A MINIATURIZED STEADY-STATE FRACTIONAL DISTILLATION UNIT FOR RESEARCH AND PILOT PLANT STUDIES

Thesis for the Degree of M. S.

MICHIGAN STATE UNIVERSITY

Darcy Antonio Paviani

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ABSTRACT

A MINIATURIZED STEADY-STATE FRACTIONAL DISTILLATION UNIT
FOR RESEARCH AND PILOT STUDIES

by Darcy A. Paviani

Simulation of industrial fractionation units on a laboratory or small pilot plant scale is often more difficult than designing the commercial scale units themselves.

The small unit requires all the major operational features of the industrial tower; such as provision for moving and controlling flows of feed, overhead and bottom products, reflux, and boilup. In addition, small units suffer from special problems related to their size. Heat losses tend to be excessive in relation to throughput capacity, and equipment for pumping, metering, and controlling flow is often less than satisfactory. Also, because of their research nature it is desirable that small units have certain other features such as visibility, ease of modification, and the ability to operate with small supplies of feedstock.

A miniaturized steady-state fractional distillation unit has been developed, designed, and constructed with a view toward incorporating the desired features. Top and bottom products are moved, metered, and controlled by the wattage input to electric heaters. Here the streams are vaporized and pass to an overhead condenser where they are

combined to give a gravity-flow feed stream. Adiabatic operation is attained by a compact design which permits all the equipment except condensers to be enclosed in electrically heated air jackets. Visibility is obtained by making the column and jackets of glass. Machined metal plate assemblies fitted with teflon gaskets into a precision-bore glass column are easily removed for modification or replacement.

A set of sieve trays was designed and constructed using the design equations usually applied to larger columns. The trays were tested in the column with ethanol, benzene, methanol, and carbon tetrachloride. Visual observation and pressure drop measurements were both consistent with design, indicating good correlation with the hydrodynamics of larger scale equipment.

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By

Darcy Antonio Paviani

A THESIS

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PART I

INTRODUCTION

INTRODUCTION

A - Purpose of Miniaturizing Distillation Units

The purpose of having small scale units which simulate their industrial counterparts is crystallized in a sentence by L. H. Baekeland:[2] "Commit your blunders on a small scale and make your profits on a large scale."

In the field of distillation, trials in a laboratory are far more economical than performing the same operation with the full scale prototype. Laboratory models which simulate the same conditions existing in a large commercial tower have a number of potential uses.

1) Process Research

While many distillation column designs can be calculated with assurance from vapor-liquid equilibrium data, it is still desirable to prove many distillation designs. This is particularly true where liquid-vapor equilibrium relations are not well established as in azeotropic systems and where tautomeric shifts or other chemical reactions occur. Also, it often proves desirable to pilot a complete chemical process on a small scale including those parts of the process involving distillation. Since steam requirements for distillation often play an important part in the over-all economics of the process, it is desirable that such measurements be made accurately on a pilot plant scale.

2) Equipment Research

Many new designs for distillation column plates have been appearing recently, including a variety of types of sieve plates with and without downcomers, grid plates, baffle plates, and so forth.[5] Important information can be obtained by evaluating the efficiency of these various plate designs on a miniaturized scale and by observing, visually, the performance of these plates.

3) Teaching Purposes

It is much easier for a student to observe what is happening in a distillation unit six or eight feet tall than in one which is several stories in height. This is particularly true if the small unit can be constructed of glass. The small unit is also easily transported and could be used as a lecture demonstration unit.

A small unit can also be brought into equilibrium faster, and relatively small quantities of utilities and feed stocks are required.

B - Problems in Miniaturizing Distillation Units

When industrial type distillation columns are scaled down to laboratory or small pilot plant size, several problems of importance arise. Some of these problems are outlined below.

1) Steady-State Operation

Since industrial distillation units operate in a steady-state with continuous feed and product draw-offs, it follows that the miniatures of these columns should operate in the same manner. A miniature column

must, therefore, be distinguished from a laboratory column which is primarily used for the purpose of making laboratory separations. Such laboratory columns give very little information that is useful in the design of industrial scale units. Miniaturized units for the purpose of simulation require all the major characteristics of the industrial tower, such as provision for moving and controlling feed rates, overhead and bottom product rates, reflux and boilup.

2) Pumping Mechanism and Metering Devices

The pumping and metering equipment available for the small flows encountered in a miniaturized unit are usually bulky and clumsy when compared with those used in industrial scale equipment. Often an entirely different type of device is used. Where a centrifugal pump is used for large flows, a pulsating reciprocating type may be best for small flows. Small flow differential-type metering devices often behave erratically especially when solid impurities collect in orifices, venturis, and so on. Valves with suitable flow characteristics are generally not available in extremely small sizes.

3) Heat Losses

The operation of large columns is substantially adiabatic even without insulation, because heat losses through the wall are such a small proportion of the total heat input to the column. This is due to their large throughput-capacity to external surface ratio. Vapor generated in the reboiler moves up in the column at a relatively constant molar rate. In small columns, where heat losses are large relative to the throughput, the vapor flow decreases as the vapor moves from the bottom of the column to the top. Unless careful provisions for heat losses are made, the reflux ratio at the top of the

column will not be a measure of the liquid-vapor ratio in the column. Sometimes the amount of heat lost by the accessories is as important as the heat loss from the column itself. Reboilers, pipes for vapor and liquid flow, metering devices, pumps, etc. must also be jacketed to minimize heat losses.

4) Feedstock

The quantity of feedstock available for process research studies is often highly limited. The pilot plant must give all the design information before the supply of feedstock runs out. It is essential, therefore, that a satisfactory miniaturized column require a minimum of feed material for operating and bringing the column into equilibrium.

5) Visibility

In research, visual observation is an important feature. Industrial units are designed for specific purposes and there is not an immediate need to observe what is happening inside. Also, in industrial towers, due to their size, it is easy to adapt glass openings or manholes for observation and accessibility. Small glass columns give good visibility. However, to construct a small glass replica of a large scale column of sophisticated design is difficult.

6) Easy Modification

Again in research, the evaluation of different design factors and physical conditions requires a flexible and easily modified unit. Tray design must be readily capable of modification. Access to the inside of the column for cleaning must be included in the design of the column.

7) Composition Analysis

In industrial columns, due to their large throughputs, fluid samples can be taken out for analysis without disturbing the operation of the column appreciably. When similar samples are taken from a miniaturized column, flow rates are changed considerably. Special analytical methods have to be used for these small scale columns.

8) Internal Design

The internal design of the distillation column itself can employ bubble cap plates, sieve plates, or packing. In the case of bubble cap design, it is generally very difficult to simulate the geometry existing in industrial towers with bubble caps three to six inches in diameter. On a miniature scale where the column itself is only one or two inches in diameter, simulation with sieve trays is much easier. Small scale columns are very adaptable to the use of packing materials, but packing materials, particularly the types used in small scale columns, are not used very much in industrial columns.

9) Stage Efficiency

In small diameter columns, stage efficiencies are usually lower than in their industrial counterparts. This is because the amount of cross flow between vapor and liquid streams is low. As Figure 1 shows, the Murphree plate efficiency (E_p) becomes considerably larger than the local Murphree efficiency (E_L) for larger diameter plates. For small diameter plates, these two efficiencies are typically equal to each other.

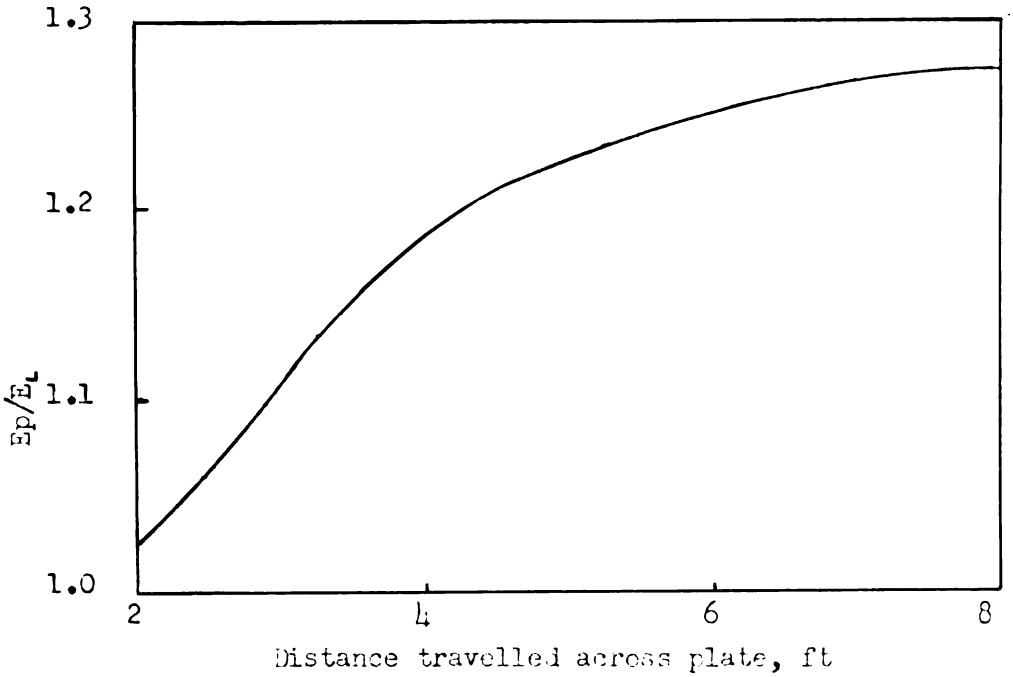


Figure 1. Influence of liquid path on plate efficiency. (From Cooper[6])

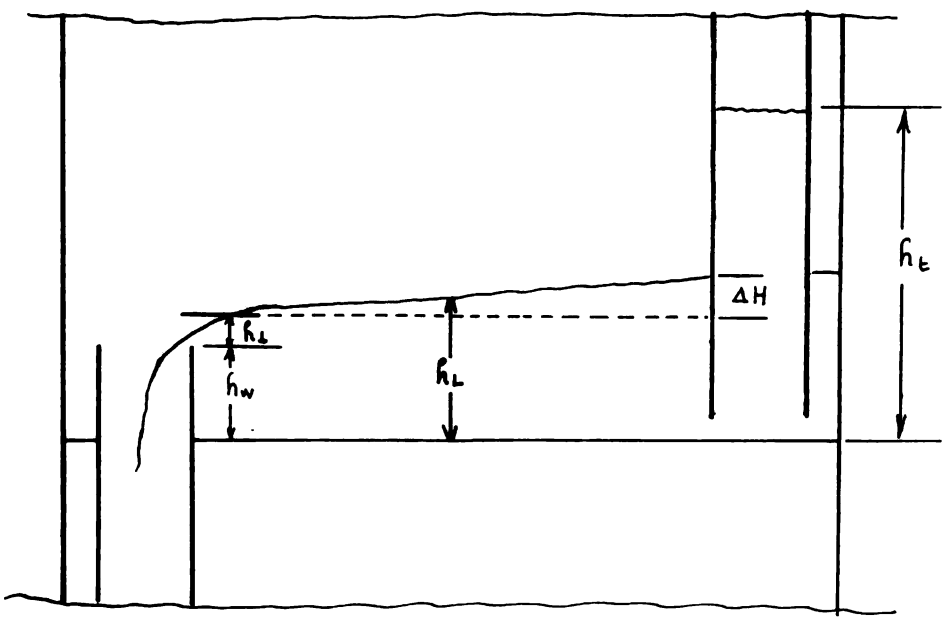
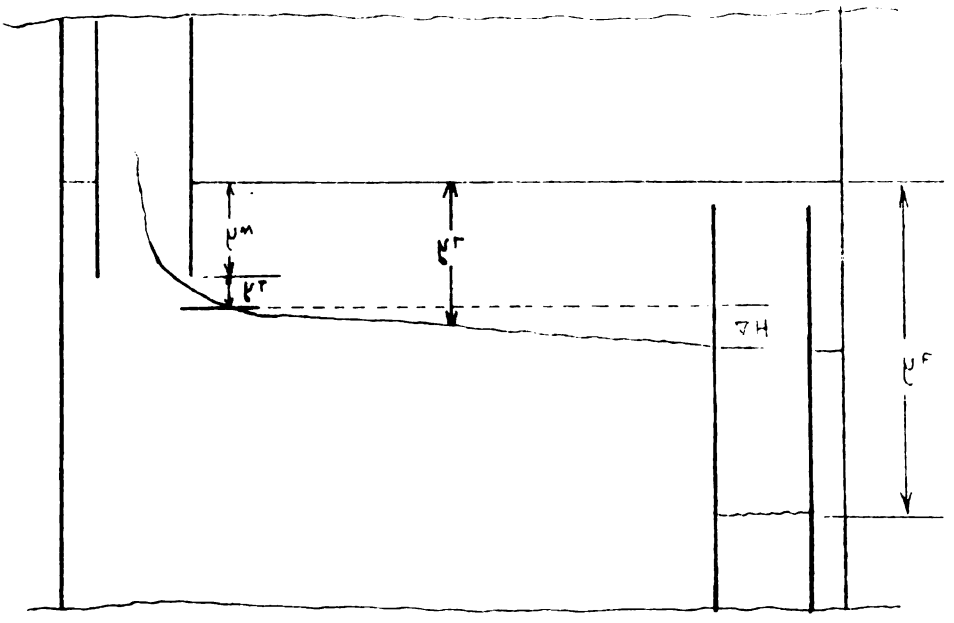
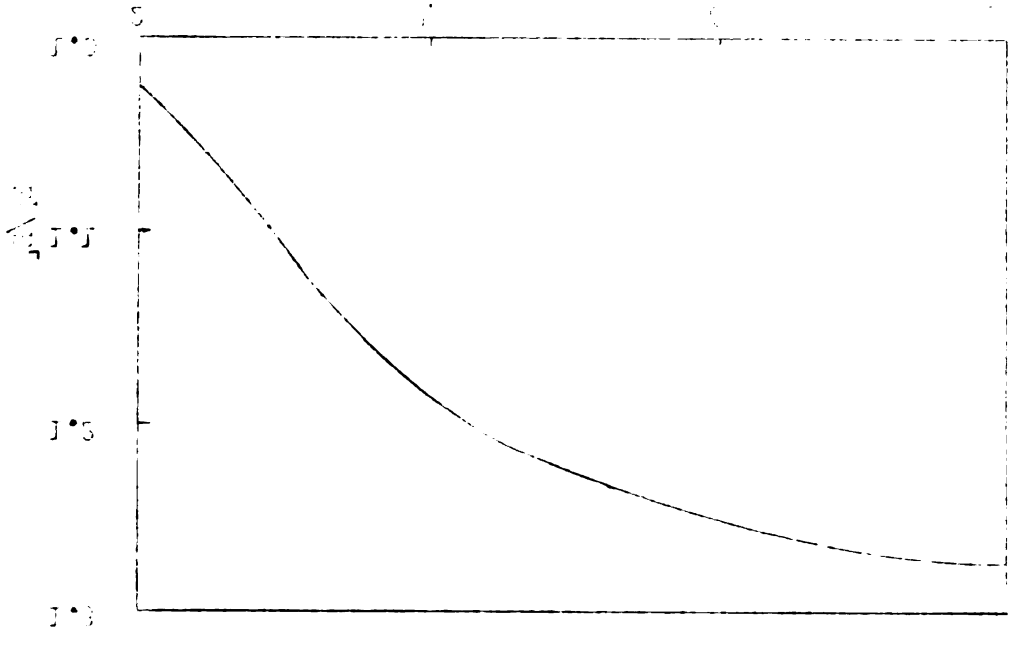


