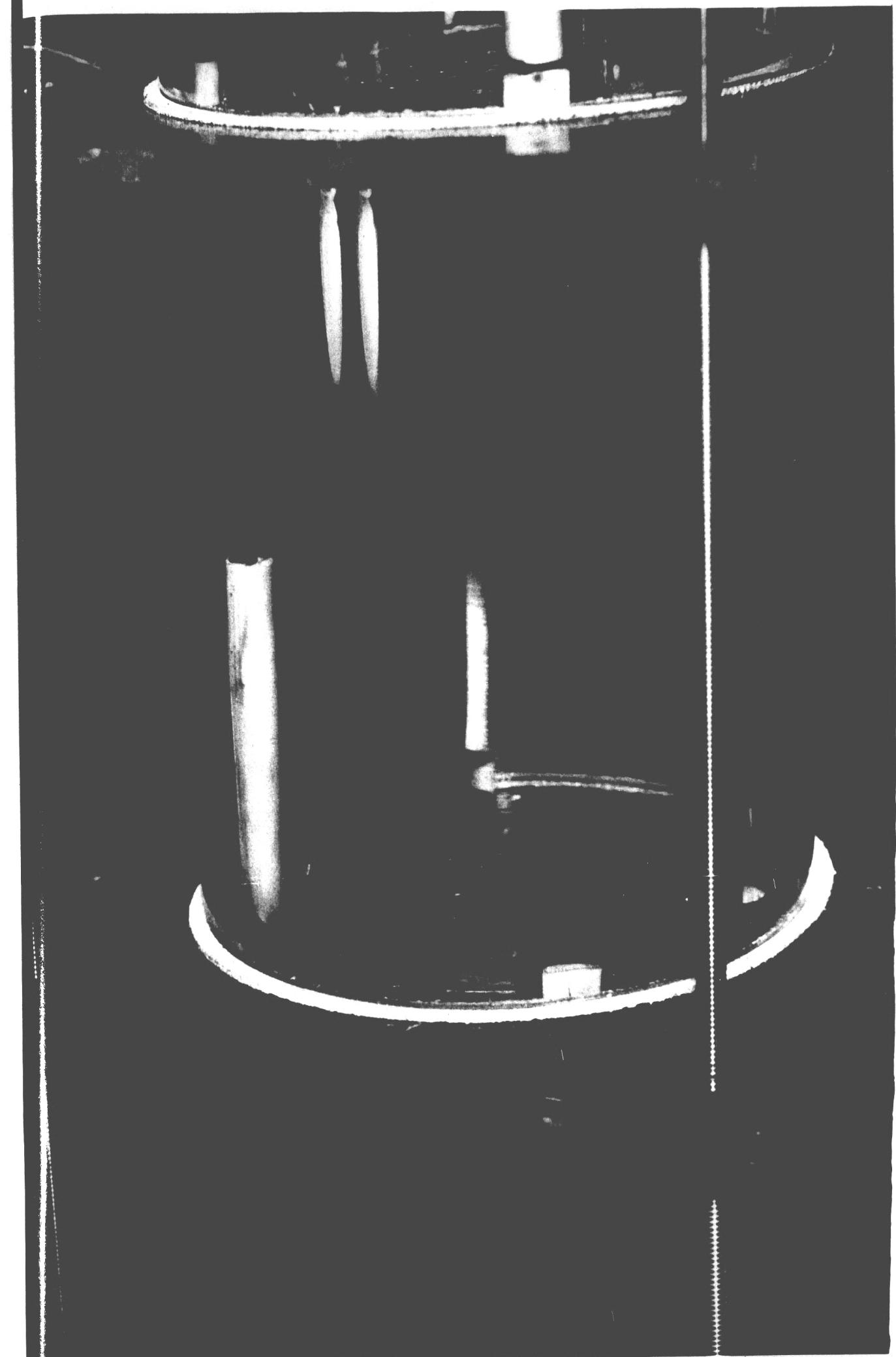
After adding water to section A of tube, a glass stopper being used above and sections B and D being closed off by glass stoppers, baffle I off.

REVERSE STOPPING

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FIGURE V.
PERFORATED PLATE
FRONT VIEW





each perforation. In addition, the upper limit of the pressure drop should be sufficiently low enough that the downcomer backup is not excessive and the column as a result, flooded. Consideration of the hole size was also made as the operating range of the column, i.e., range between the weep and flood point, is considerably reduced with an increase in hole size. In the design of any column it is desirable that the pressure drop be a minimum and yet the range between the weep and flood point be a maximum.

PLATE LAYOUT

The perforated plate was punched with the desired hole diameter punch on equilateral triangular centers with a pitch/diameter hole ratio of 3.8. Calculations for the plate layout are included in the Appendix C , page 41. The perforated plates were soldered to the plate holder. Figure VI, page 22, shows a 3/16 inch hole diameter perforated plate assembly.

VAPOR AND LIQUID SAMPLING SYSTEM

Referring to Figure IV, page 19, showing the details for the perforated plate holders, holes 1/4 inch diameter, were drilled through each side of the 3/8 inch brass plate holders. One hole was drilled through the bottom of the plate for the vapor sample and the other through the top to obtain a liquid sample; refer Figure V. Small condensers

and the kettle were set available at a temperature close
and kept above well insulated and liquid control components
placed to be reusable with the availability of tank oil quaternary ammonium
in a dry case and with a float to indicate bottom level.
and required vapor outlet sampling and liquid control
and insulation system. The sampling and liquid control
and insulation system will be discussed later in this report.
and insulation system will be discussed later in this report.

LIQUID LEVEL

Level control was provided by a float system which is
set up to indicate when liquid level has dropped to a certain
point and to automatically stop the cold water feed to the kettle.
In addition, a float system was provided to indicate when
the kettle charge tank has been filled to a certain point.

LIQUID HOLDUP AND LIQUID FLOW RATE

Level control was provided by a float system which is
set up to indicate when liquid level has dropped to a certain
point and to automatically stop the cold water feed to the kettle.
In addition, a float system was provided to indicate when
the kettle charge tank has been filled to a certain point.

for cooling the liquid sample and condensing the vapor sample
were located at each sampling outlet.

TEMPERATURE MEASUREMENT

Temperature of the liquid and vapor of each plate
in the column were obtained using copper-constantan wire,
an eleven terminal thermocouple selector and a Leeds-Northrup
potentiometer. Referring to Figure V, page 21, the vapor and
liquid thermocouple locations are shown. Holes 1/8 inch
diameter was drilled into the sides of the plate holder,
Figure IV, page 19.

DISTILLATION OR BOILUP RATE CONTROL

Control of boilup rate was accomplished by regu-
lating the steam pressure in the steam jacket of the glass-
lined kettle.

PRESSURE MEASUREMENT

The 1/4 inch holes drilled in plate holders used
for obtaining vapor samples were also used to obtain the
pressure below and above each tray.

MISCELLANEOUS COLUMN FEATURES

A charge tank was installed in which the concen-
tration of the kettle charge could be easily increased or
decreased as desired by regulating flow of the reflux and
product streams.

A valve system was also installed for continuous
distillation studies or for air water studies.

elements which will automatically burn clear and will not gall or
stain the surface due to heat transfer.

TESTING OF ORIFICE

After the nozzle has burned off "a significant"

amount of propellant mass has been added to the nozzle with the
corresponding increase in pressure downstream. In addition, removal of
the nozzle will cause a large amount of combustion products to be
dislodged. It is for this reason that it is recommended that the
nozzle be left in place until the propellant has been added. This
will enable the nozzle to be used again without difficulty.

TESTING OF ORIFICE

TESTING OF ORIFICE

After the nozzle has been removed from the test fixture

and if it is to be used again, it must be cleaned with 100%
alcohol.

TESTING OF ORIFICE

Before the nozzle is reinstalled, it is recommended that

one minute of the nozzle be cleaned with a solution of 100%
alcohol and water.

TESTING OF ORIFICE

After one minute of cleaning, the nozzle

is to be cleaned with 100% alcohol and no more
than one minute. It is recommended that the nozzle be cleaned
with a solution of 100% alcohol and water.

After the nozzle has been cleaned, it is recommended that

one minute of the nozzle be cleaned with a solution of 100%

VAPOR RATE MEASUREMENT

A sharp edge orifice, flange type, 0.75 inch hole
diameter was designed and calibrated. The calibration
curve for this orifice is included in Appendix E, Figure
VIII.

EXPERIMENTAL DATA

elod dont 87.0 kgd vanele ecotite upbe grada A
coldwille 87.0 bedwille fire boulders new material
owen & williams 87.0 boulders of ecotite and rock

100%

DATA AND RESULTS

The distillation runs and sample data obtained in this study are listed in Table IV of the Appendix F.

WET AND DRY PLATE PRESSURE DROP

Prior to starting the distillation runs using the binary, methyl alcohol and water, data were taken to obtain the resistance (pressure drop in inches of water), the 1/4 and 3/16 inch holes offered to the flow of gas. This was accomplished by recording the pressure below and above each plate when air at varying rates was pumped through the column. The difference in pressure was recorded as the pressure drop across the plate. A summary of these data recorded as dry plate pressure drop is shown in Table I, page 28.

Pressure drop data were also obtained during the distillation runs that were made in the course of the experimental study. These data for 1/8, 3/16, and 1/4 inch holes are recorded as wet plate pressure drop and are listed in Table II, page 29. Wet pressure drop data includes the resistance the gas, methanol vapor, encounters in passing through the holes in addition to supporting the liquid above it.

A graph showing the relationship between dry and wet pressure drop data for the 1/4 and 3/16 inch diameter holes is shown in Figure VII, page 30.

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MURPHREE PLATE EFFICIENCY

In Table III, page 31, is a summary of all distillation runs listing the calculated Murphree plate efficiencies with the corresponding mass flow rate. A sample calculation showing the manner in which the results were calculated from experimental data are given in Appendix C, page 41.

DRY PLATE PRESSURE DROP DATA

To determine the pressure drop across a dry plate, it is necessary to determine the total head loss across the plate. This is done by connecting two static pressure gages across the plate. The pressure drop is then calculated from the difference in pressure across the plate.

The pressure drop across the plate is determined by the formula:

$$\Delta P = \frac{G}{2} \cdot \frac{\rho}{\gamma} \cdot D^2 \cdot C_D$$

where ΔP is the pressure drop across the plate, G is the flow rate, ρ is the density of the fluid, γ is the specific weight of the fluid, and C_D is the coefficient of discharge.