Figure 2. Hydraulic relationships on a sieve plate.





PART II

PREVIOUS WORK WITH SMALL SCALE FRACTIONATORS

PREVIOUS WORK WITH SMALL SCALE FRACTIONATORS

Usually continuous small scale fractionators have diameters of four to eight inches.[6] During the literature survey of this research project, no reference was found to a continuous column with a diameter smaller than four inches.

A considerable amount of the current batch laboratory fractionating apparatus was developed during the 1930's. The expressed objectives in designing such apparatus was high efficiency, low hold-up, and low pressure drop as required for accurate analytical fractionations. This kind of apparatus is not suitable for the study of design factors. Only recently, investigators started to be more involved with small fractionators for the purpose of scaling up.[3]

1) Packed Columns

Small packed columns generally have the advantage of good efficiency, high throughput, low hold-up, and low pressure drop. A variety of packing materials have been used in the past. One-, two-, and six-turn wire helices, glass helices, carding teeth, Cholet-Vanderhoef rings, Lessing rings, jack chains, rivets, and so forth may be mentioned as examples.[7] Tongberg, Lawroski and Fenske[31] found that a single turn wire made of No. 30 B&S gage wire with an inside diameter of 3/32 inches gave the best results. With this packing, 100-theoretical-plate columns with a H.E.T.P. varying between one and two inches were easily built. Glass helices are somewhat less efficient, but they are highly resistant

to corrosion.

2) Plate Columns

Bruun[4] developed an all-glass bubble cap column with external downcomers for laboratory use. Its H.E.T.P. is about 0.75 inches, but it has a high pressure drop per plate. Krell[16] described a glass bubble cap laboratory column with internal downcomers for simulation of industrial operations. It contains one bubble cap per plate and is fitted with thermometers and sample taps.

The all-glass Oldershaw[25] perforated tray column is an improvement over the Bruun column in regard to efficiency and pressure drop. Holes are approximately 0.035 inches in diameter arranged in circles. The downcomer is located in the center of the tray and discharges the liquid on a lateral side of the tray below. One disadvantage of the Oldershaw column is that surface tension effects are magnified by the small hole diameters. The column does not operate properly with high surface tension liquids, such as water.

Plates in the Oldershaw column are perforated with red-hot tungsten wire and sealed in the column. The construction of the Oldershaw column is difficult and simplified versions of it have been reported.[17] In cases where corrosion is not important, the glass trays may be replaced by metal plates of easier construction.

Recently, Meyers[22] reported a variation of the sieve plate, called a perfo-drip tray column. Downcomers are absent and liquid drips intermittently through the holes.

3) Insulating Jackets

Thermal insulation of laboratory columns was extensively studied by Podbielniak.[26] Sleeved laboratory fractionating columns lagged with asbestos in which air at the temperature of the reboiler was blown into the lower end of the jacket, were described by Leslie and Geniesse.[18] A good review of the different types of insulating jackets is given in Weissberger.[36] Present day techniques for insulation of laboratory columns are not much different than those described by these authors.

Podbielniak[26] stated that completely silvered vacuum jackets and metal partition vacuum jackets are almost perfect insulators. However, in actual practice they present limitations. In long tubes, the differential expansion of the inner and outer tubes, bows the inner tube until fracture occurs. The use of expansion bellows is not effective and fused quartz is too expensive. Visibility in research equipment is indispensable, and none of the jackets mentioned above provides it to a full extent.

Electrically heated air jackets were improved by Todd.[30] They provide highly adiabatic conditions and excellent visibility especially under conditions where radiation shields may be dispensed with.

PART III

DESCRIPTION OF THE APPARATUS

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1 - Adiabatic Air Jacket

Two four-foot long concentric cylindrical glass tubes were used to provide for a heated air layer around the distillation unit. The outer tube has a 5.9 inch outside diameter and is 0.138 inches thick. This glass assembly encloses and accommodates the whole distillation unit with the exception of the condensers, as shown in Figure 5. Three thermometers, graduated from minus 10°C to 310°C, hooked on the inside wall of the inner jacket measure the temperature of the surroundings of the distillation column. The bulbs of the thermometers are located respectively at 1, 2, and 3 feet from the bottom of the column. Two 13-ohm electrical resistances, each one made of 20 feet long No. 20 B&S Nichrome wire, span the outside wall of the inner jacket as shown in Figure 3. The assembly of the resistances is held on the glass tube by means of two rows of hooks equally spaced around both ends of the glass tube. Variacs control the power input of the resistances in order to keep the temperature outside of the column equal to the temperature inside.

2 - Heating Element Assemblies

The immersion heating element is formed by a length of No. 24 B&S Nichrome wire, coiled on a specially designed ceramic core. The

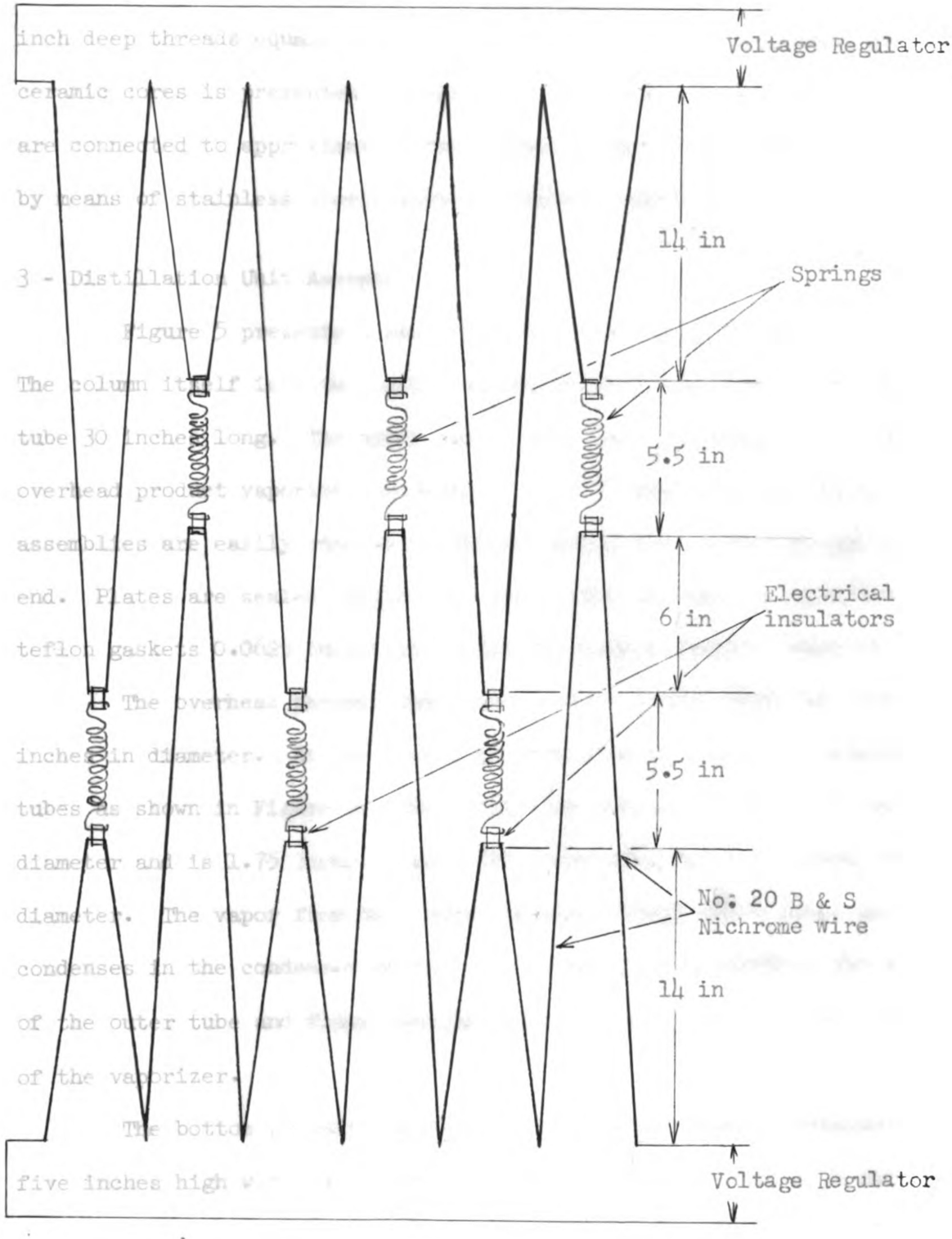

Figure 3. Resistance assembly for the air jacket.

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wire coil is held on the surface of the ceramic core by means of 0.01 inch deep threads equally spaced on the ceramic core. A drawing of the ceramic cores is presented in Figure 4. Both ends of the Nichrome wire are connected to approximately two inches of No. 18 B&S tungsten wire, by means of stainless steel sleeves directly compressed on the connections.

3 - Distillation Unit Assembly

Figure 5 presents a sketch of the entire distillation unit. The column itself is a two inch internal diameter precision bore glass tube 30 inches long. The upper end of this tube is connected to the overhead product vaporizer by means of a 55/50 taper joint. Plate assemblies are easily removed or placed inside the column through this end. Plates are sealed against the glass wall by means of machined teflon gaskets 0.0625 inches thick and 2.0 inches outside diameter.

The overhead product vaporizer is four inches high and three inches in diameter. At the center of this container are two concentric tubes as shown in Figure 5. The inner tube has a 0.75 inch internal diameter and is 1.75 inches high. The outer tube has a 1.0 inch internal diameter. The vapor from the column passes through these tubes and condenses in the condenser above. The liquid is collected on the wall of the outer tube and flows down between the tubes and into the container of the vaporizer.

The bottom product vaporizer is an annular shaped container five inches high with outer and inner diameters of 2.2 and 0.75 inches respectively. The heater of the bottom product vaporizer is mounted on a glass tube. This tube has a 0.75 inch inside diameter and runs all

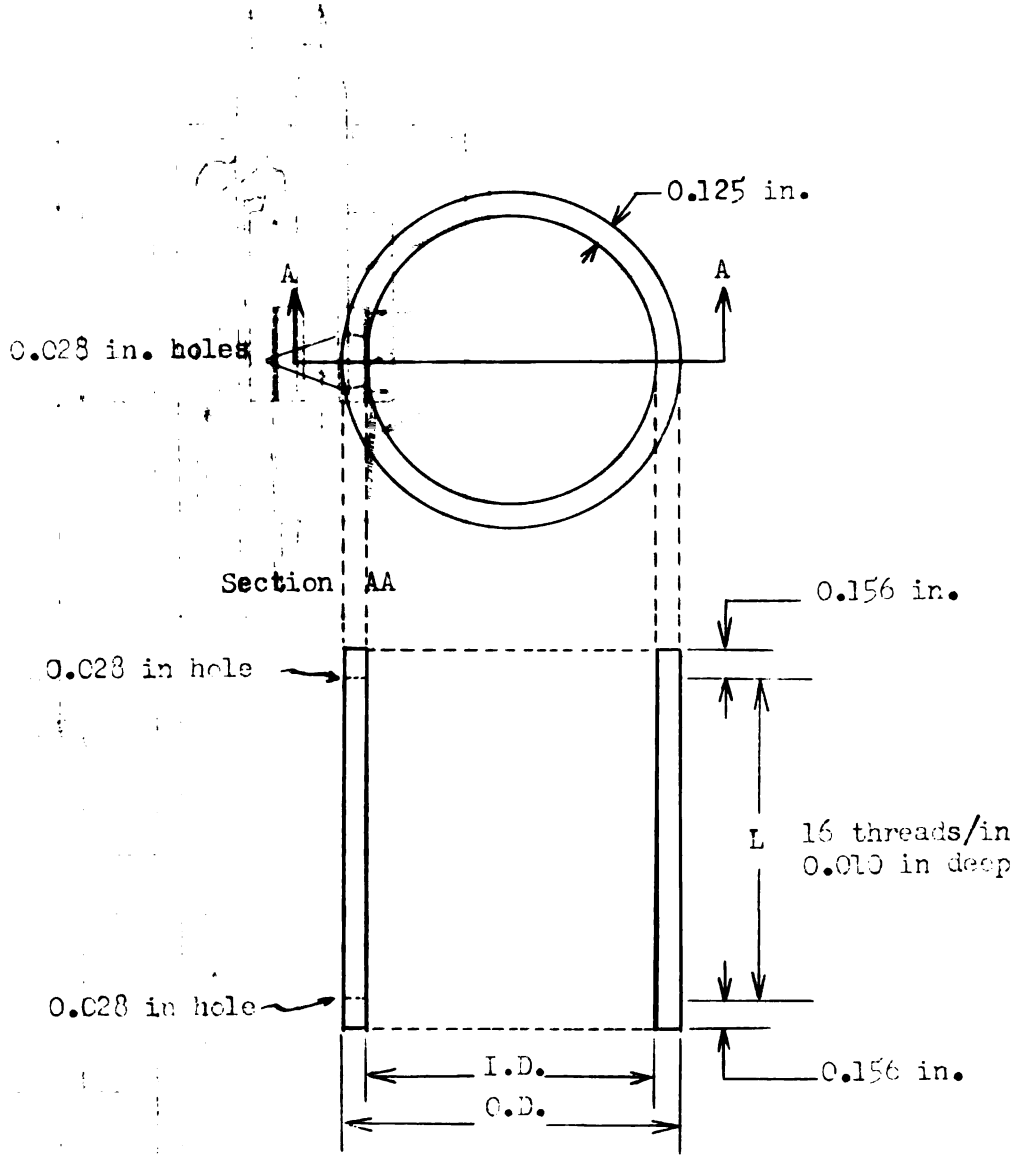
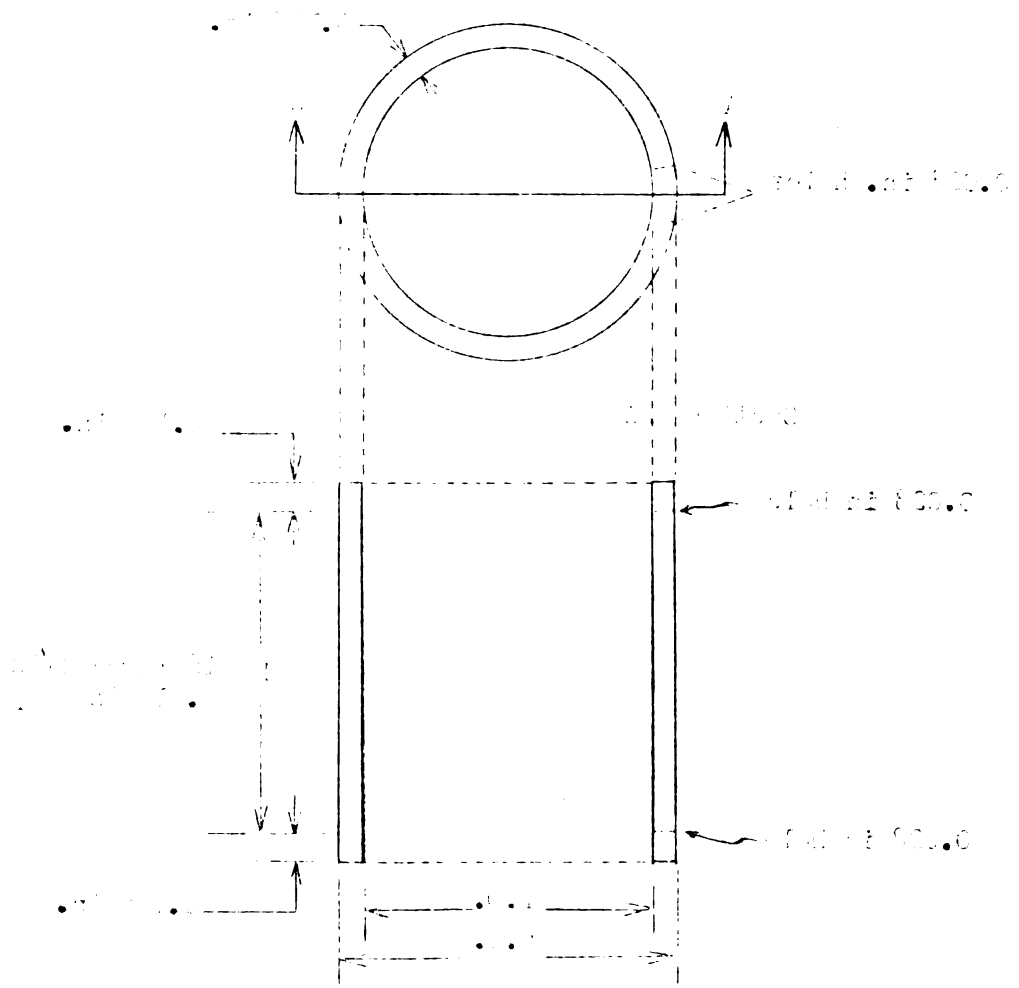


Figure 4. A typical ceramic heater core.



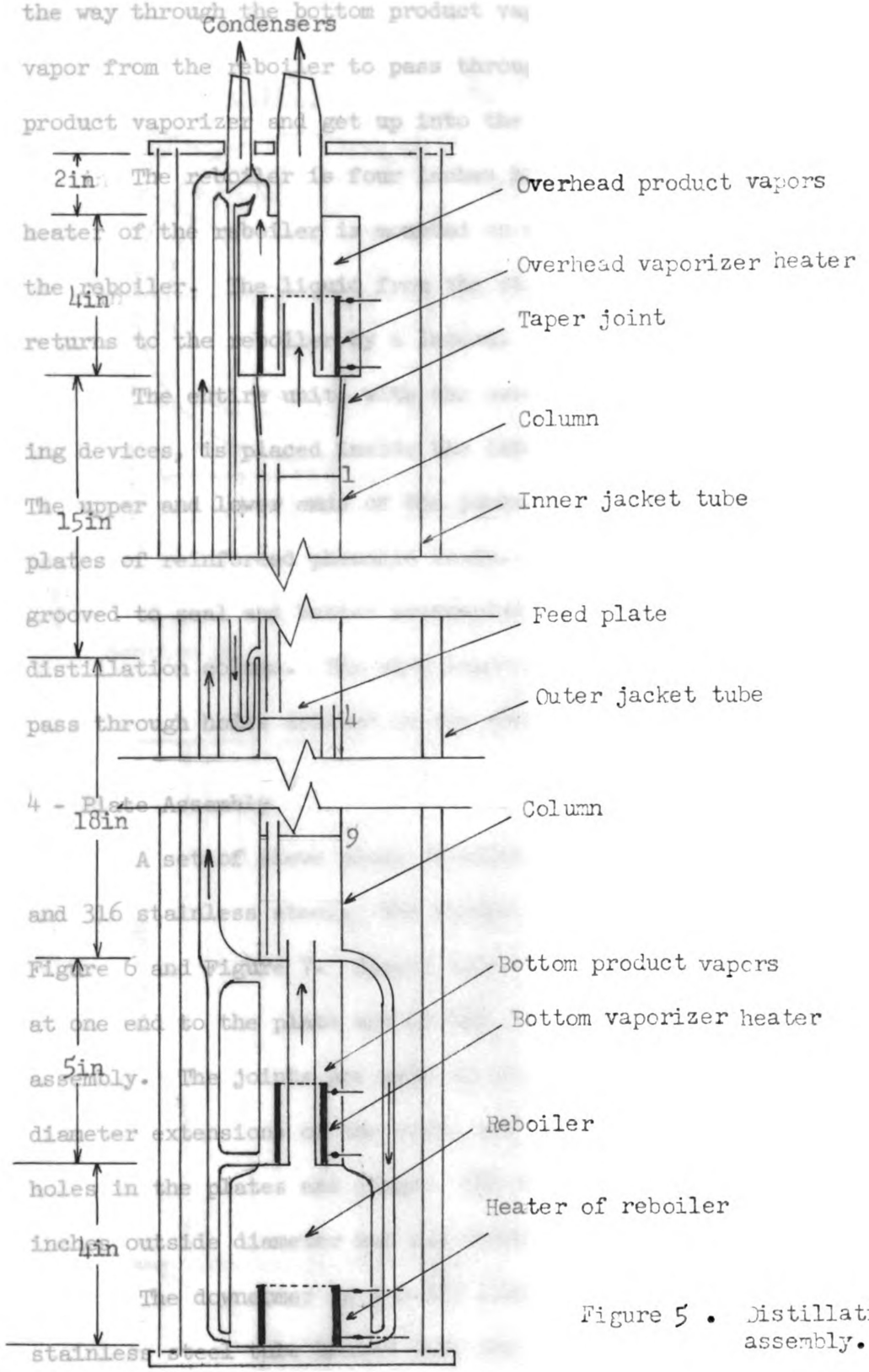


Figure 5 . Distillation unit assembly.

2in
4in
12in
18in
5in
4in

the way through the bottom product vaporizer. This tube permits the vapor from the reboiler to pass through the center tube of the bottom product vaporizer and get up into the bottom of the column.

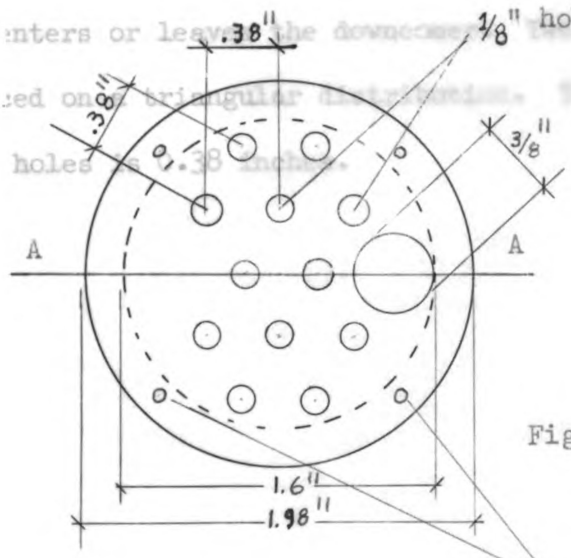
The reboiler is four inches high with a 3.5 inch diameter. The heater of the reboiler is mounted on a glass assembly on the bottom of the reboiler. The liquid from the stripping section of the column returns to the reboiler by a lateral tube as shown in Figure 5.

The entire unit, with the exception of the condensers and metering devices, is placed inside the inner tube of the adiabatic jacket. The upper and lower ends of the jacket are closed by means of two plates of reinforced phenolic resin. These plates are conveniently grooved to seal and better accommodate the tubes of the jackets and distillation column. The wire leads and the two tubes to the condensers pass through holes drilled in the phenolic plates.

4 - Plate Assembly

A set of sieve plate assemblies was constructed with Types 304 and 316 stainless steel. The design and dimensions are sketched in Figure 6 and Figure 7. Spacer rods 3/16 inch in diameter are attached at one end to the plate and at the other to the ring at the bottom of the assembly. The joints are made by drilling small holes in 1/16 inch diameter extensions of the rods, and expanding these extensions into holes in the plates and rings. The ring is 0.046 inches thick, 1.98 inches outside diameter and 1.6 inches inside diameter.

The downcomer is a 0.375 inch outside diameter, 0.028 inch thick stainless steel tube brazed into the plate. Both ends of the downcomer have 1/16 inch semicircular cuts as shown in Figure 7. The purpose of



Number of holes	12
Free area	4.7%
Downc. free area	2.5%
Pitch/diameter	3.04

Figure 6. Top view of plate assembly.