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TABLE I

Summary of Dry Plate Pressure Drop Data for 1/4, 3/16 Inch Diameter Holes Plate Series

No. of Reading	Orifice Reading In.-H ₂ O	P ₁ In.	P ₂ In.	P ₃ Red Oil	P ₄	P ₁ -P ₂ In. H ₂ O	P ₂ -P ₃ In. H ₂ O	P ₃ -P ₄	Avg. P In. H ₂ O	G Lb. Hr-Ft ²	Hole Diameter Inches
1	2.50	2.90	3.35	4.0	4.18	1.33	1.03	1.40	1.25	168	1/4
2	1.95	2.25	2.50	2.90	3.20	0.738	1.18	0.885	0.93	148	1/4
3	1.00	1.20	1.35	1.55	1.70	0.442	0.59	0.442	0.49	107	1/4
4	0.41	0.60	0.61	0.75	0.83	-	0.44	0.222	0.22	68	1/4
5	1.85	2.10	2.40	2.75	3.00	0.885	1.03	0.736	0.88	145	1/4
6	3.80	4.25	4.85	5.53	6.15	1.77	2.01	1.83	1.87	209	1/4
7	0.20	0.30	0.31	0.40	0.44	0.030	0.265	0.127	0.14	48	1/4
8	3.80	4.35	4.90	5.55	6.25	1.62	1.92	2.07	1.87	209	1/4
<hr/>											
1	2.60	3.45	3.90	4.33	4.73	1.33	1.27	1.18	1.26	171	3/16
2	0.58	0.80	0.90	1.00	1.12	0.295	0.295	0.354	0.32	80	3/16
3	0.20	0.33	0.35	0.43	0.45	0.074	0.22	0.074	0.56	48	3/16
4	0.20	0.30	0.35	0.43	0.45	0.148	0.22	0.074	0.15	48	3/16
5	0.78	1.10	1.20	1.35	1.50	0.295	0.443	0.443	0.39	94	3/16
6	1.65	2.30	2.60	2.95	3.28	0.885	1.03	0.974	0.96	137	3/16
7	2.80	3.85	4.38	4.90	5.47	1.565	1.535	1.68	1.62	179	3/16
8	3.25	4.45	5.10	5.70	6.30	1.92	1.77	1.77	1.82	193	3/16
9	0.20	0.30	0.35	0.40	0.45	0.147	0.147	0.147	0.15	48	3/16

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Run No.	Time	Plate				P ₂ -P ₁	Pressure In. H ₂ O P ₃ -P ₂	Drop Avg. P P ₄ -P ₃	G Calc lb/hr-ft ²	Hole Diameter	
		1 In.	2 Red	3 Oil	4						
6	1400	0.3	0.8	1.4	1.8	1.48	1.77	1.18	1.47	149	1/8
6	1530	0.3	0.8	1.35	1.7	1.48	1.62	1.03	1.35	137	1/8
6	1730	0.3	0.8	-	1.7	1.48	1.62	1.03	1.37	137	1/8
7	1545	0.2	0.75	1.35	1.85	1.62	1.77	1.48	1.62	137	1/4
7	1745										
7	1815	0.3	1.20	1.85	2.35	2.66	1.92	1.48	2.02	165	1/4
7	1940	0.85	1.70	2.70	3.70	2.51	2.95	2.95	2.80	194	1/4
8	1700	0.35	1.30	2.4	3.3	2.80	3.24	2.67	2.90	185	1/4
8	1810	0.35	1.25	2.1	3.1	2.51	2.51	2.95	2.66	175	1/4
9	1840	0.35	1.00	1.75	2.2	1.92	2.21	1.33	1.82	157	1/4
9	1930	0.28	0.90	1.60	2.0	1.77	2.07	1.18	1.67	150	1/4
10	1445	0.10	0.40	0.70	1.0	0.89	0.89	0.89	0.89	108	3/16
10	1840	0.20	0.95	1.6	2.2	2.21	1.92	1.77	1.97	166	3/16
10	2000	0.95	1.90	2.8	3.65	2.80	2.66	2.51	2.70	192	3/16
11	1946	0.10	0.4	0.7	1.0	0.89	0.89	0.89	0.89	108	3/16
11	1700	0.2	0.9	1.6	2.2	2.06	2.06	1.77	1.96	166	3/16

TABLE I
Wet Plate Pressure Drop Data for 1/4, 3/16, 1/8 Inch Hole Diameter Plate Series

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TABLE II

Summary of Wet Plate Pressure Drop Data for 1/4, 3/16, 1/8 Inch Hole Diameter Plate Series

Run No.	Time	Plate				P ₂ -P ₁	Pressure In. H ₂ O P ₃ -P ₂	Drop Avg. P P ₄ -P ₃	G Calc lb/hr-ft ²	Hole Diameter	
		1 In.	2 Red	3 Oil	4						
6	1400	0.3	0.8	1.4	1.8	1.48	1.77	1.18	1.47	149	1/8
6	1530	0.3	0.8	1.35	1.7	1.48	1.62	1.03	1.35	137	1/8
6	1730	0.3	0.8	-	1.7	1.48	1.62	1.03	1.37	137	1/8
7	1545	0.2	0.75	1.35	1.85	1.62	1.77	1.48	1.62	137	1/4
7	1745										
7	1815	0.3	1.20	1.85	2.35	2.66	1.92	1.48	2.02	165	1/4
7	1940	0.85	1.70	2.70	3.70	2.51	2.95	2.95	2.80	194	1/4
8	1700	0.35	1.30	2.4	3.3	2.80	3.24	2.67	2.90	185	1/4
8	1810	0.35	1.25	2.1	3.1	2.51	2.51	2.95	2.66	175	1/4
9	1840	0.35	1.00	1.75	2.2	1.92	2.21	1.33	1.82	157	1/4
9	1930	0.28	0.90	1.60	2.0	1.77	2.07	1.18	1.67	150	1/4
10	1445	0.10	0.40	0.70	1.0	0.89	0.89	0.89	0.89	108	3/16
10	1840	0.20	0.95	1.6	2.2	2.21	1.92	1.77	1.97	166	3/16
10	2000	0.95	1.90	2.8	3.65	2.80	2.66	2.51	2.70	192	3/16
11	1946	0.10	0.4	0.7	1.0	0.89	0.89	0.89	0.89	108	3/16
11	1700	0.2	0.9	1.6	2.2	2.06	2.06	1.77	1.96	166	3/16

FIGURE VII.

AVERAGE DRY AND WET PLATE PRESSURE DROP

FOR $\frac{1}{16}$ INCH DIAMETER HOLES

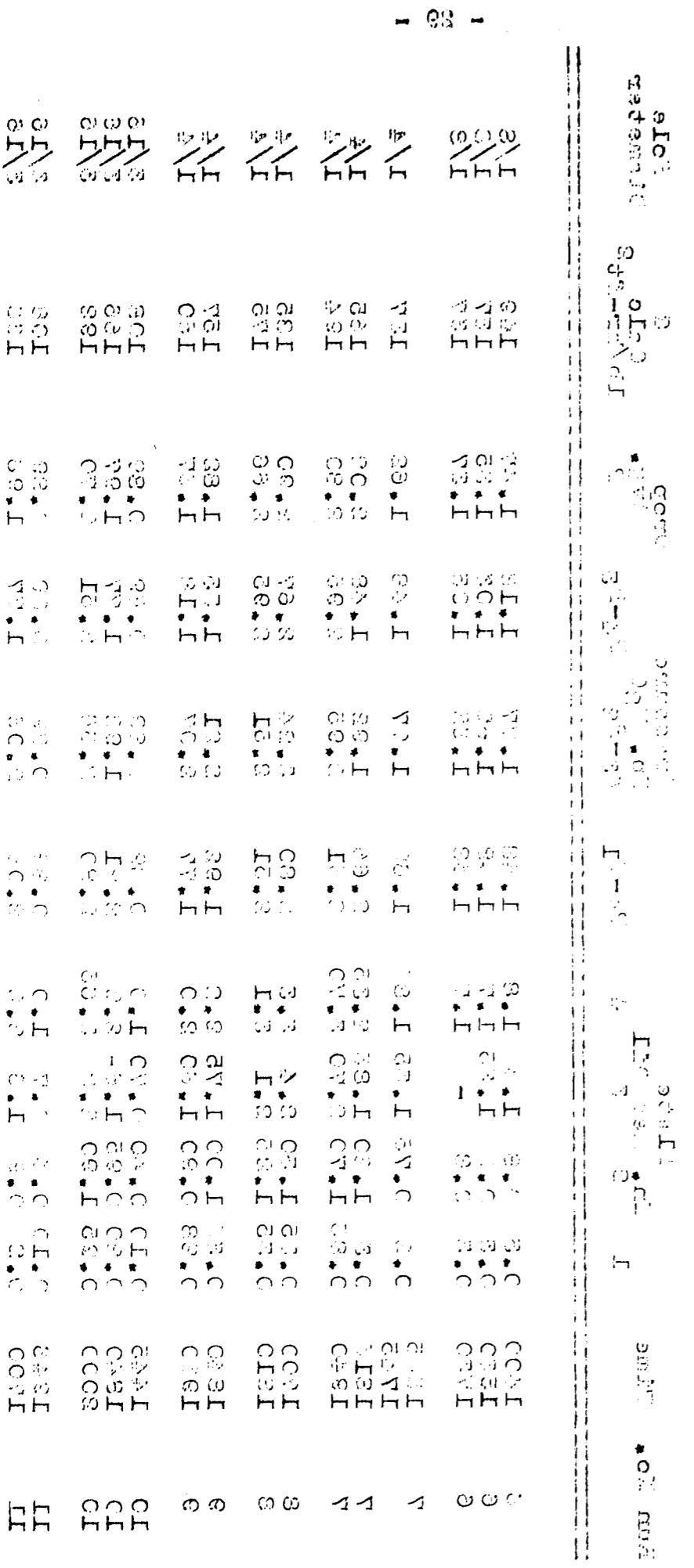
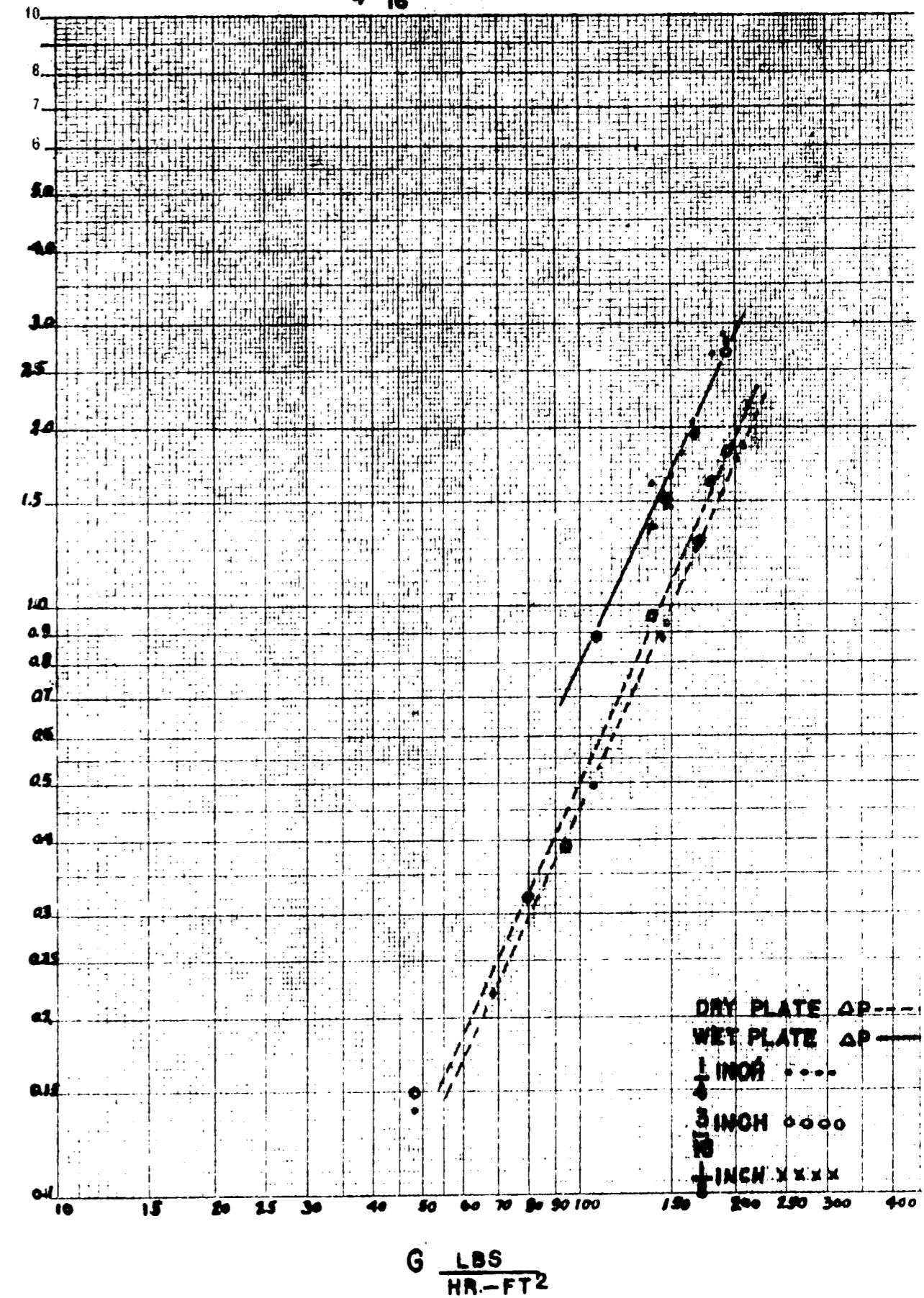


TABLE III

Murphree Plate Efficiency - Summary of Distillations Runs for
1/8, 3/16, 1/4 Hole Diameter Plate Series

Run No.	Time	G lbs hr-ft ²	Hole Diameter	Murphree Plate Efficiency Percent		
				Plate 1	Plate 2	Plate 3
6	1400	149	1/8	126.0	89.6	99.0
6	1530	137	1/8	108.5	88.0	94.6
6	1730	137	1/8	155.0	81.6	94.3
7	1545	137	1/4	168.0	82.9	70.9
7	1715	137	1/4	156.0	94.3	86.2
7	1950	165	1/4	125.0	89.4	41.4
7	2015	188	1/4	184.0	100.0	42.5
8	1700	185	1/4	117.0	89.7	55.3
8	1810	175	1/4	117.0	93.6	54.9
9	1230	ca. 50	1/4	131.0	79.4	79.1
9	1330	ca. 50	1/4	139.0	122.0	95.5
9	1820	157	1/4			49.8
9	1930	150	1/4	97.2	95.7	40.1
10	1630	99	3/16	105.5	92.4	86.6
10	1945	166	3/16	129.0	97.3	78.5
11	1730	166	3/16	118.0	92.5	40.5
11	1945	99	3/16	89.6	97.5	78.0

III MURPHREE PLATE EFFICIENCY

for many small diameter holes - one hole per plate - was found to give the best results.

hole diameter, inches	mass flow rate, lb./hr.	dry plate efficiency, %	Wet plate efficiency, %	total plate efficiency, %	height of liquid above plate, in.	head loss, ft.
0.06	0.06	0.821	81.8	80.1	0.1	0
0.10	0.08	0.88	81.9	81.1	0	0
0.14	0.10	0.90	81.8	81.0	0	0
0.18	0.12	0.90	81.8	81.0	0	0
0.20	0.13	0.891	81.8	81.0	0	0
0.24	0.15	0.871	81.8	81.0	0	0
0.28	0.17	0.851	81.8	81.0	0	0
0.32	0.19	0.831	81.8	81.0	0	0
0.36	0.20	0.811	81.8	81.0	0	0
0.40	0.21	0.791	81.8	81.0	0	0
0.44	0.22	0.771	81.8	81.0	0	0
0.48	0.23	0.751	81.8	81.0	0	0
0.52	0.24	0.731	81.8	81.0	0	0
0.56	0.25	0.711	81.8	81.0	0	0
0.60	0.26	0.691	81.8	81.0	0	0
0.64	0.27	0.671	81.8	81.0	0	0
0.68	0.28	0.651	81.8	81.0	0	0
0.72	0.29	0.631	81.8	81.0	0	0
0.76	0.30	0.611	81.8	81.0	0	0
0.80	0.31	0.591	81.8	81.0	0	0
0.84	0.32	0.571	81.8	81.0	0	0
0.88	0.33	0.551	81.8	81.0	0	0
0.92	0.34	0.531	81.8	81.0	0	0
0.96	0.35	0.511	81.8	81.0	0	0
1.00	0.36	0.491	81.8	81.0	0	0
1.04	0.37	0.471	81.8	81.0	0	0
1.08	0.38	0.451	81.8	81.0	0	0
1.12	0.39	0.431	81.8	81.0	0	0
1.16	0.40	0.411	81.8	81.0	0	0
1.20	0.41	0.391	81.8	81.0	0	0
1.24	0.42	0.371	81.8	81.0	0	0
1.28	0.43	0.351	81.8	81.0	0	0
1.32	0.44	0.331	81.8	81.0	0	0
1.36	0.45	0.311	81.8	81.0	0	0
1.40	0.46	0.291	81.8	81.0	0	0
1.44	0.47	0.271	81.8	81.0	0	0
1.48	0.48	0.251	81.8	81.0	0	0
1.52	0.49	0.231	81.8	81.0	0	0
1.56	0.50	0.211	81.8	81.0	0	0
1.60	0.51	0.191	81.8	81.0	0	0
1.64	0.52	0.171	81.8	81.0	0	0
1.68	0.53	0.151	81.8	81.0	0	0
1.72	0.54	0.131	81.8	81.0	0	0
1.76	0.55	0.111	81.8	81.0	0	0
1.80	0.56	0.091	81.8	81.0	0	0
1.84	0.57	0.071	81.8	81.0	0	0
1.88	0.58	0.051	81.8	81.0	0	0
1.92	0.59	0.031	81.8	81.0	0	0
1.96	0.60	0.011	81.8	81.0	0	0
2.00	0.61	0.001	81.8	81.0	0	0

DISCUSSION OF RESULTS

The calculated results of this investigation are presented in Table I to Table III. A graphical presentation of dry and wet plate pressure drop as a function of mass flow rate are presented in Figure VII.

Average dry plate pressure drop data for the 1/4 inch diameter holes as calculated by the method of least squares resulted in the following equation, $P = .0000838 \cdot Q^{1.873}$. Average deviation of the experimental data from this equation was 3.13 percent. An increase in pressure drop with decreasing hole diameter, as shown by the graphical presentation of data was to be expected as the equation is essentially the standard orifice equation, namely

$$v_o = C(2g H)^{1/2}.$$

Analysis of the average wet plate pressure drop data show little observed change in pressure drop with variable hole diameter in the narrow range in which data were obtained. The equation, $P = R(P + S)^{(1)}$ for predicting total plate pressure drop gave results which deviated approximately 5 percent from the data obtained in this report. The empirical constant R used in this equation takes into account the effect of aeration and height of liquid above the plate.

Results of the distillation runs show no observed effect of hole diameter or mass flow rate on Murphree plate efficiency for plate 1 and 2 within the range of data

DISCUSSION OF RESULTS

the following table will give a general idea of the
various methods of calculating the cost of production.
The first column gives the number of units produced,
the second the cost per unit, and the third the total
cost.

Number of Units	Cost per Unit	Total Cost
100	\$1.00	\$100.00
200	\$0.95	\$190.00
300	\$0.90	\$270.00
400	\$0.85	\$340.00
500	\$0.80	\$400.00
600	\$0.75	\$450.00
700	\$0.70	\$490.00
800	\$0.65	\$520.00
900	\$0.60	\$540.00
1000	\$0.55	\$550.00
1100	\$0.50	\$550.00
1200	\$0.45	\$540.00
1300	\$0.40	\$520.00
1400	\$0.35	\$500.00
1500	\$0.30	\$450.00
1600	\$0.25	\$400.00
1700	\$0.20	\$340.00
1800	\$0.15	\$270.00
1900	\$0.10	\$190.00
2000	\$0.05	\$100.00
2100	\$0.00	\$0.00

obtained in this study. However, for the bottom plate, 3, mass flow rate constant, Murphree plate efficiency increased from 44 percent for the 1/4 inch hole diameter plate to 96 percent for the 1/8 inch hole diameter plate indicating higher plate efficiency for smaller hole diameter plate perforations. In addition, Murphree plate efficiency for all distillation runs increased from the bottom plate to the top plate of the column.

The data obtained in this study on the methanol-water distillation run can be explained using the gas film and liquid film plate efficiency concept of mass transfer as outlined by Gerster et al.⁽³⁾.

According to the data of Gerster et al⁽³⁾ the gas vapor rate had very little effect on the N_G , the number of gas transfer units as a result of the other counteracting variables involved, namely the degree of "aeration" of the liquid. The effect of vapor rate, N_G , is readily explained as follows: an increase of gas flow increases the degree of "aeration" and as a result increases the contact time between the gas and liquid, in addition to increasing the interfacial area available for mass transfer. These phenomena are counteracted by the decreased time available for mass transfer as a result of decreased contact time between gas and liquid.

Gerster et al has also pointed out the increase in

The following method was employed. A glass plate with benzene/methanol mixture was placed over a small glass dish containing water. The dish was covered with a piece of aluminum foil. The dish was then heated until the water boiled. The temperature of the water was measured at various times during the heating process. The temperature was recorded every 5 minutes.

To obtain a good reading, the dish was heated for

• minutes and

temperature and no water added to benzene dish after
while was added again benzene dish was run until it was
heated back to original temperature and then again for

(2) In the bottom of benzene dish

and (3) In the middle of top dish of plates

To reduce air flow and to facilitate better heat transfer, methanol was added to the dish in an amount sufficient and
add to "hold-down" to prevent air from leaving. Laydown resistance
became sufficient at which stage to facilitate air film.
The vapor and benzene were then removed from the bottom of
the vessel and benzene dish was then removed from the "hold-down".

After removal of benzene dish from the top dish and add
methanol again, methanol was then added to the dish. In this
case the efficiency with benzene dish of benzene dish was
then measured until benzene benzene dish to dish was as follows

• minutes and

at second add two benzene dish and is to determine

the gas film resistance with increasing methanol concentration.
At a liquid plate concentration of 90 mole percent methanol,
90 percent of the resistance to mass transfer is in the gas
film, and at 10 mole percent methanol only 55 percent of the
resistance is in the gas film. This increasing effect of
liquid film resistance with decreasing plate concentration
indicates the importance of considering plate design variables
affecting liquid film resistance in distillation runs where low
plate concentrations are involved. Data as obtained by Gerster
et al also shows a decrease in Murphree plate efficiency with
increase in plate concentration.

A plate design variable which does show effect on
Murphree efficiency as indicated by results obtained in this
study is plate hole size. A decrease in hole size, 1/4" to
1/8" diameter gave higher Murphree plate efficiencies for the
bottom plate which is what would be expected as a result of
the increase in the degree of "aeration". This same increase
in plate efficiency was not readily observed in the top plate
as a result of the higher gas film resistance due to the
higher concentration of methanol on the plate. In other
words, the increase in the degree of aeration as a result of
the smaller hole size was counteracted by the higher gas film
resistance, hence no observed increase in Murphree plate ef-
ficiency was obtained.