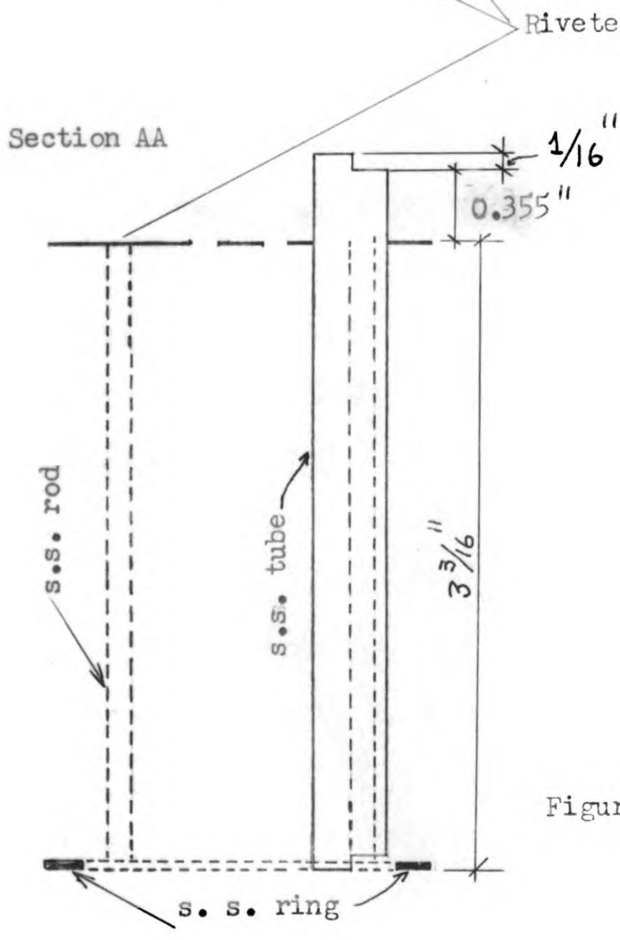
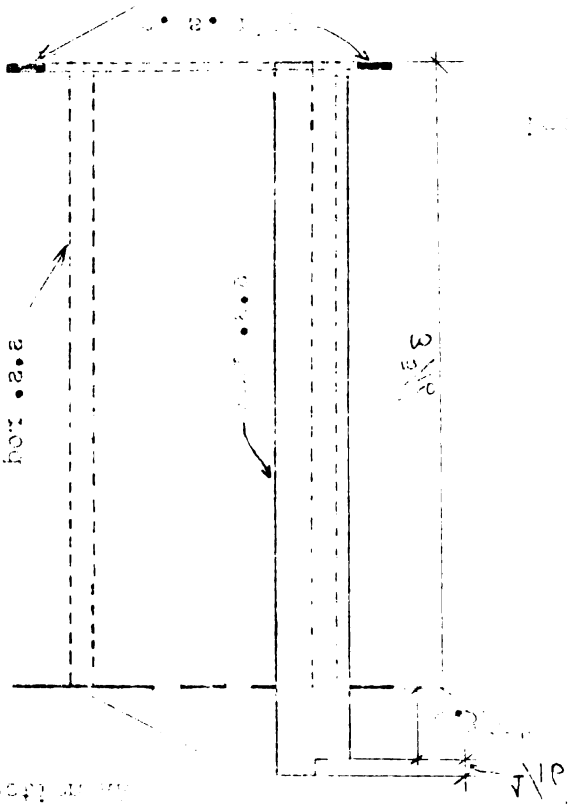
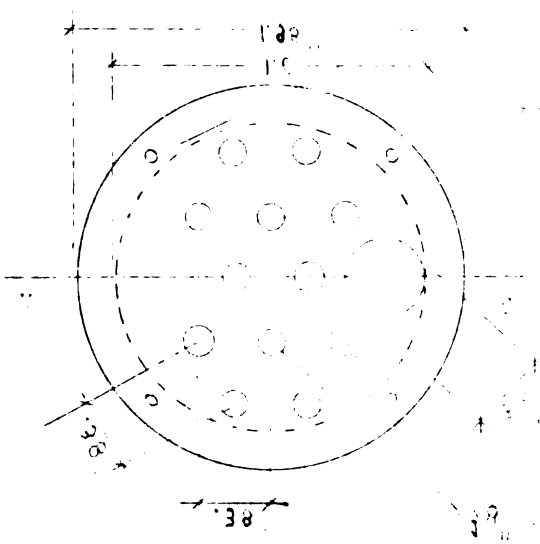
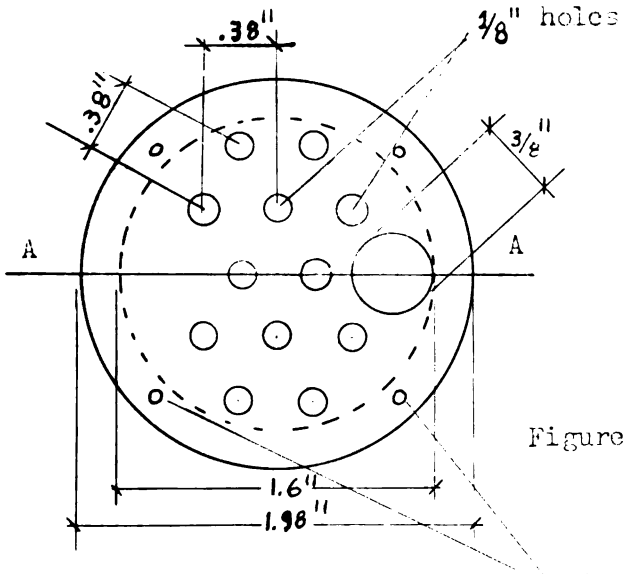


Figure 7. Cross-section view of plate assembly.





these cuts is to direct the liquid flow to the side of the column as it enters or leaves the downcomer. Twelve 0.125 inch holes are uniformily spaced on a triangular distribution. The center-to-center spacing of the holes is 0.38 inches.



Number of holes	12
Free area	4.7%
Downc. free area	2.5%
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Figure 6. Top view of plate assembly.

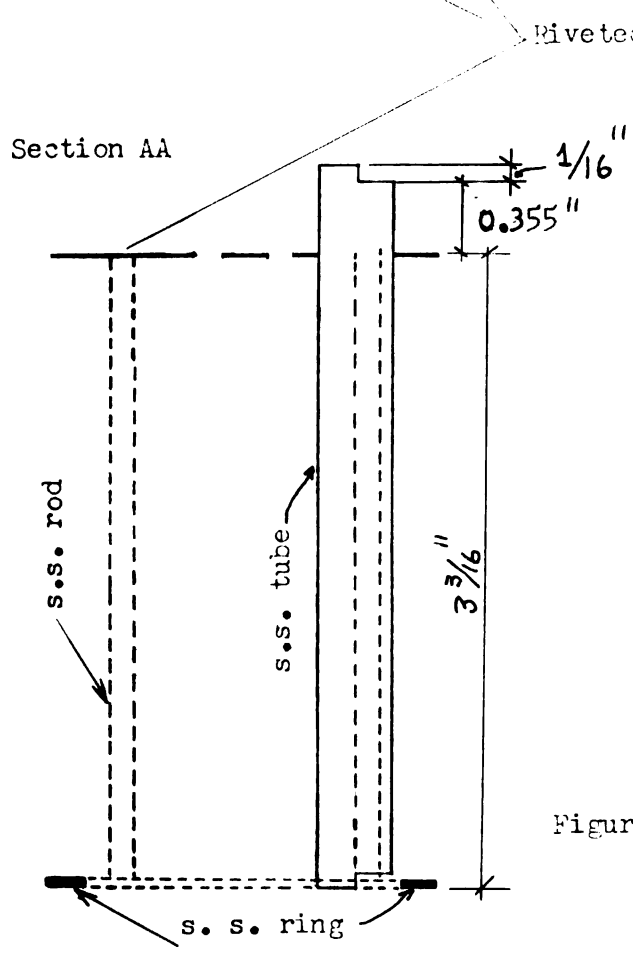
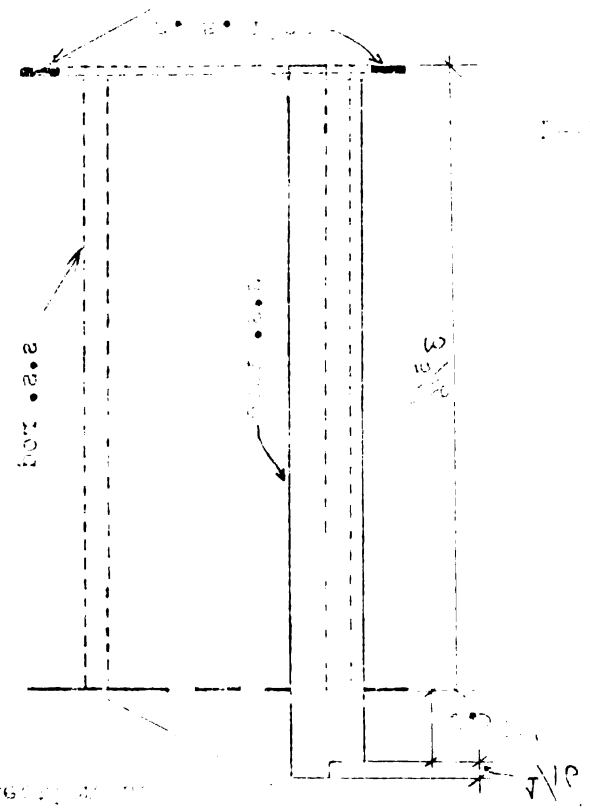
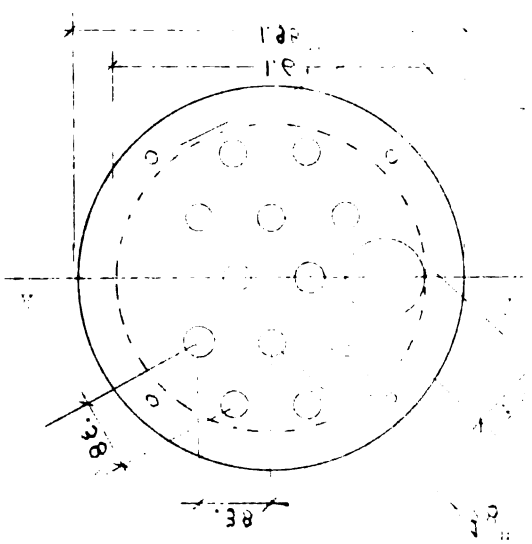


Figure 7. Cross-section view of plate assembly.





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PART IV

DEVELOPMENT AND EXPERIMENTAL WORK

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1 - Adiabatic Air Jacket

All parts of the unit excluding condensers and controlling devices are enclosed in an electrically heated air jacket. The jacket contains an upper and lower heater separately controlled to take into account the different temperatures in the top and bottom parts of the column. Each heater is a single Nichrome wire arranged in a zig-zag pattern to promote coverage and visibility. The temperatures in the jacket are controlled by Variacs so as to be the same as the temperatures in the column. By this, conduction and convection are eliminated and radiation, at least at low temperatures, is negligible.[26]

Experiments were performed by the author with a Todd[30] laboratory fractionating column to gain experience and observe the operation of the insulating jacket. This jacket was formed by two concentric pyrex tubes 39.4 inches long. The outer tube had a diameter of 2.95 inches and two-thirds of it was surrounded by a polished steel jacket. The clearance between the glass tubes was 0.44 inches. The reboiler was not jacketed. Vapor from the reboiler flowed through a three inch long glass neck before entering the insulating jacket at the bottom of the packed column. The over-all heat transfer coefficient between jacket and surroundings was found to be 0.813 Btu/hr ft^2°F at 212°F and 1.17 Btu/hr ft^2°F at 412°F.

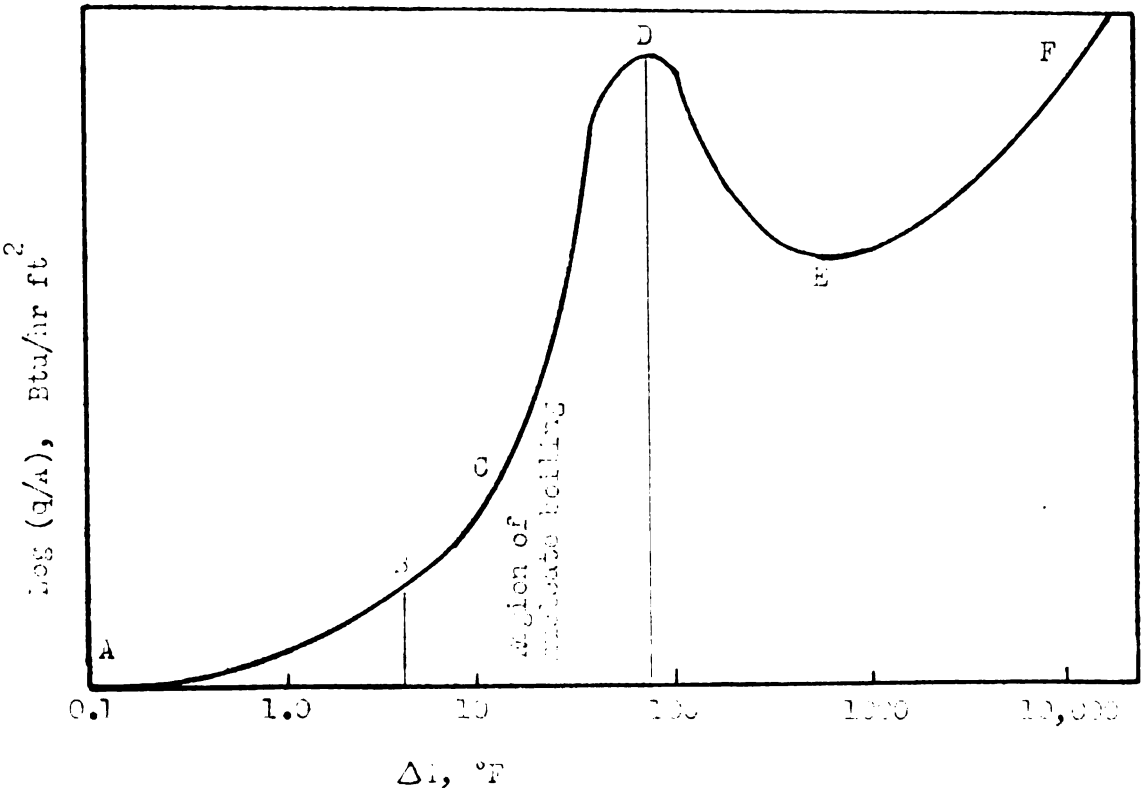
The glass jacket proved to be effective on the basis of semi-quantitative determinations, as follows: The column was run with distilled water and the number of drops at the top and bottom of the column were counted. At total reflux, the number of drops at the bottom was a little larger than at the top. At zero reflux, water continued to drip at the bottom at the rate of 2.5 - 3 drops/minute. Even when the temperature in the jacket was increased, the dropping at the bottom could not be stopped. This indicated that essentially all the condensation was taking place in the neck of the reboiler and not in the column itself.