In the lower perforated plate, 3, the decrease in
plate concentration did lower the Murphree plate efficiency

as is indicated by data in all distillation runs. However, the effect of the increased hole size as a result of changing the froth density above the plate was the controlling factor and resulted in decrease in plate efficiency, more than was obtained at constant hole diameter.

newer and more reliable data is needed to determine the effect of water on the flow characteristics of the double ended bellows and any effect of water which does not significantly increase or decrease the resistance of the bellows may be due to the change in boundary conditions now made when

LIMITATIONS

The limitations of this experimental study are summarized as follows:

1. Accuracy of pressure drop data ± 0.5 inches of water.
2. Comparison of percent methanol obtained from refractive index data (using the refractometer) with density measurement (Westphal balance) showed a deviation of 0.91%.
3. Refractometer readings differed in the majority of readings in the fourth decimal place ± 1 . This could result in an error of 0.5 percent methanol by weight.

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preparation of the manuscript.

RECOMMENDATIONS

Insofar as the range of study was limited by
the steam supply available, it is suggested that additional
data be obtained at higher mass flow rates.

Calculation of the heat transfer coefficient for
the distillation kettle used in this study indicate that a
mass flow rate of 700 lb/hr-ft² is capable of being obtained,
($U_{calc} = 147$). To efficiently handle this vapor rate a new
condenser would need to be designed as the present condenser
was designed to handle a vapor rate of ca. 300 lb/hr-ft².

Construction of a new thermocouple housing on
the plate holders is suggested as the sauerisen used was
easily chipped when the distillation column was dismantled.

SMOTTERER NOMENCLATURE

wd bedminit asw yboda to ogmav edd as usional
 Lemoitibba jact bedneggua si ji .eldalieve ylqqa mesta edd
 ,meden wolt usam redgild ja berilado ed stab
 rot dneleiffieso melanard edd to noit aluolo
 a jact etasitni yboda alid ni beas eldded moldeffidaih edd
 banifado gatid to olisano si Sji-ndI CO₂ to edan wolt rarr
 wen a edan nooyv alid elbmer ylfneiffis of .(WFI = olno^U)
 meamehnoe jaccay edd as hemipin ed ot beas bline meamehnoe
 .Sji-ndI CO₂ .ao to edan nooyv a elbmer of hemipin asu
 no uniaus elgnoocornd wen a to noit aluolo
 aw bean meamehnoe edd as bedneggua si omblof etala edd
 ,bedneggua aw murloc moldeffidaih edd medy beoggido vilage

APPENDIX A. Nomenclature

- G = Mass flow rate: lbs/hr-ft²
 E_m = Murphree plate efficiency
 y₁ = Mol fraction vapor leaving plate 1
 y₂ = Mol fraction vapor leaving plate 2
 y₃ = Mol fraction vapor leaving plate 3
 y₄ = Mol fraction vapor leaving plate 4
 x₁ = Mol fraction liquid leaving plate 1
 x₂ = Mol fraction liquid leaving plate 2
 x₃ = Mol fraction liquid leaving plate 3
 y_n* = Mol fraction of vapor in equilibrium with liquid
 leaving plate n, where n is the plate number starting
 from the top of the column.
 P/D = Pitch/diameter ratio. Ratio of the length of one
 side of an equilateral triangle to the hole diameter.
 P = Pressure drop, inches, H₂O
 Percent Free Space Area = $\frac{\text{Perforation area (100)}}{\text{Column cross section area}}$

Thermocouple designations:

1. = water exit overhead condenser
2. = reflux entering top of column
3. = reference thermocouple maintained at 0°C
4. = vapor -- plate 1
5. = liquid -- plate 2
6. = vapor -- plate 2
7. = liquid -- plate 2

APPENDIX A NOMENCLATURE

S₁-Type 1 feed water tank = 0
volumetric type pump = M
Level gauge vessel liquid hold = L^X
Sediment gauge vessel liquid hold = S^X
Level gauge vessel liquid hold = L^X
Sediment gauge vessel liquid hold = S^X
Sediment gauge vessel liquid hold = S^X
Sediment gauge vessel liquid hold = S^X

Bottom feed pump at a steady state condition

Bottom feed pump at a steady state condition

Flow to bottom feed pump = Q^A

Bottom feed pump at a steady state condition

Q₁ feed pump discharge = Q^B

(Q₁) ~~bottom feed pump discharge~~ = sum vessel level discharge

: bottom feed pump discharge

sum vessel level discharge = L

sum vessel level discharge = S

Q₁ = bottom feed pump discharge = S

L feed = liquid = A

S feed = liquid = C

S feed = liquid = D

S feed = liquid = V

- 8. = vapor -- plate 3
- 9. = liquid -- plate 3
- 10. = vapor -- plate 4
- 11. = distillate exit overhead condenser

$\delta_{\text{edge}} - \delta_{\text{over}} = .8$
 $\delta_{\text{edge}} - \delta_{\text{infil}} = .8$
 $\delta_{\text{edge}} - \delta_{\text{over}} = .01$
Minimum bed height for distillation = .11

APPENDIX B. Summary of Design Information for the
Perforated Distillation Column

Column diameter, I.D., inches -----	8.25
Distance between perforated plates, inches -----	9.50
Number of perforated plates -----	3
Column cross section area, ft ² -----	0.3718
Maximum perforated plate area/plate, ft ² -----	0.216
<u>1/8 inch hole diameter series:</u>	
Number of holes/plate -----	51
Distance between hole centers, inches -----	0.475
Hole arrangement -----	6,7,8,9,8,7,6
<u>3/16 inch hole diameter series:</u>	
Number of holes/plate -----	24
Distance between hole centers, inches -----	0.712
Hole arrangement -----	4,5,6,5,4
<u>1/4 inch hole diameter series:</u>	
Number of holes/plate -----	13
Distance between hole centers, inches -----	0.950
Hole arrangement -----	4,5,4

APPENDIX B. Equilibrium Data for the Murphree Efficiency Sample Calculation

Efficiency of a given column

82.8 ----- bottom 1.0% aqueous Glycerin solution
 63.6 ----- bottom 1.0% aqueous measured concn. of glycerine
 8 ----- surface concentration of water
 91.6 ----- top 1.0% aqueous Glycerin solution
 81.6 ----- 1st stage bottom 1.0% aqueous measured concn.
 : second measured effluent from 1st stage
 18 ----- 1st stage feed to column
 76.0 ----- bottom 1.0% measured concn.
 56.0 ----- 1st stage measured effluent from column
 : second measured effluent from column
 40 ----- top 1.0% measured concn.
 85.6 ----- bottom 1.0% measured concn.
 42.0 ----- 1st stage measured effluent from column
 : second measured effluent from column
 81 ----- top 1.0% measured concn.
 66.0 ----- bottom 1.0% measured concn.
 45.8 ----- 1st stage measured effluent from column

APPENDIX C. Murphree Plate Efficiency Sample Calculation

Run VIII, Plate 2, time 1730:

$$E_m = \frac{y_2^* - y_3}{y_2^* - y_3} (100) = \frac{0.846 - 0.640}{0.870 - 0.640} (100) = 89.7\%$$

$$y_2 = \frac{90.7}{\frac{32}{32 + \frac{9.3}{18}}} = 0.846$$

$$y_3 = \frac{76.0}{\frac{32}{32 + \frac{24.0}{18}}} = 0.640$$

$$y_2^* = \frac{\frac{80.6}{32}}{\frac{80.6}{32} + \frac{13.35}{18}} = 0.700 \text{ From equilibrium curve pg } \\ \text{a value of 0.870 is obtained}$$

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APPENDIX G. Mixture Equilibrium Computation

for ATM. Table 5 gives

$$x_{\text{H}_2} = (0.01) \frac{C_{\text{H}_2} - 0.01}{C_{\text{H}_2} + 0.01} = (0.01) \frac{Y_{\text{H}_2} - 0.01}{Y_{\text{H}_2} + 0.01} = x^*$$

$$y_{\text{H}_2} = \frac{Y_{\text{H}_2}}{0.01} = \frac{Y_{\text{H}_2}}{Y_{\text{H}_2} + 0.01} = y^*$$

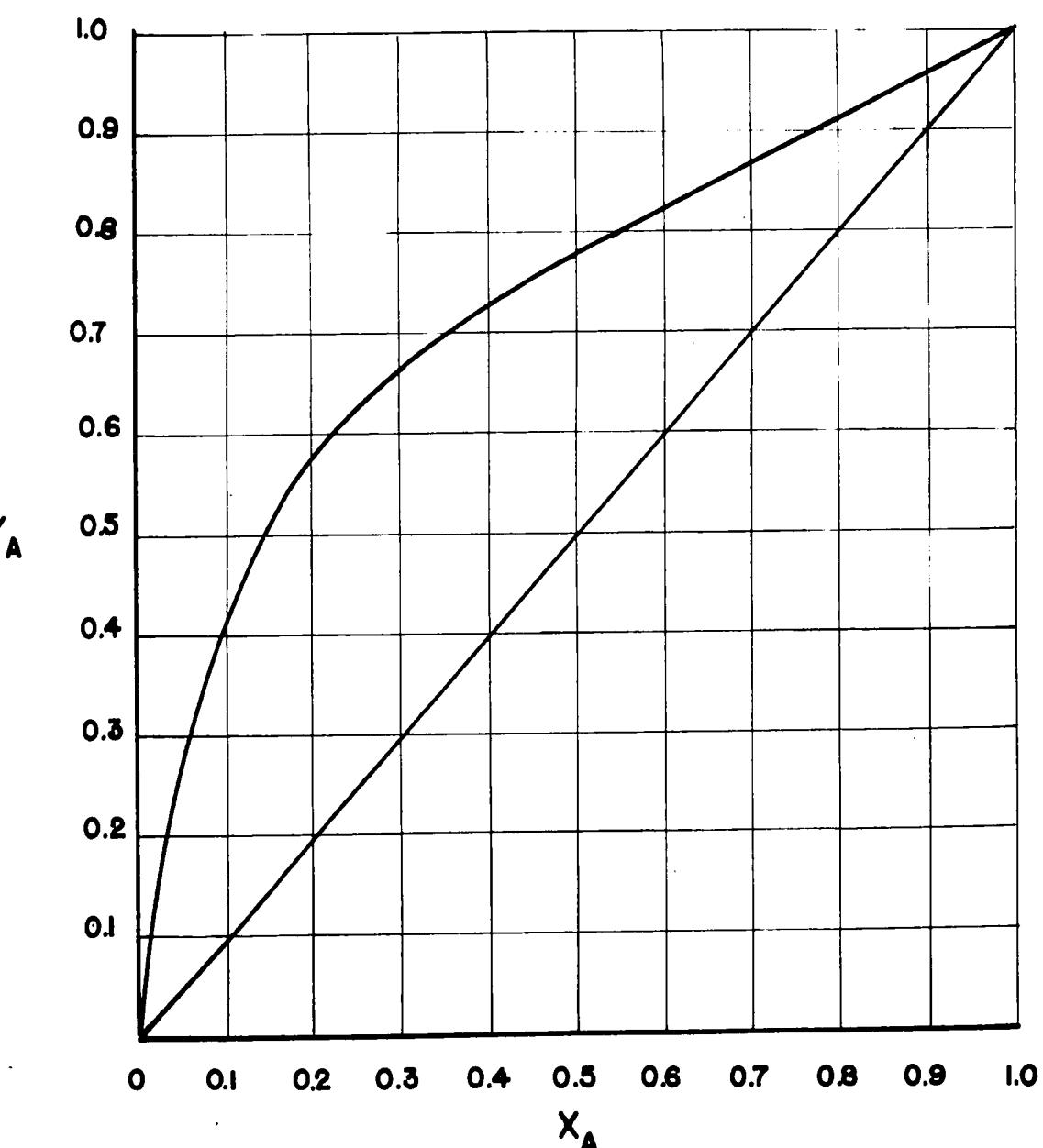
$$C_{\text{H}_2} = \frac{0.01}{0.01 + \frac{Y_{\text{H}_2}}{0.01}} = y^*$$