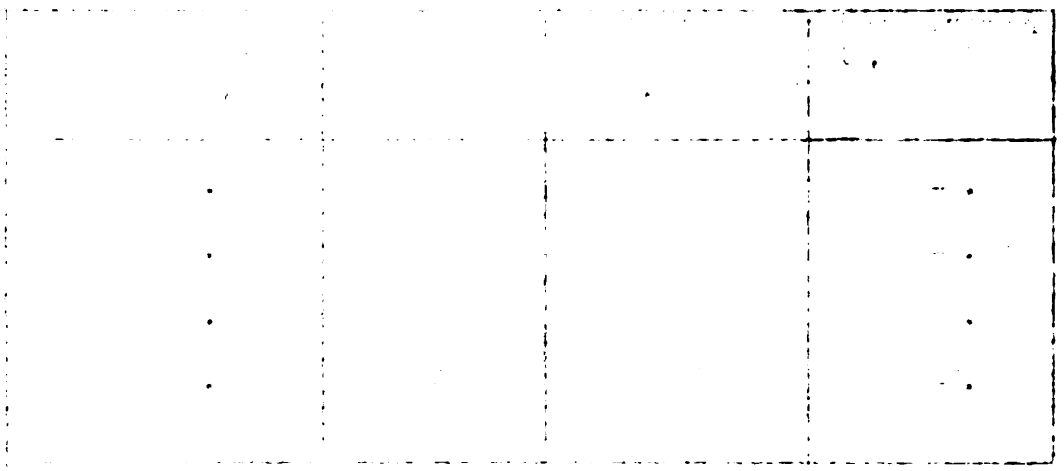
Heat losses from the present unit were determined in the following manner: The temperature of the jacket was set at 0° to 1.5°C over the temperature in the column. A fixed wattage was input to the overhead vaporizer. The wattage to the reboiler was raised in steps until the column could be operated for two hours without depletion of liquid in the overhead vaporizer. Results obtained with pure methanol in the column are shown in Table I. The wattage input to the top of the column was about five watts lower than the input to the bottom. However, there appeared to be a small amount of spilling from the condenser into the top of the column. About six drops per minute of methanol could be observed running down the column wall at the top. On the basis of 20 drops of methanol per cc., six drops per minute correspond to about 4.1 watts. From this result, it may be concluded that at boiling methanol temperatures, the column operated within one or two watts of completely adiabatic conditions.

Table I. Effective rates of the Adiabatic Air Jacket for Air and

Temperature in the jacket, °C	Rate of heat to overhead vaporizer, watts	Rate of heat to reboiler, watts	Rate of heat to system, watts
65.5–66	[illegible]	[illegible]	1.0
65.5–66	214	850	1.2
65.5–66	113	110	1.0
65.5–66	225	130	1.0

Figure 3. Characteristic boiling curve.





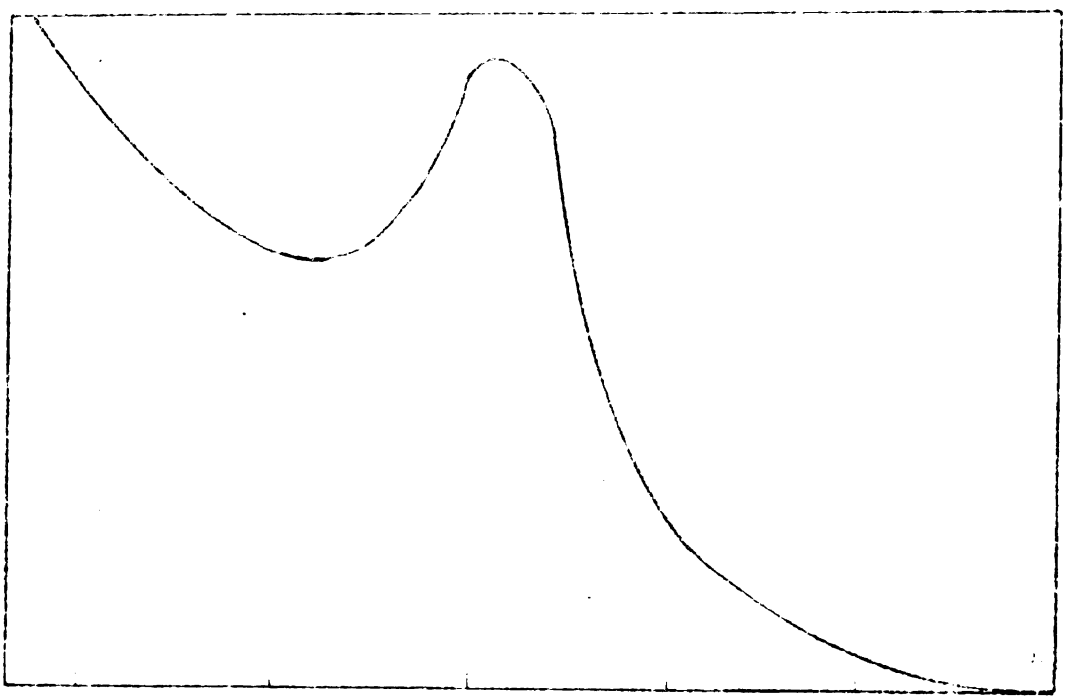


TABLE II. Design Summary for the Adiabatic Air Jacket

Length, in	48
Outer Jacket Diameter, in	5.9
Inner Jacket Diameter, in	4.72
Thickness of the Air Space, in	0.45
Resistance of Each Heater, ohm	13

2 - Heating Element Assemblies

Since electrical heaters are easy to control, and electrical energy may be measured accurately, it was decided to move the fluids electrically. Specially designed immersion heating elements which operate smoothly and without excessive surface temperatures are necessary to accomplish the operation. The heater designs finally used were developed through testing a variety of designs during the course of this work. Suitable commercial heaters meeting the requirements of this project were not found.

When a hot metal surface is submerged in a pool of liquid, various regimes of boiling are observed as it is shown in Figure 8. In range AB, heat is transferred to the liquid by natural convection. Range BD defines the region of nucleate pool boiling. In this range, at first the bubbles are small (BC), and as ΔT increases the bubbles become larger and more numerous (CD). Range DE defines the region of transition boiling. In this region, an unstable vapor film forms around the surface of the heater, collapses and re-forms rapidly

under the action of circulating currents. Around E this vapor film is stable and for values of ΔT beyond 1,000°F, nucleation occurs only at the interface between this film and the liquid.

The boiling curve of Figure 8 is easily obtained if the inside surface of the heater is heated by condensing vapor. But, when electrical heating is used, regimes DEF are not obtained. When heat flux is raised above the peak of the boiling curve, D, conditions change immediately to those of point F, causing a sudden increase in temperature. Very often, point F is well above the melting point of the heater. Since it takes only a second or so for the temperature of the heater to increase from the temperature at point D to the temperature at point F, D is taken as the criterion to define the "burn out point" or the critical heat load of the heater.

Equation 1[28] correlates most of the data available on burn out heat fluxes (9, 19).

$$(q/A)_{crit} = 143\ \rho_v\ h_{fg}\ \left(\frac{\rho_L - \rho_v}{\rho_v}\right)^{0.6} \qquad (1)$$

Except for very small wires (<0.005 in) and very thin strips (<0.006 in), the magnitude of $(q/A)_{crit}$ for submerged wires, vertical and horizontal plates, cylinders, and so on is independent of the geometrical shape or position of the heater.[29]

For obvious reasons, when boiling liquids by electrical means, it is desirable to remain in the region of nucleate boiling. Rohsenow[29] suggests the following equation to correlate heat flux for nucleate boiling.

$$\Delta T = \frac{C_{sf}}{c} \left[\frac{q/A\ h_{fg}^2}{\mu_l}\sqrt{\frac{g_c\ \sigma}{g(\rho_L-\rho_v)}}\right]^{1/3} (Pr)^{1.7} \qquad (2)$$

In Equation 2, C_{sf} accounts for the geometry of the heater and liquid combination. Data for C_{sf} in terms of fluid and heating surface combination are available.[29] For instance, C_{sf} is equal to 0.013 for the boiling of benzene and water with clean and smooth Nichrome surfaces. Calculated data for water and benzene were compared with the same data calculated by Insinger and Bliss[14] correlation. The results were in very good agreement.

Based upon the information discussed so far, a set of compact immersion heating elements were developed and tested during the design work of this research project. It was a problem to find a material for the core of the heaters that can be machined, has good electrical properties (electrical insulation) and stands high working temperatures. After some investigation, it was found that silicates of aluminum or magnesium, known respectively as Lava grade A and Lava 1136 (American Lava Corporation, Chatanooga, Tennessee) could solve the problem. These materials, in the hydrous state, are easily machined into any desirable form or model. Then, by firing the model, chemically bound water is eliminated modifying the crystalline structure and converting the material into a hard product. The electrical properties of the fired Lavas resemble those of porcelain. These properties are described in Table III.

TABLE III. Physical Properties of the Lavas After the Firing Operation

	Lava Grade A	Lava 1136
Safe Operating Temperature, °F	2,012	2,282
Hardness, Mohs	6	6
Coefficient of Thermal Expansion	3.6×10^{-6}	12×10^{-6}
Thermal Conductivity, Btu/hr ft°F	0.725	1.21
Dielectric Strength	80	100
Dielectric Constant	5.3	5.8
Resistivity, ohm cm	0.6×10^{12}	9×10^{12}

The preparation of the heater cores was performed as follows: Cores of Lava 1136, machined as shown in Figure 4, were put in a cold electrical furnace and heated at the rate of 600°F per hour to avoid cracking. The temperature was held at 1,900°F for half an hour to complete the curing process. After curing, the heat was turned off and the cores allowed to cool in the furnace.

Before coiling, the Nichrome V wire was stretched in order to straighten it out. The wire was coiled under tension in order to avoid excessive expansion during the glass sealing operation. Both ends of the Nichrome V wire were tied to the core of the heater by means of finer Nichrome wire and a pair of small holes were drilled through the core on each end of the helical threads. Tungsten wires, approximately two inches long, were connected on both ends of the Nichrome V wire by means of stainless steel sleeves compressed on the connections. The

tungsten wires are required in order to make a vacuum sealed junction through the glass wall. Glass matching alloys, such as Therlo (Driver-Harris Company, New Jersey) could also have been used. Silver brazed connections of tungsten to the Nichrome were tried first. But, during the sealing operation of the tungsten wire through the glass, the silver would melt, oxidize, and make the connection loose. Spot welding of the tungsten and Nichrome wire is difficult due to the high melting point of the tungsten.

TABLE IV. Design Summary for the Heating Elements

Heater	Reboiler	Overhead Vaporizer	Bottom Vaporizer
Length of Wire, ft.	9.35	10.9	10.3
Impedance, ohms	16.4	18.4	18.2
Resistance, ohms	14.8	17.1	16.4
Number of Wire Turns	21	23	28
Height of the Coil, In.	1.31	1.44	1.75
Power Density, Btu/hr ft^2	58,000	51,000	58,500
Outside Diameter, In.	1.835	1.75	1.5
Inside Diameter, In.	1.585	1.5	1.25

Power densities were calculated at 130 volts. This voltage is the maximum output given by the Variacs used in this work.

3 - Distillation Unit Assembly

The necessity for incorporating a continuous distillation unit in a small cylindrical jacket gave rise to several problems during the

design and construction of the apparatus. Several design trials were made in order to compact the distillation unit as much as possible. Figure 5 presents a sketch of the entire unit which is self-explanatory. The upper end of the column is detachable by means of a 55/50 taper joint. Different plate designs may be removed and placed inside the column through this end with the help of a three foot long wire hook.

The plates are sealed against the glass wall of the precision glass tube by means of teflon gaskets. It was observed that teflon shrinks when submitted to the action of heat and stresses, as in the case of the gaskets in between the plate assemblies. A reduction of about 0.020 inches was observed in machined two inch diameter gaskets of extruded teflon. Reinforced molded teflon (15 per cent glass) shrinks less than the extruded form, but after two hours under the action of common solvents it became harder and did not conform well to the surface of the plates. Better results were achieved with machined gaskets of molded teflon tubes which were preheated 48 hours in boiling mineral oil. In this case, the shrinkage was only about 0.005 inches per two inches of teflon.

Electrical energy is supplied to the heaters by means of wire leads directly connected to the exposed tungsten wires on the outside of the column wall. The true power supplied to every heater is measured by wattmeters and controlled by Variacs. With alternating current, voltmeters read effective voltage drop and ammeters read effective current. The power factor of the heater must be known in order to compute the averaged power input from voltmeter and ammeter readings.

The reboiler is charged through the overhead condenser and drained through an opening at its bottom.

Vapor generated in the reboiler moves up through the column into an overhead condenser outside the adiabatic jacket. The liquid from the condenser is received in the overhead product container. Liquid accumulates in this container until it overflows the internal tube and returns to the column as reflux. The amount of reflux is controlled by the heat input to the overhead vaporizer.