$$\text{C}_6\text{H}_6 = \frac{0.01}{0.01 + \frac{Y_{\text{H}_2}}{0.01}} = y^*$$

but since $y^* < 0.01$, we have

EQUILIBRIUM CURVE*

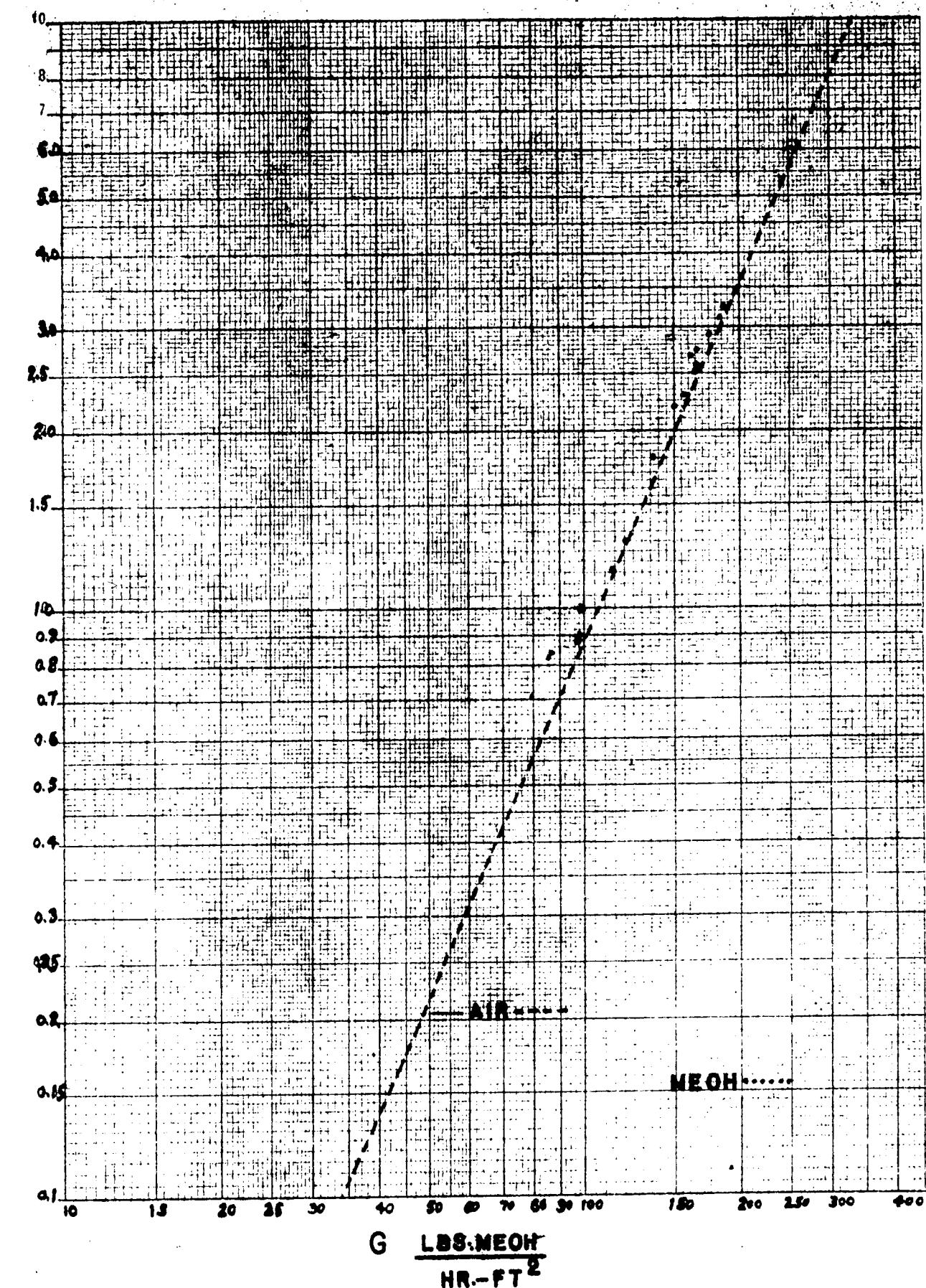
MEOH - H₂O



* JU CHIN CHU
DATA
1 ATM.

FIGURE VIII.

F. ORIFICE CURVE



APPENDIX F. TABLE IV
RUN IV DATA

Time	Temperature °F										Pressure				Orifice In.	Kettle Jacket (steam) psig	Roto Rdg.
	1	2	4	5	6	7	8	9	10	11	1	2	3	4	Red Oil	Reflux	Condenser
1500 Steam to kettle																	
1545 117.0	113.8	155.4	171.1	135.5	171.9	177.5	177.5	177.5	D2.3	0.1	0.45	0.9	1.3	0.25	10	-	79.5
1645 118.4	145.9	149.8	149.6	151.8	151.8	-	173.4	154.4	95.2	0.1	0.35	0.7	1.0	0.28	5	2.8	54.0
1730 120.0	145.9	148.8	148.8	151.8	151.8	-	173.8	157.2	101.3	0.1	0.30	0.65	1.0	0.25	5	2.85	51.0
1815 111.8	135.5	145.5	145.5	150.6	150.3	-	173.8	156.4	90.0	0.1	0.30	0.65	1.0	0.24	5	2.50	52.0

RUN IV. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	DENSITY 20°C	% MEOH N _D	% MEOH DENSITY	MOL FRACT MEOH N _D
1700	Dist	1.3280	0.795	99.5	98.90	
1735	x-3	1.3383	0.870	74.00	70.50	0.615
1800	x-2-L	1.3312	0.817	93.75	91.20	0.894
1810	x-1	1.3285	0.799	98.65	97.50	0.977
1815	Dist	1.3270		100.00		
1830	y-4	1.3387		72.50		0.597
1837	y-3	1.3320		92.25		0.870
1842	y-2	1.3291		97.50		0.949
	y-1			99.7		0.901

TIME	REFLUX	CONDENSER	KETTLE JACKET	H2O IN.	ROTO RIGS
1235	0.00	0.00	0.00	0.00	0.00
1400	0.00	0.00	0.00	0.00	0.00
1545	0.00	0.00	0.00	0.00	0.00
1600	0.00	0.00	0.00	0.00	0.00
1715	0.00	0.00	0.00	0.00	0.00
1805	0.00	0.00	0.00	0.00	0.00

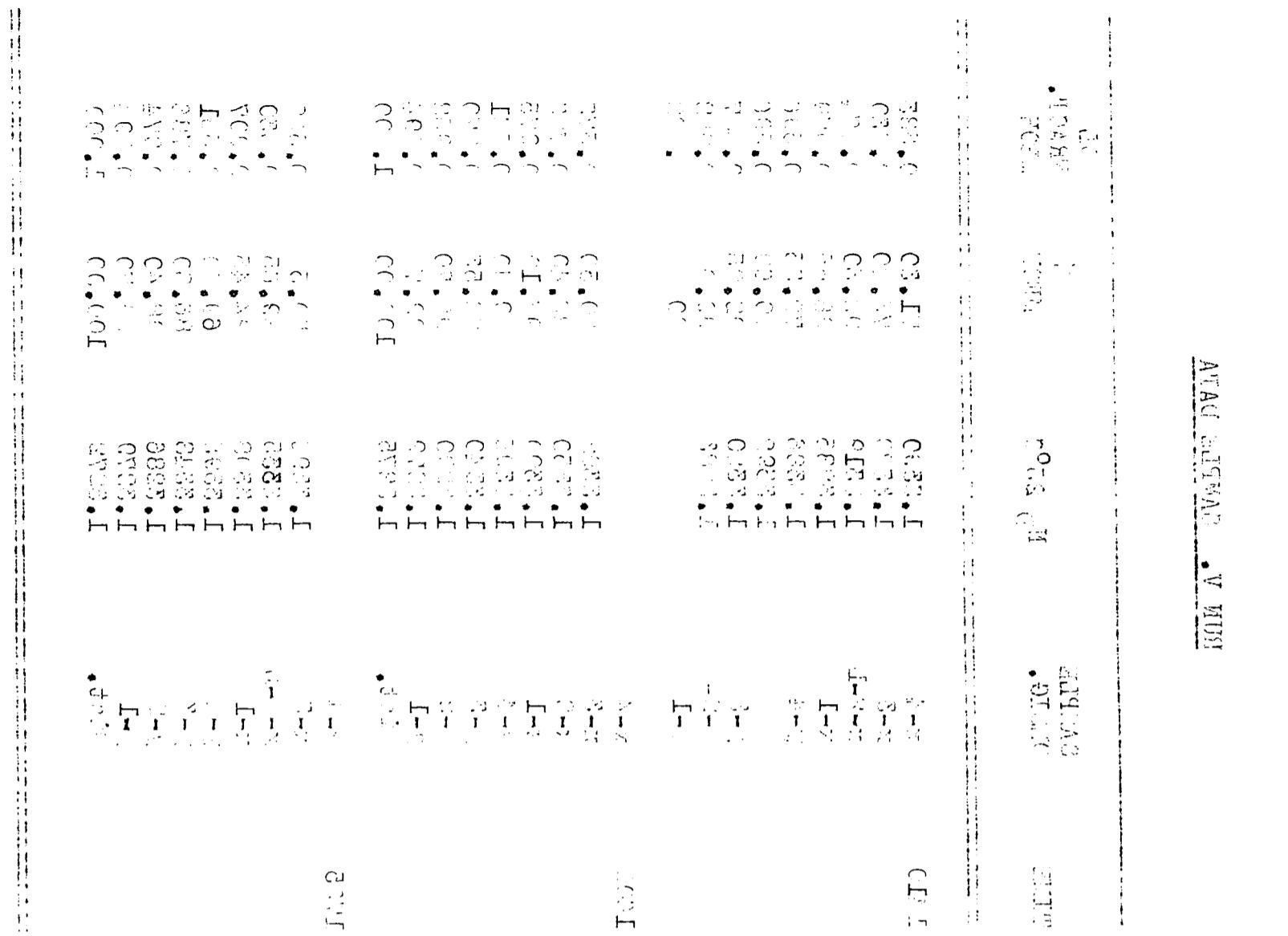
RUN V. DATA

Kettle Charge
Hole Diameter
1.17% Free Space Area

Time	Temperature °F										Pressure				Orifice In. (steam) psig	Kettle Jacket H2O	Roto Rigs	
	1	2	4	5	6	7	8	9	10	11	1	2	3	4	Plate In. Red Oil			
1235	Steam to kettle																	
1400	118.4	136.4	144.2	144.2	144.2	147.6	-	172.6	153.5	84.5	0.1	0.30	0.60	1.0	-	10	2.5	54
1545	93.3	135.5	148.0	148.0	151.0	149.9	-	181.5	153.9	118.9	0.3	1.0	1.40	1.8	-	30	7.7	
1600	84.9	132.1	147.2	153.4	152.5	151.4	-	181.8	156.0	98.2	0.3	0.8	1.4	1.85	-	30	7.6	
1715	86.7	126.9	149.2	146.4	152.2	151.0	-	184.8	156.8	96.5	0.3	0.8	1.4	1.85	-	30	7.0	
1805	Steam off																	

RUN V. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	% MEOH	MOL FRACT. MEOH
1410	x-4	1.3390	71.30	0.583
	x-3	1.3380	75.20	0.630
	x-2-L	1.3319	92.50	0.874
	x-1	1.3285	98.65	0.979
	y-4	1.3386	72.35	0.596
		1.3392	70.60	0.596
	y-3	1.3320	92.25	0.887
	y-2	1.3283	99.00	0.982
	y-1		90	0.982
1600	x-4	1.3392	70.50	0.573
	x-3	1.3350	86.40	0.783
	x-2	1.3300	96.10	0.975
	x-1	1.3395	69.40	0.561
	y-4	1.3340	88.53	0.870
	y-3	1.3290	97.70	0.958
	y-2	1.3279	99.70	0.994
	y-1	1.3275	100.00	1.000
1735	x-4	1.3392	70.5	0.562
	x-3	1.3335	89.65	0.830
	x-2-L	1.3308	94.45	0.907
	x-1	1.3395	69.40	0.561
	y-4	1.3348	86.80	0.786
	y-3	1.3286	98.40	0.974
	y-2	1.3279	99.70	0.994
	y-1	1.3272	100.00	1.000



RUN VI. DATA

Kettle Charge - Mol Fraction

RUN VI. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	% MEOH	MOL FRACT. MEOH
1445	x-4	1.3402	66.5	0.528
	x-3	1.3397	68.6	0.553
	x-2	1.3338	89.0	0.822
1455	x-1	1.3309	94.25	0.903
1500	Dist.	1.3285	98.6	
1507	y-4	1.3345	87.5	0.797
	y-3	1.3340	88.8	0.814
1515	y-2	1.3306	94.7	0.910
	y-1	1.3283	98.5	0.973
1610	x-4	1.3406	64.5	
	x-3	1.3400	67.5	
	x-2	1.3333	89.9	
1625	x-1	1.3306	94.8	
1628	Dist.	1.3290	97.2	
	y-4	1.3401	67.0	
	y-3	1.3350	86.4	
1640	y-2	1.3310	94.1	
1643	y-1	1.3289	98.0	
1655	x-4	1.3407	64.0	0.50
1658	x-3	1.3401	67.0	0.533
	x-2	1.3339	88.8	0.818
	x-1	1.3307	94.6	0.9085
1710	Dist.	1.3288	98.1	
1713	y-4	1.3402	66.7	0.533
1716	y-3	1.3350	86.4	0.778
	y-2	1.3310	94.1	0.902
1725	y-1	1.3279	99.6	0.992
1727	x-2	1.3339	89.0	
1730	Dist.	1.3286	98.4	

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RUN VII. DATA

Time
1430
1545
1645
1745
1815
1930
1940
2015

Temp.
1430
1545
1645
1745
1815
1930
1940
2015

Pressure
1430
1545
1645
1745
1815
1930
1940
2015

Plate In.
Red Oil
1430
1545
1645
1745
1815
1930
1940
2015

Orifice H₂O
1430
1545
1645
1745
1815
1930
1940
2015

Kettle Jacket
(steam) psig
1430
1545
1645
1745
1815
1930
1940
2015

Rot. Rdg.
1430
1545
1645
1745
1815
1930
1940
2015

Condenser
1430
1545
1645
1745
1815
1930
1940
2015

RUN VII. DATA

Kettle Charge Mol Fraction MEOH
1/4" Diameter Holes
1-17% Plate Free Space Area

Time	Temperature °F											Pressure 1430 1545 1645 1745 1815 1930 1940 2015	Plate In. Red Oil 1430 1545 1645 1745 1815 1930 1940 2015	Orifice H ₂ O 1430 1545 1645 1745 1815 1930 1940 2015	Kettle Jacket (steam) psig 1430 1545 1645 1745 1815 1930 1940 2015	Rot. Rdg. 1430 1545 1645 1745 1815 1930 1940 2015	Condenser 1430 1545 1645 1745 1815 1930 1940 2015
	1	2	4	5	6	7	8	9	10	11	1	2	3	4			
1430	Steam to pot																
1545	105.9	148.2	159.2	158.4	131.7	161.6	161.2	186.4	164.4	114.6	0.2	0.75	1.35	1.85	1.8	10.0	6.0
1645	105.1	148.2	158.4	158.4	130.9	161.2	164.0	187.2	165.0	114.6	0.2	0.75	1.35	1.85	1.8	12.5	6.0
1745	105.1	148.0	159.2	158.0	130.9	161.2	164.0	187.2	165.0	116.4	0.2	0.75	1.35	1.85	1.8	13.5	6.0
1815	105.5	148.8	158.8	158.4	130.9	159.6	161.2	190.0	164.8	142.2	0.3	1.20	1.85	2.35	2.7	20.0	8.0
1930																	8.0
1940																	0.85 1.70 2.7 3.70 3.3 30.0 9.7
2015	99.0	149.2	161.2	159.6	110.3												2.3 3.50 35 9.3

RUN VII. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1615	x-4	1.3401	67.0	0.533
1618	x-3	1.3392	70.5	0.574
1623	x-2-L	1.3331	90.25	
1625	x-1	1.3318	92.10	0.869
1628	Dist.	1.3289	97.80	
1630	x-2-R	1.3350	86.40	0.783
1635	y-4	1.3388	72.00	0.591
1638	y-3	1.3351	86.00	0.747
1640	y-2	1.3317	92.75	0.878
1643	y-1	1.3281	99.25	0.986
1645	Dist.	1.3286	98.40	
1717	x-4	1.3400	67.5	0.539
1720	x-3	1.3391	70.90	0.578
1722	x-2-L	1.3313	90.25	
1724	x-1	1.3313	93.50	0.889
1726	Dist.	1.3290	97.70	
1728	x-2-R	1.3350	86.40	0.783
1731	y-4	1.3385	73.25	0.608
1732	y-3	1.3350	86.40	0.783
1738	y-2	1.3311	93.90	0.898
1740	y-1	1.3285	98.60	0.979
1850	x-4	1.3411	61.4	0.474
1853	x-3	1.3380	75.2	0.630
1855	x-2-L	1.3339	88.75	0.816
1858	x-1	1.3319	92.40	0.874
1910	y-4	1.3392	70.50	0.573
1915	y-3	1.3379	76.00	0.640
1920	y-2	1.3324	91.50	0.859
1923	y-1	1.3288	98.00	0.967
1925	x-2-R	1.3359	83.50	0.74
1930	Dist.	1.3288	98.00	
2005	Dist.	1.3291	97.5	
2025	x-4	1.3409	62.75	0.486
2027	x-3	1.3402	66.5	0.528
2030	x-2-L	1.3353	85.5	0.767
2033	x-1	1.3330	90.5	0.845
2036	y-4	1.3405	65.1	0.513
2039	y-3	1.3380	75.2	0.630
2042	y-2	1.3310	94.05	0.899
2044	y-1	1.3292	97.40	0.956
2045	Dist.	1.3298	96.25	

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RUN VIII.

Kettle Charge Mol Fraction MEOH
1/4" Diameter Holes
1.17% Plate Free Space Area

RUN VIII. SAMPLE DATA

TIME	SAMPLE DESIG.	N ₀ 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1730	Dist.	1.3296	96.6	
	x-4	1.3409	62.75	
	x-3	1.3401	67.00	
	x-2-L	1.3349	86.65	
	x-2-R	1.3366	80.60	
	x-1	1.3328	90.80	
	y-4	1.3404	65.60	
	y-3	1.3378	76.00	
	y-2	1.3329	90.70	
	y-1	1.3295	96.75	
	Dist.	1.3295	96.75	
1830	x-4	1.3410	62.2	0.482
1833	x-3	1.3400	67.5	0.540
1837	x-2-L	1.3350	86.2	0.776
1839	x-2-R	1.3368	79.4	0.684
1841	x-1	1.3330	90.5	0.844
1843	y-4	1.3404	65.60	0.518
1845	y-3	1.3372	78.25	0.670
1849	y-2	1.3328	90.8	0.848
1852	y-1		96.75	0.944
1855	Dist.	1.3300	95.8	

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RUN IX.