Column pressure drop forces an equivalent amount of liquid from the reboiler into the bottom product vaporizer. The vapor generated by both the bottom product and overhead vaporizers combines into another overhead condenser to form a gravity-flow stream which returns into the column as feed. Total column pressure drop is measured by the difference of liquid height in the bottom product container and the liquid height in the reboiler. The readings are taken directly from scales placed alongside the external tubes of the reboiler and bottom product vaporizer.

TABLE V. Design Summary for the Distillation Unit Assembly

Column Internal Diameter, In.	2.00
Height of the Column, In.	29.7
Reboiler Capacity, $In.^3$	38
Capacity of the Overhead Vaporizer, $In.^3$	10.5
Capacity of Bottom Vaporizer, $In.^3$	13.5
Total Height of the Distillation Unit, In.	47
Height of Jackets, In.	48

Outer Jacket Diameter, In.	5.9
Inner Jacket Diameter, In.	4.72
Air Space between Jackets, In.	0.45
Resistance of each Heater of the Jacket, ohm	13
Impedance of the Overhead Vaporizer Heater, ohm	18.4
Impedance of the Heater of the Reboiler, ohm	16.4
Impedance of the Bottom Product Vaporizer, ohm	18.2

4 - Plate Assembly

The behavior of the apparatus was determined with a set of nine sieve plates with 0.125 inch diameter holes.

These plates were designed according to the best information available on the dynamics of sieve trays. The reasons for selecting sieve trays instead of bubble caps may be justified by the fact that the use of sieve plates as contactors is becoming more and more popular.[8,20,27,35] Another reason is the fact that perforated trays are geometrically less complicated than bubble cap trays.

Laboratory sieve plate columns generally have smaller holes than their industrial counterparts. However, the hole size chosen above is in the usual range of industrial designs (0.125 to 0.375 inches).

In operation, a sieve tray differs somewhat from a bubble cap tray. It requires a minimum hole vapor velocity to maintain the liquid depth on the plate. The hydraulic relationships on a sieve tray are shown in Figure 2.

One of the most important characteristics of a contactor is its pressure drop. Total plate pressure drop is determined by the combined effects of:

1) A dry plate pressure drop resulting from the flow of vapor through the holes without liquid on it.

2) An effective liquid head resulting from the depth of turbulent liquid above the holes.

3) A surface tension effect resulting from the resistance to bubble formation in the liquid above the holes.

Dry plate pressure drop has been the subject of several investigations.[1,15,20] Equation 3 was taken from Hunt et al.[13] in which the factor k was taken equal to 1.14. McAllister et al.[21] expresses k as a function of the ratio of plate thickness to hole diameter. Figure 9 is a plot of this function.

$$h_{DP} = k \frac{U_o^2}{2g_c} [0.4 (1.25 - \beta) + (1 - \beta^2)] \qquad (3)$$

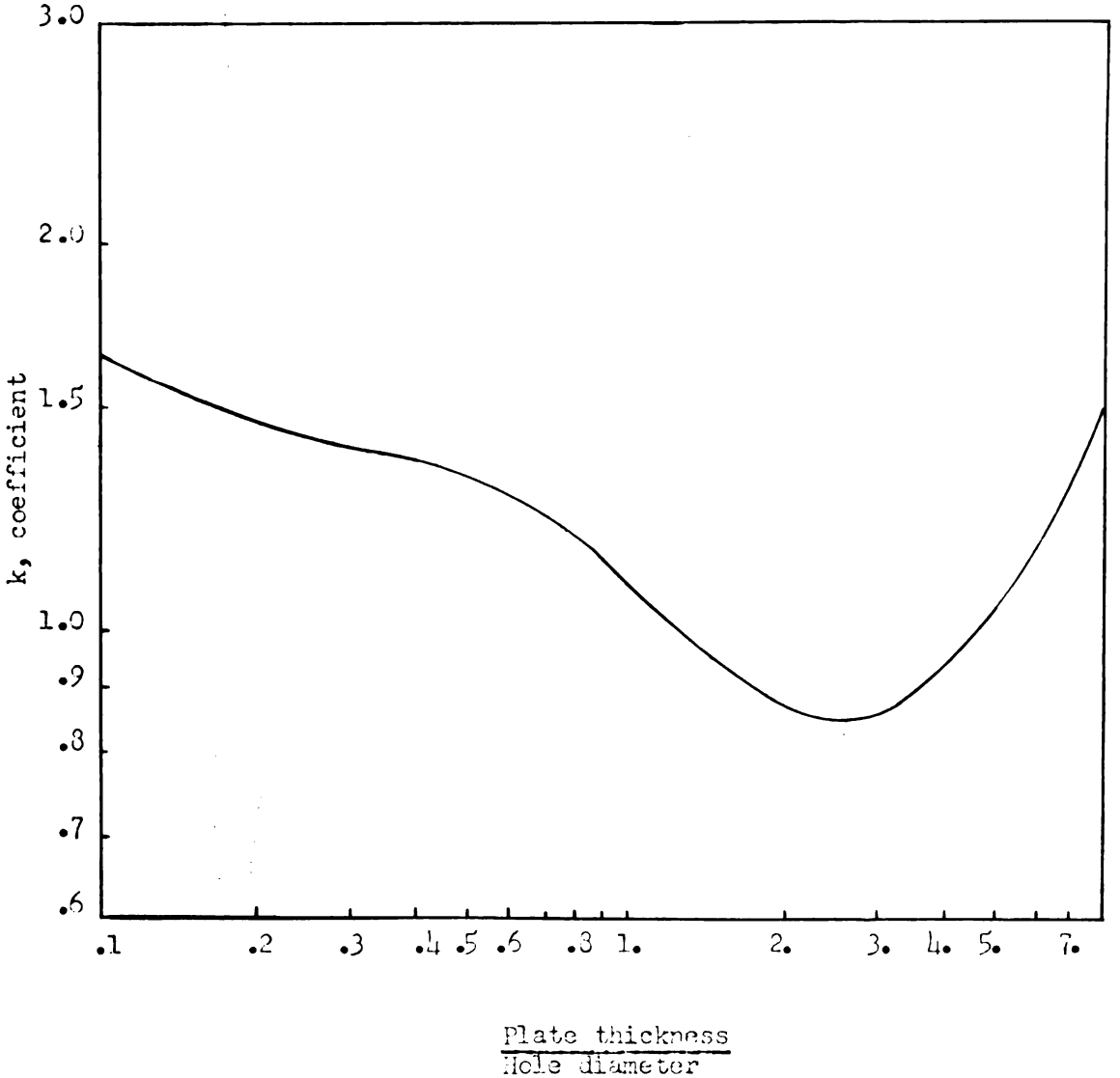
Hughmark and O'Connell[12] correlated dry plate pressure drop directly as a function of the orifice coefficient as follows:

$$h_{DP} = \frac{(1 - \beta^2) U_o^2}{C^2 \quad 2g_c} \qquad (4)$$

Values for the orifice coefficient, C, are given by Figure 10. Either Equation 3 or 4 represent with good accuracy dry plate pressure drop for any gas flowing through plates with sharp edged holes on a triangular distribution.

The effective liquid head is calculated by the height of the weir

Figure 9. Dry plate coefficient k, as a function of plate thickness to hole diameter ratio. (From McAllister et al.[21])



5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
K, coefficient

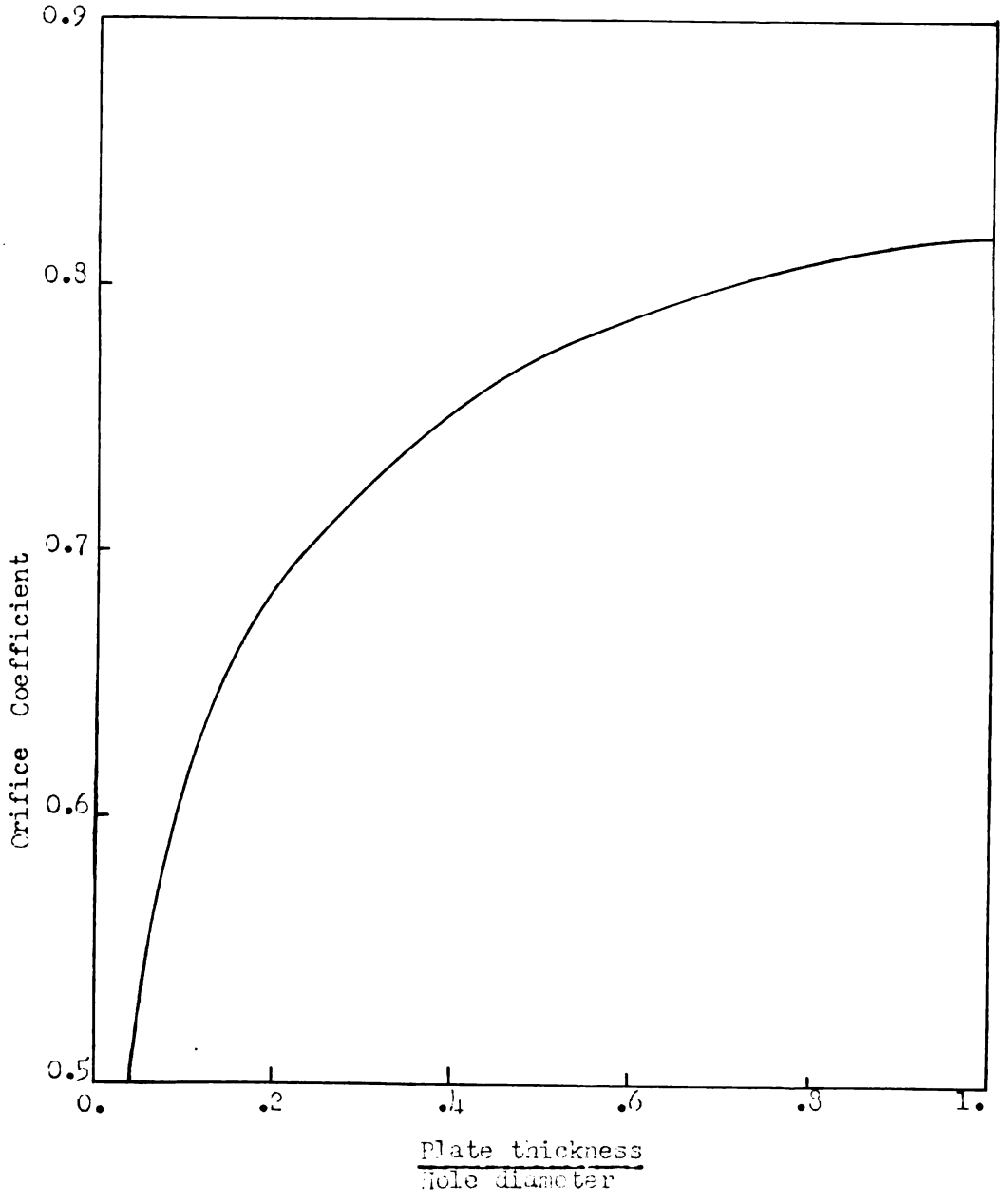


Figure 10. Orifice coefficient versus plate thickness to hole diameter ratio. (After Hughmark et al.[12])

Figure 1 - [illegible]

plus the effects of the weir crest and hydraulic gradient.

$$h_L = (h_w + h_l + 1/2 \ \Delta H)a \tag{5}$$

Here, h_w is the height of the weir, h_l is the weir crest, ΔH is the hydraulic gradient, and a is an aeration factor which takes into account the fact that the indicated height is made up of both liquid and vapor.

For circular downcomers and small flow rates, the weir crest is calculated by the Francis weir formula.

$$h_l = 3.9 \left(\frac{Q}{L}\right)^{2/3} \tag{6}$$

However, in columns such as the one of this project, the weir crest effect is very small and may be neglected. Also an aeration factor, $a = 0.8$ is recommended.[6] Also, because the column diameter is so small, the hydraulic gradient may be neglected.[20] Thus, Equation 5 which gives the effective liquid head may be simplified to:

$$h_L = 0.8 \ h_w \tag{7}$$

The surface tension effect is determined by the following relationship.[32]

$$h_s = \frac{48\sigma}{D_h \rho_L} \tag{8}$$

Winkle et al.[32,33] performed an extensive study of the effects of physical factors and operating conditions on the design and efficiency of sieve trays.

A set of sieve trays has been designed using his data in addition

to the equations given above. The plates were assembled as a unit for the purpose of easy removal and placement inside the column.

TABLE VI. Design Summary for the Plate Assembly

Number of Plates	9
Feed Plate Number	5
Plate Spacing, In.	3.25
Plate Thickness, In.	0.036
Hole Diameter, In.	0.125
Number of Holes	12
Per Cent Free Area	4.7
Pitch-Diameter Ratio	3.04
Weir Height, In.	0.355
Downcomer Internal Diameter, In.	0.32
Per Cent Downcomer Area	2.54

5 - Performance of the Unit

a) Visual Observations. The performance of the unit was observed for long periods of time under conditions of total reflux, zero reflux, and a variety of intermediate reflux and boilup ratios.

After equilibrium was reached, the operation of the column was smooth, completely stable, and would respond quickly to any external variation of heat input. No signs of internal condensation in either column or accessories were observed.