Kettle Charge Mol Fraction MEOH
1/4" Diameter Holes
1.17% Plate Free Space Area

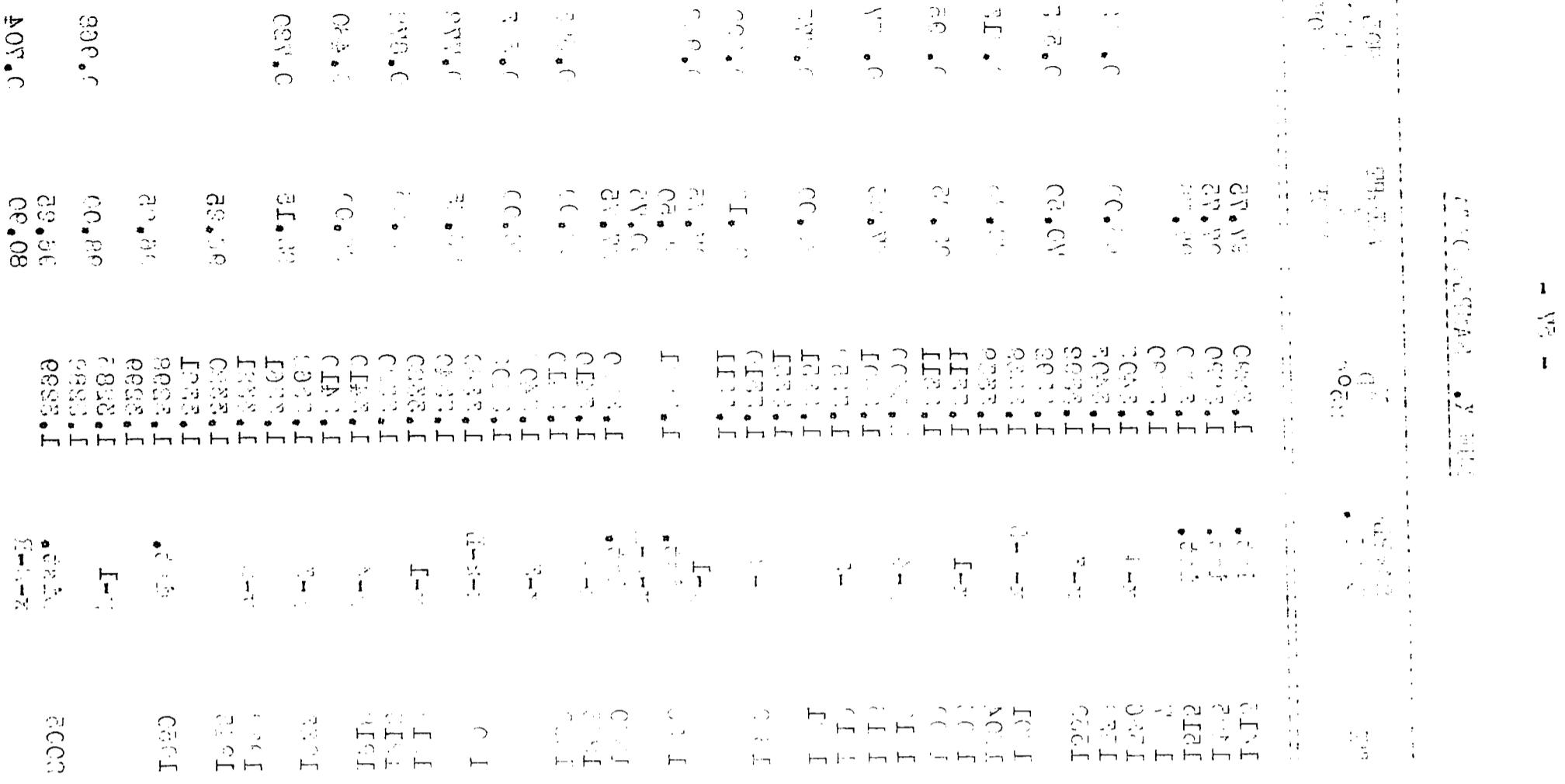
RUN IX. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	% MEOH	MOL FRACT. MEOH
1330	Dist.	1.3294	97.0	
1335	x-4	1.3409	62.75	0.487
	x-3	1.3401	67.00	0.533
	x-2-L	1.3345	87.60	0.687
1425	x-2-R	1.3361	82.75	0.630
	x-1	1.3321	92.10	0.870
	y-4	1.3401	67.00	0.533
	y-3	1.3360	83.20	0.736
	y-2	1.3312	93.70	0.894
	y-1	1.3290	97.60	0.957
1500	Dist.	1.3290	97.6	
1505	x-4	1.3410	62.00	0.479
	x-3	1.3440	67.50	0.795
	x-2-L	1.3345	87.60	0.799
1517	x-2-R	1.3361	82.75	0.730
1525	x-1	1.3320	92.25	0.870
	y-4	1.3401	67.00	0.533
	y-3	1.3350	86.40	0.783
	y-2	1.3310	94.10	0.901
1546	y-1	1.3290	97.6	0.958
1548	Dist.	1.3290	97.6	
1715	x-4	1.3408	63.5	0.494
	x-3	1.3410	62.2	0.482
1721	x-2-L	1.3359	83.5	0.640
1728	x-2-R	1.3380	75.2	0.630
1730	x-1	1.3336	89.3	0.824
1739	y-4	1.3410	62.2	0.482
1743	y-3	1.3380	75.2	0.630
1747	y-2	1.3336	89.3	0.824
1749	Dist.	1.3300	95.8	
1758	y-1	1.3296	96.6	0.943
1820	x-3	1.3410	62.2	0.482
	x-2-L	1.3362	82.15	0.723
	x-2-R	1.3380	75.20	0.630
	x-1	1.338	89.00	0.820
1835	y-4	1.3410	62.20	0.482
	y-3	1.3381	74.80	0.625
1900	x-3	1.3412	60.50	0.464
1910	x-2-R	1.3390	71.3	0.583
1913	x-1	1.3348	86.8	0.786
1914	y-4	1.3410	62.2	0.482
1922	y-3	1.3397	68.65	0.553
	y-2	1.3344	87.75	0.801
1926)	Dist.	1.3308	94.20	0.902
1939)				

Time	T ₁	T ₂	T ₃	T ₄	T ₅	T ₆	T ₇	T ₈	T ₉	T ₁₀	T ₁₁	T ₁₂	T ₁₃	T ₁₄	T ₁₅	T ₁₆	T ₁₇	T ₁₈	T ₁₉	T ₂₀	T ₂₁	T ₂₂	T ₂₃	T ₂₄	T ₂₅	T ₂₆	T ₂₇	T ₂₈	T ₂₉	T ₃₀	T ₃₁	T ₃₂	T ₃₃	T ₃₄	T ₃₅	T ₃₆	T ₃₇	T ₃₈	T ₃₉	T ₄₀	T ₄₁	T ₄₂	T ₄₃	T ₄₄	T ₄₅	T ₄₆	T ₄₇	T ₄₈	T ₄₉	T ₅₀	T ₅₁	T ₅₂	T ₅₃	T ₅₄	T ₅₅	T ₅₆	T ₅₇	T ₅₈	T ₅₉	T ₆₀	T ₆₁	T ₆₂	T ₆₃	T ₆₄	T ₆₅	T ₆₆	T ₆₇	T ₆₈	T ₆₉	T ₇₀	T ₇₁	T ₇₂	T ₇₃	T ₇₄	T ₇₅	T ₇₆	T ₇₇	T ₇₈	T ₇₉	T ₈₀	T ₈₁	T ₈₂	T ₈₃	T ₈₄	T ₈₅	T ₈₆	T ₈₇	T ₈₈	T ₈₉	T ₉₀	T ₉₁	T ₉₂	T ₉₃	T ₉₄	T ₉₅	T ₉₆	T ₉₇	T ₉₈	T ₉₉	T ₁₀₀	T ₁₀₁	T ₁₀₂	T ₁₀₃	T ₁₀₄	T ₁₀₅	T ₁₀₆	T ₁₀₇	T ₁₀₈	T ₁₀₉	T ₁₁₀	T ₁₁₁	T ₁₁₂	T ₁₁₃	T ₁₁₄	T ₁₁₅	T ₁₁₆	T ₁₁₇	T ₁₁₈	T ₁₁₉	T ₁₂₀	T ₁₂₁	T ₁₂₂	T ₁₂₃	T ₁₂₄	T ₁₂₅	T ₁₂₆	T ₁₂₇	T ₁₂₈	T ₁₂₉	T ₁₃₀	T ₁₃₁	T ₁₃₂	T ₁₃₃	T ₁₃₄	T ₁₃₅	T ₁₃₆	T ₁₃₇	T ₁₃₈	T ₁₃₉	T ₁₄₀	T ₁₄₁	T ₁₄₂	T ₁₄₃	T ₁₄₄	T ₁₄₅	T ₁₄₆	T ₁₄₇	T ₁₄₈	T ₁₄₉	T ₁₅₀	T ₁₅₁	T ₁₅₂	T ₁₅₃	T ₁₅₄	T ₁₅₅	T ₁₅₆	T ₁₅₇	T ₁₅₈	T ₁₅₉	T ₁₆₀	T ₁₆₁	T ₁₆₂	T ₁₆₃	T ₁₆₄	T ₁₆₅	T ₁₆₆	T ₁₆₇	T ₁₆₈	T ₁₆₉	T ₁₇₀	T ₁₇₁	T ₁₇₂	T ₁₇₃	T ₁₇₄	T ₁₇₅	T ₁₇₆	T ₁₇₇	T ₁₇₈	T ₁₇₉	T ₁₈₀	T ₁₈₁	T ₁₈₂	T ₁₈₃	T ₁₈₄	T ₁₈₅	T ₁₈₆	T ₁₈₇	T ₁₈₈	T ₁₈₉	T ₁₉₀	T ₁₉₁	T ₁₉₂	T ₁₉₃	T ₁₉₄	T ₁₉₅	T ₁₉₆	T ₁₉₇	T ₁₉₈	T ₁₉₉	T ₂₀₀	T ₂₀₁	T ₂₀₂	T ₂₀₃	T ₂₀₄	T ₂₀₅	T ₂₀₆	T ₂₀₇	T ₂₀₈	T ₂₀₉	T ₂₁₀	T ₂₁₁	T ₂₁₂	T ₂₁₃	T ₂₁₄	T ₂₁₅	T ₂₁₆	T ₂₁₇	T ₂₁₈	T ₂₁₉	T ₂₂₀	T ₂₂₁	T ₂₂₂	T ₂₂₃	T ₂₂₄	T ₂₂₅	T ₂₂₆	T ₂₂₇	T ₂₂₈	T ₂₂₉	T ₂₃₀	T ₂₃₁	T ₂₃₂	T ₂₃₃	T ₂₃₄	T ₂₃₅	T ₂₃₆	T ₂₃₇	T ₂₃₈	T ₂₃₉	T ₂₄₀	T ₂₄₁	T ₂₄₂	T ₂₄₃	T ₂₄₄	T ₂₄₅	T ₂₄₆	T ₂₄₇	T ₂₄₈	T ₂₄₉	T ₂₅₀	T ₂₅₁	T ₂₅₂	T ₂₅₃	T ₂₅₄	T ₂₅₅	T ₂₅₆	T ₂₅₇	T ₂₅₈	T ₂₅₉	T ₂₆₀	T ₂₆₁	T ₂₆₂	T ₂₆₃	T ₂₆₄	T ₂₆₅	T ₂₆₆	T ₂₆₇	T ₂₆₈	T ₂₆₉	T ₂₇₀	T ₂₇₁	T ₂₇₂	T ₂₇₃	T ₂₇₄	T ₂₇₅	T ₂₇₆	T ₂₇₇	T ₂₇₈	T ₂₇₉	T ₂₈₀	T ₂₈₁	T ₂₈₂	T ₂₈₃	T ₂₈₄	T ₂₈₅	T ₂₈₆	T ₂₈₇	T ₂₈₈	T ₂₈₉	T ₂₉₀	T ₂₉₁	T ₂₉₂	T ₂₉₃	T ₂₉₄	T ₂₉₅	T ₂₉₆	T ₂₉₇	T ₂₉₈	T ₂₉₉	T ₃₀₀	T ₃₀₁	T ₃₀₂	T ₃₀₃	T ₃₀₄	T ₃₀₅	T ₃₀₆	T ₃₀₇	T ₃₀₈	T ₃₀₉	T ₃₁₀	T ₃₁₁	T ₃₁₂	T ₃₁₃	T ₃₁₄	T ₃₁₅	T ₃₁₆	T ₃₁₇	T ₃₁₈	T ₃₁₉	T ₃₂₀	T ₃₂₁	T ₃₂₂	T ₃₂₃	T ₃₂₄	T ₃₂₅	T ₃₂₆	T ₃₂₇	T ₃₂₈	T ₃₂₉	T ₃₃₀	T ₃₃₁	T ₃₃₂	T ₃₃₃	T ₃₃₄	T ₃₃₅	T ₃₃₆	T ₃₃₇	T ₃₃₈	T ₃₃₉	T ₃₄₀	T ₃₄₁	T ₃₄₂	T ₃₄₃	T ₃₄₄	T ₃₄₅	T ₃₄₆	T ₃₄₇	T ₃₄₈	T ₃₄₉	T ₃₅₀	T ₃₅₁	T ₃₅₂	T ₃₅₃	T ₃₅₄	T ₃₅₅	T ₃₅₆	T ₃₅₇	T ₃₅₈	T ₃₅₉	T ₃₆₀	T ₃₆₁	T ₃₆₂	T ₃₆₃	T ₃₆₄	T ₃₆₅	T ₃₆₆	T ₃₆₇	T ₃₆₈	T ₃₆₉	T ₃₇₀	T ₃₇₁	T ₃₇₂	T ₃₇₃	T ₃₇₄	T ₃₇₅	T ₃₇₆	T ₃₇₇	T ₃₇₈	T ₃₇₉	T ₃₈₀	T ₃₈₁	T ₃₈₂	T ₃₈₃	T ₃₈₄	T ₃₈₅	T ₃₈₆	T ₃₈₇	T ₃₈₈	T ₃₈₉	T ₃₉₀	T ₃₉₁	T ₃₉₂	T ₃₉₃	T ₃₉₄	T ₃₉₅	T ₃₉₆	T ₃₉₇	T ₃₉₈	T ₃₉₉	T ₄₀₀	T ₄₀₁	T ₄₀₂	T ₄₀₃	T ₄₀₄	T ₄₀₅	T ₄₀₆	T ₄₀₇	T ₄₀₈	T ₄₀₉	T ₄₁₀	T ₄₁₁	T ₄₁₂	T ₄₁₃	T ₄₁₄	T ₄₁₅	T ₄₁₆	T ₄₁₇	T ₄₁₈	T ₄₁₉	T ₄₂₀	T ₄₂₁	T ₄₂₂	T ₄₂₃	T ₄₂₄	T ₄₂₅	T ₄₂₆	T ₄₂₇	T ₄₂₈	T ₄₂₉	T ₄₃₀	T ₄₃₁	T ₄₃₂	T ₄₃₃	T ₄₃₄	T ₄₃₅	T ₄₃₆	T ₄₃₇	T ₄₃₈	T ₄₃₉	T ₄₄₀	T ₄₄₁	T ₄₄₂	T ₄₄₃	T ₄₄₄	T ₄₄₅	T ₄₄₆	T ₄₄₇	T ₄₄₈	T ₄₄₉	T ₄₅₀	T ₄₅₁	T ₄₅₂	T ₄₅₃	T ₄₅₄	T ₄₅₅	T ₄₅₆	T ₄₅₇	T ₄₅₈	T ₄₅₉	T ₄₆₀	T ₄₆₁	T ₄₆₂	T ₄₆₃	T ₄₆₄	T ₄₆₅	T ₄₆₆	T ₄₆₇	T ₄₆₈	T ₄₆₉	T ₄₇₀	T ₄₇₁	T ₄₇₂	T ₄₇₃	T ₄₇₄	T ₄₇₅	T ₄₇₆	T ₄₇₇	T ₄₇₈	T ₄₇₉	T ₄₈₀	T ₄₈₁	T ₄₈₂	T ₄₈₃	T ₄₈₄	T ₄₈₅	T ₄₈₆	T ₄₈₇	T ₄₈₈	T ₄₈₉	T ₄₉₀	T ₄₉₁	T ₄₉₂	T ₄₉₃	T ₄₉₄	T ₄₉₅	T ₄₉₆	T ₄₉₇	T ₄₉₈	T ₄₉₉	T ₅₀₀	T ₅₀₁	T ₅₀₂	T ₅₀₃	T ₅₀₄	T ₅₀₅	T ₅₀₆	T ₅₀₇	T ₅₀₈	T ₅₀₉	T ₅₁₀	T ₅₁₁	T ₅₁₂	T ₅₁₃	T ₅₁₄	T ₅₁₅	T ₅₁₆	T ₅₁₇	T ₅₁₈	T ₅₁₉	T ₅₂₀	T ₅₂₁	T ₅₂₂	T ₅₂₃	T ₅₂₄	T ₅₂₅	T ₅₂₆	T ₅₂₇	T ₅₂₈	T ₅₂₉	T ₅₃₀	T ₅₃₁	T ₅₃₂	T ₅₃₃	T ₅₃₄	T ₅₃₅	T ₅₃₆	T ₅₃₇	T ₅₃₈	T ₅₃₉	T ₅₄₀	T ₅₄₁	T ₅₄₂	T ₅₄₃	T ₅₄₄	T ₅₄₅	T ₅₄₆	T ₅₄₇	T ₅₄₈	T ₅₄₉	T ₅₅₀	T ₅₅₁	T ₅₅₂	T ₅₅₃	T ₅₅₄	T ₅₅₅	T ₅₅₆	T ₅₅₇	T ₅₅₈	T ₅₅₉	T ₅₆₀	T ₅₆₁	T ₅₆₂	T ₅₆₃	T ₅₆₄	T ₅₆₅	T ₅₆₆	T ₅₆₇	T ₅₆₈	T ₅₆₉	T ₅₇₀	T ₅₇₁	T ₅₇₂	T ₅₇₃	T ₅₇₄	T ₅₇₅	T ₅₇₆	T ₅₇₇	T ₅₇₈	T ₅₇₉	T ₅₈₀	T ₅₈₁	T ₅₈₂	T ₅₈₃	T ₅₈₄	T ₅₈₅	T ₅₈₆	T ₅₈₇	T ₅₈₈	T ₅₈₉	T ₅₉₀	T ₅₉₁	T ₅₉₂	T ₅₉₃	T ₅₉₄	T ₅₉₅	T ₅₉₆	T ₅₉₇	T ₅₉₈	T ₅₉₉	T ₆₀₀	T ₆₀₁	T ₆₀₂	T ₆₀₃	T ₆₀₄	T
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RUN X. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1415	Dist.	1.3290	97.75	
1425	Dist.	1.3290	97.75	
1515	Dist.	1.3290	97.75	
1527		1.3290		
1536	x-4	1.3403	66.00	0.523
1538		1.3403		
1559	x-3	1.3392	70.50	0.583
		1.3392		
1601	x-2-L	1.3339	88.85	0.813
1604		1.3339		
1606	x-1	1.3311	93.85	0.895
1609		1.3311		
1613	y-4	1.3400	67.25	0.537
1616		1.3401		
1619	y-3	1.3352	86.00	0.775
1621		1.3351		
		1.3351		
1629	y-2	1.3310	94.10	0.899
		1.3311		
	y-1		97.75	0.958
1636	Dist.	1.3291	97.50	
	x-2-R		80.75	
1840	Dist.	1.3290	97.75	
1855	x-4	1.3410	62.00	0.523
1859		1.3410		
	x-3	1.3404	66.00	0.523
		1.3403		
1906	x-2-L	1.3349	86.65	0.778
		1.3349		
1912	x-1	1.3320	92.25	0.872
1915		1.3320		
1918	y-4	1.3410	62.00	0.480
		1.3410		
1933	y-3	1.3362	82.15	0.720
		1.3361		
1938	y-2	1.3321		
1945		1.3320	96.25	
		1.3321		
1950	Dist.	1.3298	96.25	
		1.3299		
	y-1	1.3285	98.00	0.968
2005	Dist.	1.3299	96.25	
	x-2-R		80.90	0.704