Some leakage of liquid through the teflon gaskets existed particularly at low vapor velocities. However, it did not seem to be

enough to disturb the operation, and as the vapor velocity was increased, the leakage disappeared almost completely.

The immersion heaters have good performance in regard to boiling. Under normal operating conditions, the heat required by the reboiler to operate the column varies between 1,000 and 3,000 Btu/hr. The heaters of the overhead and bottom product vaporizers have to generate smaller amounts of heat in order to accomplish their functions.

About 3,000 Btu/hr were generated by the heater of the reboiler without excessive turbulence or foaming. This corresponds to the maximum output of the Variacs used in this experimental work.

Under normal operating conditions, the heaters of the overhead and bottom product vaporizers gave satisfactory operation.

On the basis of visual observations, the unit seems to be operating satisfactorily in every way.

b) Pressure Drop. Two scales located along the external tubes of the reboiler and bottom product vaporizer permit making readings of the height of liquid in each container. The total pressure drop of the column is given by the difference of two readings. A maximum error of about five per cent is possible in taking the reading, due to the oscillation of the liquid level in the external tube of the reboiler caused by liquid overflowing from the column.

Total plate pressure drop data for benzene, carbon tetrachloride, ethanol, and methanol, at total reflux, were taken as a function of hole vapor velocity. See Appendix C. These data are plotted in Figure 11 and Figure 12. Data for benzene, carbon tetrachloride, and ethanol were correlated with the following equation.

$$h_t = 0.385\ (\rho_v/\rho_L)\ U_o^2 + 0.8\ h_w + \frac{48\sigma}{D_h \rho_L} \qquad (9)$$

Data for methanol can be plotted in a line with the same slope as the one of Equation 9, but with an intercept 0.1 inch lower.

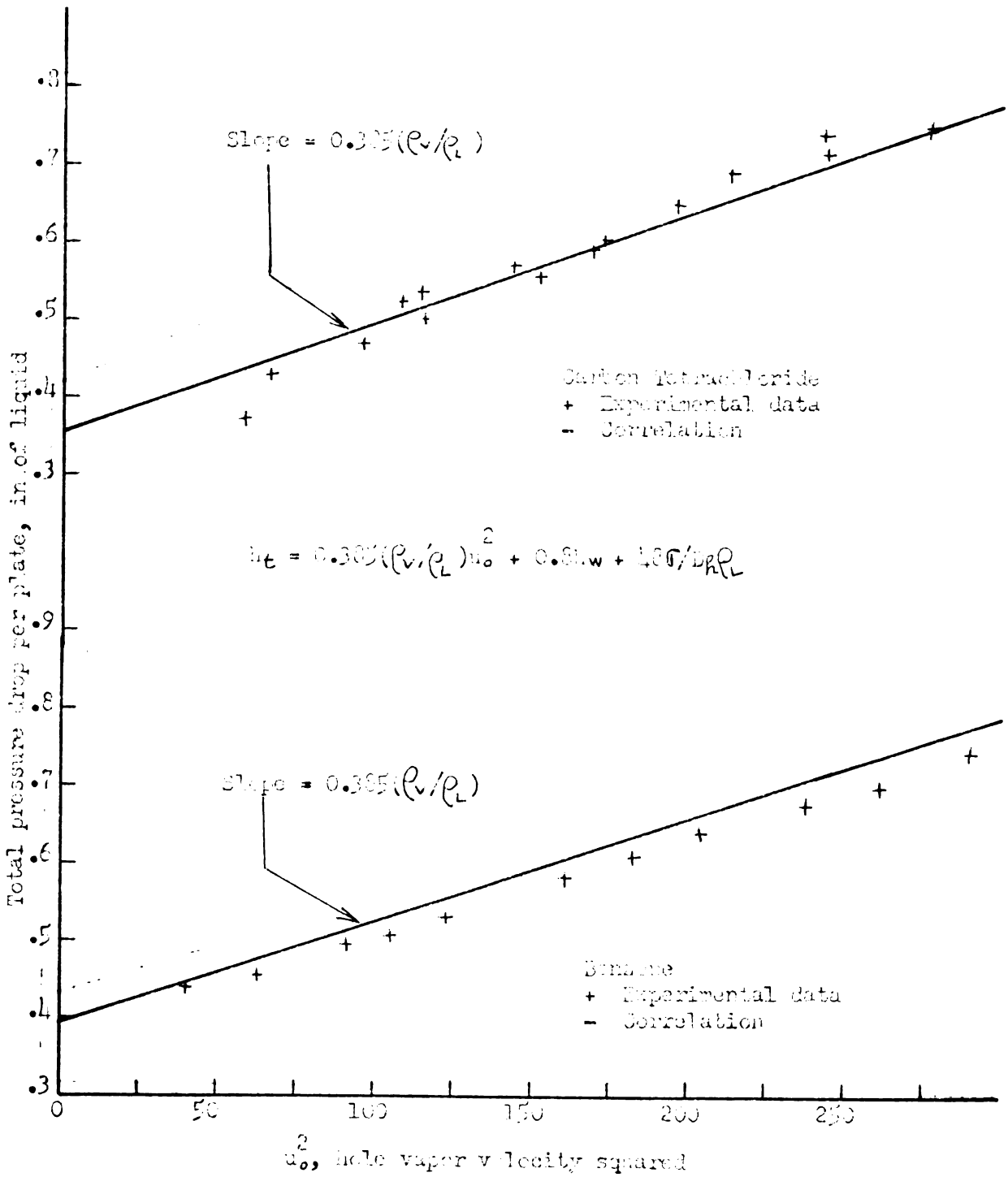
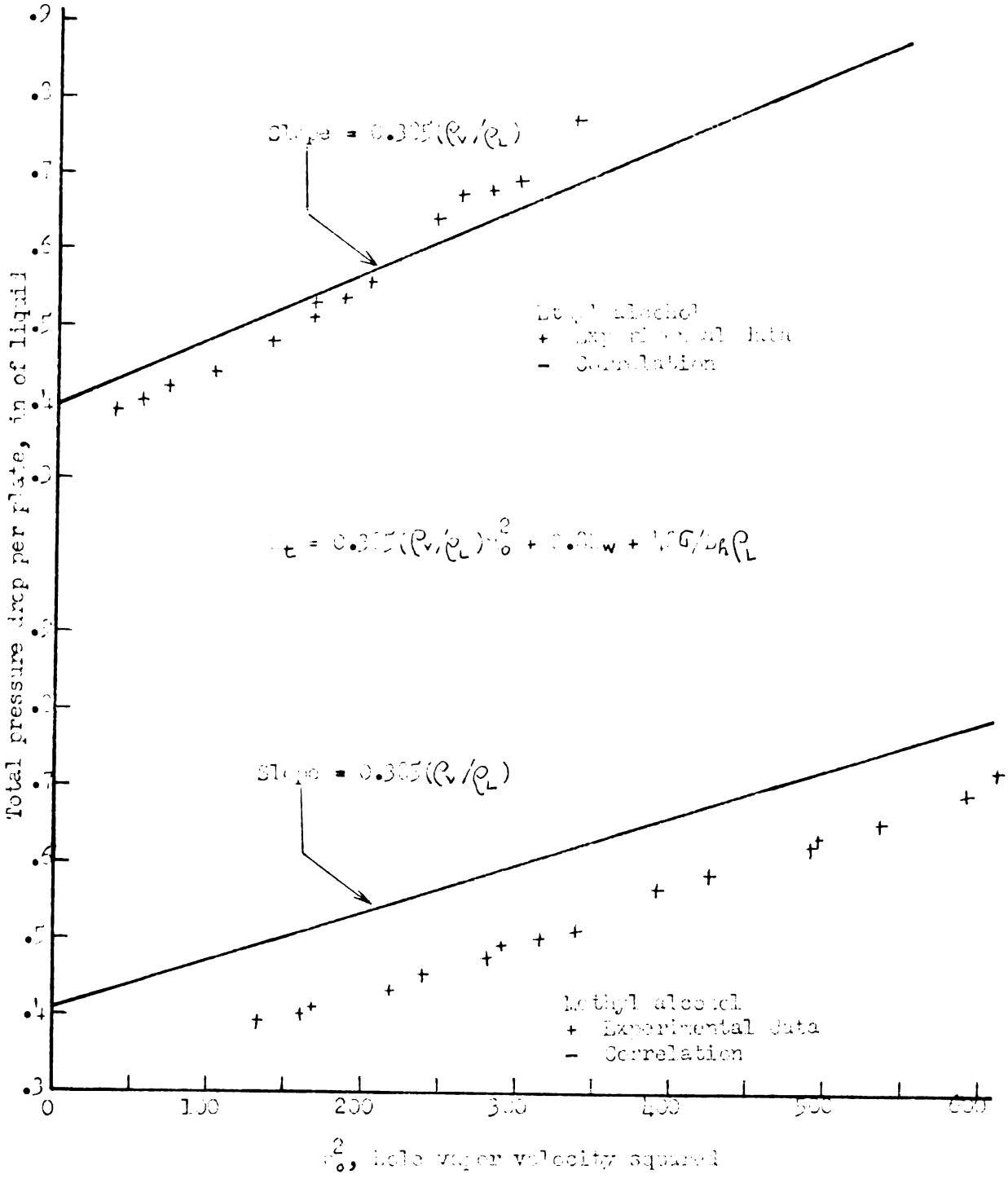
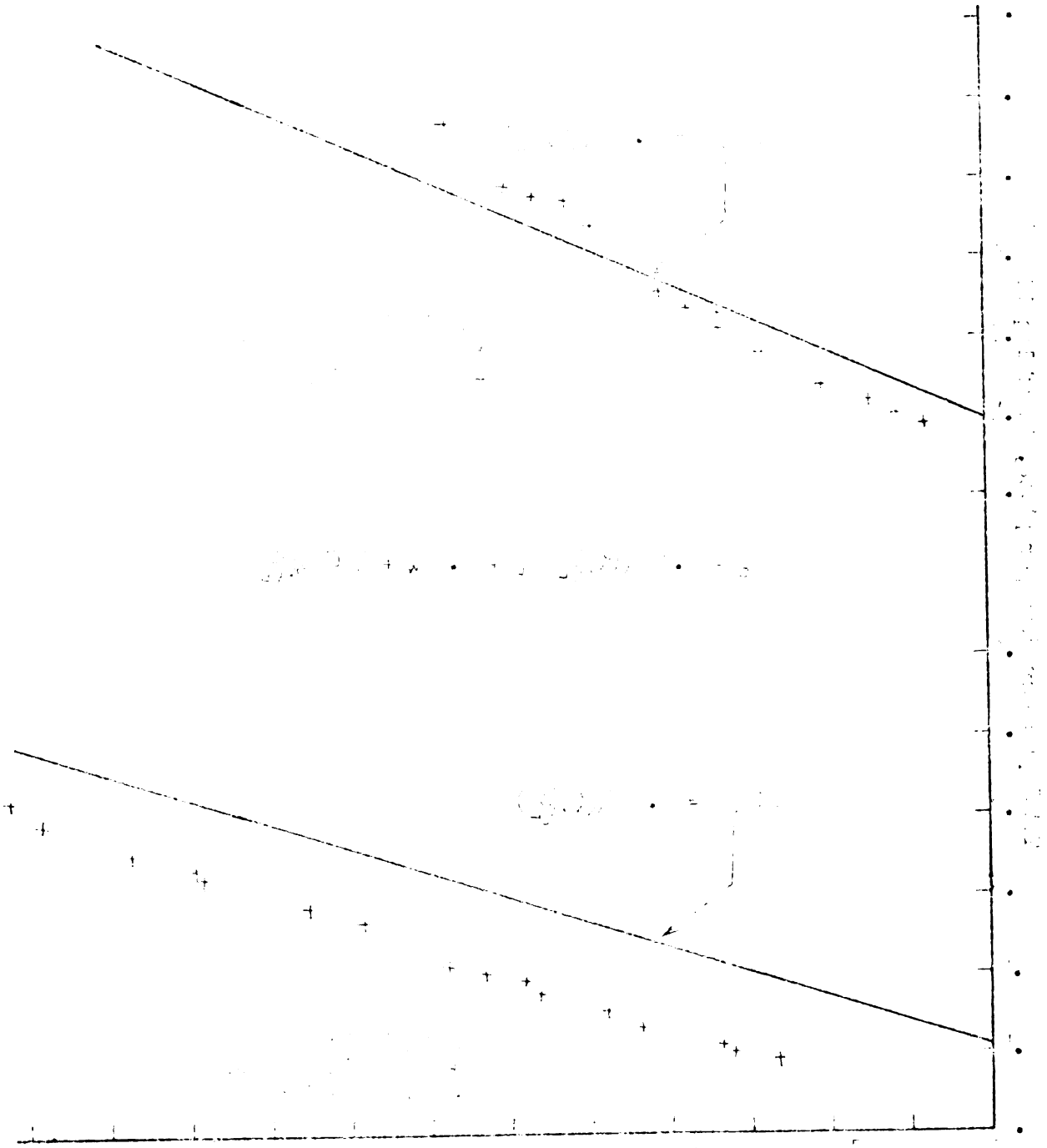


Fig. 11. Total plate pressure drop for benzene and carbon tetrachloride versus hole vapor velocity squared.



V_o^2, hole vapor velocity squared

Fig. 12. Total plate pressure drop for ethanol and methanol versus hole vapor velocity squared.