RUN XI.

Kettle Charge
3/16" Hole Diameter
1.17% Free Space Area

Time	Thermocouple Designation	Temperature °F											Pressure				Roto Rdg			
		1	2	4	5	6	7	8	9	10	11	1	2	3	4	Orifice In. H ₂ O	Kettle Jacket (steam) psig	Reflux	Condens-	ser
1500	Steam to pot																			
1645	93 147.8 152.2 149.9 132.8 154.8 - 190.0 - - 0.2 0.9 1.6 2.2 2.5 38 8.0																			
1730													0.2	0.95	1.6	2.2	2.5	39	8.0	
1900	96.5 140.0 152.6 150.6 133.7 154.8 - 195.9 - - 0.19 0.9 1.6 2.2 2.4 43 7.5																			
1917																				
1945	118.4 147.0 153.4 151.8 135.0 156.8 - 195.9 - - 0.1 0.4 0.7 1.0 24 4.0																			
2130	Steam off																			

TIME	SAMPLE DESIG.	N _D 25°C		WEIGHT % MEOH	MOL FRACT. MEOH
		1.3299	96.1		
1700	Dist.	1.3299	96.1		
1730	x-4	1.3405			
		1.3406	64.7	0.508	
		1.3407			
1735	x-3	1.3408	63.3	0.492	
1745		1.3409			
1753	x-2-L	1.3361			
1758		1.3361	82.5		
		1.3361			
1805	x-1	1.3332	90.2	0.837	
1807		1.3332			
1809	x-2-R	1.3382	74.4	0.621	
1815		1.3382			
1821	y-4	1.3409	62.75	0.487	
1824		1.3409			
1827	y-3	1.3385	73.0	0.603	
1830		1.3386			
1837	y-2	1.3340	88.6	0.815	
		1.3340			
1847	Dist.	1.3304	95.1		
1900		1.3305			
1852	y-1	1.3292	97.1	0.95	
1915		1.3294			
1946	Dist.	1.3305	95.0		
1940					
1944	Dist.	1.3305	95.0		
1955		1.3305			
2014	x-3	1.3405	65.0	0.512	
		1.3405			
2025	x-2-L	1.3381	74.75		
		1.3380			
2040	x-2-R	1.3396	69.00	0.554	
2045		1.3396			
2047	x-2-L	1.3381	74.75		
	x-1	1.3345	87.60	0.798	
2055	y-4	1.3399	68.00	0.553	
2057		1.3399			
2100	y-3	1.3396	69.00	0.554	
2105		1.3396			
	y-2	1.3346	87.30	0.795	
2112	Dist.	1.3310	94.10	0.899	
		1.3310			
	y-1		94.1	0.899	

RUN XI. SAMPLE DATA

TIME	SAMPLE DESIG.	N _D 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1700	Dist.	1.3299	96.1	
1730	x-4	1.3405		
		1.3406	64.7	0.508
		1.3407		
1735	x-3	1.3408	63.3	0.492
1745		1.3409		
1753	x-2-L	1.3361		
1758		1.3361	82.5	
		1.3361		
1805	x-1	1.3332	90.2	0.837
1807		1.3332		
1809	x-2-R	1.3382	74.4	0.621
1815		1.3382		
1821	y-4	1.3409	62.75	0.487
1824		1.3409		
1827	y-3	1.3385	73.0	0.603
1830		1.3386		
1837	y-2	1.3340	88.6	0.815
		1.3340		
1847	Dist.	1.3304	95.1	
1900		1.3305		
1852	y-1	1.3292	97.1	0.95
1915		1.3294		
1946	Dist.	1.3305	95.0	
1940				
1944	Dist.	1.3305	95.0	
1955		1.3305		
2014	x-3	1.3405	65.0	0.512
		1.3405		
2025	x-2-L	1.3381	74.75	
		1.3380		
2040	x-2-R	1.3396	69.00	0.554
2045		1.3396		
2047	x-2-L	1.3381	74.75	
	x-1	1.3345	87.60	0.798
2055	y-4	1.3399	68.00	0.553
2057		1.3399		
2100	y-3	1.3396	69.00	0.554
2105		1.3396		
	y-2	1.3346	87.30	0.795
2112	Dist.	1.3310	94.10	0.899
		1.3310		
	y-1		94.1	0.899

АГАСТИКИМЕ · IX · 11

APPENDIX G. BILBIOGRAPHY

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