PART V

DISCUSSION

DISCUSSION

A - Attainment of Objectives

1) Steady State. The unit as designed operates with continuous feed addition and continuous top and bottom product removal. Operation of the column with pure liquids gave no evidence of varying liquid hold-up in any point of the system. Therefore, it may be concluded that true steady-state performance will be attained for mixtures when composition equilibrium is reached. It remains for a future investigator to show how long this will take.

2) Pumping mechanism. The unit presents all the characteristics of a continuous fractionating column without using any pump or mechanical device. Fluids are moved electrically by specially designed immersion heating elements which operate smoothly and without heat losses. These heaters vaporize liquid from the bottom and overhead product containers. The two vapor streams generated in those containers are combined in an overhead condenser to give a gravity-flow liquid feed that returns to the column onto the feed plate. The heaters are effective and extremely easy to operate and control. The peculiar problems of small pumping equipment such as clumsiness, air-trapping, control, and so on have been eliminated.

3) Metering Devices. Since the wattage input to the heaters can be measured accurately, feed flow, reflux ratios, and heat input are controlled and metered by Variacs and wattmeters respectively. Since the

heaters respond quickly, we can correct quickly and easily any small change or variation in the system. This is much more satisfactory than trying to control flows with rotameters and valves. At these small flow rates such mechanical devices are awkward and give poor operation.

4) Heat Losses. By having the entire unit, with the exception of the condensers, enclosed in an electrically heated air jacket, heat losses have been substantially eliminated.

The experiments with methanol show that with this jacket the column can be operated adiabatically. Due to the fact that the wires of the resistances of the air jacket are at a higher temperature than the column, some heat may be gained by the column by radiation from the wires. This heat tends to compensate for the small amount of heat lost from the column by radiation.

5) Feedstock Supply. The unit requires only about 30 cubic inches of feedstock to operate continuously for an indefinite period of time. This is attained by causing the unit to combine its own products and circulate them back into the column as feed at the boiling point. The amount of material needed is simply the quantity required to cover the heaters and provide for column holdup.

6) Visibility. All parts of the distillation unit, except heaters and tray assemblies, are fabricated with pyrex glass.

7) Easy Modification. A great deal of versatility is provided by having the upper end of the column connected to the accessories at the top by means of a taper joint. This connecting joint is big enough to permit the removal or introduction of the plate assemblies. Thus,

column internals can be easily removed, replaced, or substituted by other types of plates or packing. In the case of packing, supports and possibly distributors are needed to transform the column into an efficient continuous packed column.

8) Composition Analysis. In such a small column, it is not practical to take out samples for typical gravimetric and volumetric analysis. The small quantities required for analysis by refractive index or by vapor-phase chromatography would create less disturbance of the column operation. A method for analyzing plate composition without any disturbance at all has been suggested and could be developed in the future. This method consists of measuring the temperature of the mixture on the plate at its boiling point. An accuracy of at least 0.1°C in these measurements is required.[36] Thermistors give very accurate temperature readings. A correction for pressure can easily be made based on the levels in the bottom product vaporizer and reboiler.

9) Stage Efficiency. In small columns, flow patterns are somewhat different from the ones in large diameter columns, and stage efficiencies may also be different. The tested set of sieve plates was designed following the best recommendations for high efficiency plate designs.[10,11,23,33] Due to their size, it is expected that the Murphree plate efficiency of this unit would be approximately equal to the local Murphree efficiency. The plate efficiency for a large column might then be calculated using a correlation such as Figure 1. This correlated efficiency would thus be related to the efficiency of the miniature column, but it would not be necessarily equal. It remains for a future investigator to determine how effective such a procedure would be.

B - Sieve Plate Performance

Experimental data for the runs with benzene, carbon tetrachloride, ethanol, and methanol are plotted in Figure 11 and Figure 12. Data for methanol had an intercept 0.1 inches of liquid lower than the expected values from Equation 9. This lower intercept might have been caused by surface active impurities in the methanol. However, no surface tension measurements were made with the feedstocks. In any case, a difference of 0.1 inch is not significant when these data are extended to large industrial columns.

Equation 9 was proposed on the basis of an average experimental slope from Figure 11 and Figure 12 of 0.385 (ρ_V/ρ_L). The corresponding value calculated from Equation 3 is equal to 0.368 (ρ_V/ρ_L). However, Equation 3 with values of k from Figure 9, was developed for plates with a large number of holes.[21] For plates with one hole, in the Reynolds number range of the experiments, the slope of the correlating equation should be equal to 0.48 (ρ_V/ρ_L). It seems reasonable, therefore, that the slope of the proposed correlation should lie somewhere between these two values.

Under conditions of minimum hole vapor velocity, all the plates started dripping at the same time. This indicated uniformity in the operation and good bubbling action on the plates. Hole vapor velocities were calculated from the power supplied to the reboiler.

Conditions of maximum hole vapor velocity were not achieved, because the power supplied by the Variacs was not enough to generate a sufficient amount of vapor to reach the flooding point.

PART VI

CONCLUSIONS

CONCLUSIONS

Simulation of industrial fractionation units on a miniaturized scale is often more difficult than designing the commercial scale units themselves.

In scaling down a commerical unit into a laboratory or small pilot plant scale, all major operational characteristics of the former must be incorporated in the latter. However, considerations such as economic conditions, predictability of performance, and mechanical strength are peculiar problems of large scale equipment and have no significance in laboratory size units. On the other hand, considerations that have no importance in large columns are important in small columns. In this category should be mentioned heat losses, easy modification of the unit, easy assembly and disassembly, and so on.

Heat losses in small columns tend to be excessive due to their comparatively large ratio of superficial area to throughput capacity. Heat losses upset the operation of the column and provisions must be made to reduce them to a minimum amount or eliminate them completely.

Industrial columns are designed for specific purposes. They are designed to produce products and not for the purpose of experimentation. In miniaturized columns, due to their small size and research nature, considerations such as easy modification, assembly, and disassembly are important. These considerations must be incorporated in the design of the unit as much as possible in order to get the best possible performance of the apparatus as a research unit.

The amounts of feedstock available for laboratory or pilot plant studies are usually very small. As a consequence, flow rates are also small and continuous columns face the problem of moving these small streams. The normally available pumping mechanisms and metering devices are not able to handle such streams satisfactorily. At these rates, they give clumsy operation and unreliable results.

In this research project, heat losses were essentially eliminated by enclosing the fractionating column and accessories in a jacket. The design is compact enough to guarantee the use of a relatively small diameter glass tube as a jacket. Fluids are caused to flow by means of specially designed immersion heating elements. These heaters operate smoothly without excessive surface temperatures, and are easily controlled by Variacs. The all-glass construction provides complete visibility. Additional strength and protection against breakage is also provided by the jacket.

The accessories at the top are detachable from the rest of the column. Plates are removed through the open end for modification, substitution, or cleaning. Any type of trays can be used including packing.

Overhead product rate, boilup rate, and bottom product rate are controlled and metered by the wattage input to these heaters. From these, reflux ratio and boilup ratio may be calculated.

The set of stainless steel plates, designed to test the performance of the unit, gave experimental data consistent with some of the design equations used for larger diameter columns. It appears that good

correlation between the hydrodynamics of this column and larger scale columns may be obtained. Plate efficiencies were not determined, because such studies were not within the scope of this particular work.

As with most research involving the building of new apparatus, much of the time spent was devoted to problems of little scientific or technical interest. Typical of such problems were procurement of the tungsten wire and the precision bore tube, difficulties with the teflon gaskets, and development of a satisfactory heater core. Additional reference to these problems appears in the main body of this thesis.

In conclusion, the author believes that the original purpose of this work has been accomplished satisfactorily. However, with some additional work the value and applicability of the column can be enhanced in a number of ways as indicated in the following pages.

PART VII

RECOMMENDATIONS FOR FUTURE WORK

RECOMMENDATIONS FOR FUTURE WORK

1) Analysis by Temperature

The composition of the liquid or vapor can be determined by reading the temperature of the mixutre at its boiling point. The overhead condenser has an enlarged opening to permit the introduction of thermocouples or thermistors through it and through the plates to take temperature readings at any desirable point. This method, if carried out carefully, will give a measure of the composition on each plate and permit estimation of plate efficiencies.

2) Feed and Product Withdrawal

Several ways may be suggested in order to provide the unit with product withdrawal and external feed. However, due to reliability and accuracy of control, electrically controlled methods would be the best ones. By these means, overhead and bottom products could be taken out through the bottom of the column, separately as vapor streams. This way, the rate of product withdrawal can be metered and controlled accurately.

For feed admission, a surge tank could be placed inside the electrically heated air jacket. Liquid from this tank can be vaporized electrically and sent to an overhead condenser to produce a gravity-flow liquid stream that can be fed into the column in the same way as in the present unit.

3) Azeotropic Distillation

The top of the column can be modified so as to incorporate a decanter for azeotropic fractionations. Only a minor modification of the top head design is needed to provide for withdrawal and separation of azeotropic layers.

4) Plate and Packing Studies

The unit was primarily designed for the evaluation of design factors and tray dynamics of plate-type vapor-liquid contactors. However, it can be easily modified to operate with packing materials. Vapor and liquid distributors should be used to guarantee good distribution over the packing.

5) Systems Studies

The column is well suited to systems studies for a number of reasons. Flow rates and heat inputs are measured and controlled electrically. The electrically heated air jacket creates conditions of adiabatic operation which are essential in operating small columns. Lag times and disturbances caused by the presence of mechanical devices are absent. For these reasons, disturbances can be applied and the effects readily measured and recorded to give data which can be analyzed by systems techniques to suggest mathematical models for packing or trays. If the column is adapted to operate with external feed and product withdrawal, and with temperature analysis; techniques such as frequency response of feed and product disturbances can be valuable tools in analyzing the dynamic behavior of large industrial columns.

6) Process Studies

Actual processes generally operate under conditions close to minimum reflux ratios and with a large number of theoretical plates. The unit developed in this work can be adapted to simulate cases like this. The use of low H.E.T.P. packing such as wire or glass helices would provide a large number of plates, and approach the conditions of a large unit for studying continuous separations of mixtures.

7) Distillation Demonstrating Unit

The advantage of using a versatile and flexible unit to simulate industrial operations for teaching purposes were emphasized in Part I, Section 3. Also, this apparatus permits the determination of accurate heat balances and gives accurate measurements of feed rates. These data would be well suited for constructing Ponchon-Savarit or McCabe-Thiele diagrams. A student could easily operate the unit, collect the data, and construct his own diagrams.

8) Vacuum Operation

Only three taper joints were used in the construction of the apparatus. The entire unit is tight enough to undergo vacuum operation. Vacuum fractionations are particularly helpful when processing heat sensitive compounds. Vacuum could be applied to either one of the overhead condensers.

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APPENDIX A

TABLE VII. Physical Properties of Liquids at the Atmospheric Boiling Point

Liquid	T, °F	ρ_L, lb/ft^3	ρ_v, lb/ft^3	k', $\frac{Btu}{hr ft °F}$	μ_l, lb/fthr	μ_v, lb/fthr	σ, lb/ft	h_{fg}, Btu/lb	c, Btu/lb°F
Benzene	177	51.0	0.172	0.083	0.764	0.0218	0.00145	170	0.464
CCl_4	170.2	92.5	0.343	0.056	1.180	0.0290	0.00137	83.5	0.211
Ethanol	173	46.0	0.103	0.092	1.065	0.0254	0.00115	368	0.731
Methanol	148.4	46.9	0.0762	---	0.825	0.0290	0.00135	473	0.770
n-Heptane	209	38.5	0.209	0.077	0.496	---	0.00088	137	0.625
n-Pentane	97	37.8	0.180	0.077	0.501	0.0152	0.00098	146	0.557
Water	212	59.8	0.037	0.393	0.687	0.0302	0.00404	970	1.003

APPENDIX B

NOMENCLATURE

C	orifice coefficient
c	specific heat, Btu/lb°F
D	hole diameter, in
D_h	hole diameter, ft
E_L	local Murphree efficiency
E_p	Murphree plate efficiency
G	superficial mass velocity, lb/hr ft^2
g	acceleration of gravity, ft/sec^2
g_c	gravitational constant, ft/sec^2
h_{fg}	enthalphy of vaporization, Btu/lb
ΔH	hydraulic gradient, in of liquid
h_L	effective liquid head on the plate, in of liquid
h_s	pressure drop due to the surface tension effect, in of liquid
h_t	total pressure drop per plate, in of liquid
h_w	weir height, in of liquid
h_{DP}	dry plate pressure drop, ft of gas
h_l	liquid height over the weir, in of liquid
k	factor depending upon plate thickness to hole diameter ratio
k'	thermal conductivity, Btu/hr ft°F
L	length of weir, ft

Pr	= c μ/k', Prandtl number
Q	liquid flow rate, ft^3/sec
Q_v	vapor flow rate, ft^3/sec
q/A	heat flux, Btu/hr ft^2
$(q/A)_{crit}$	critical heat flux, Btu/hr ft^2
ΔT	temperature difference between heating surface and saturated liquid, °F
U	column vapor velocity, ft/sec
U_o	hole vapor velocity, ft/sec

GREEK SYMBOLS

β	ratio of area of perforations to cross-sectional area of the column
μ_l	viscosity of saturated liquid, lb/hr ft
μ_v	viscosity of saturated vapor, lb/hr ft
ρ_L	liquid density, lb/ft^3
ρ_v	vapor density, lb/ft^3
σ	surface tension, lb/ft

APPENDIX C

Experimental Data on Pressure Drop

Conditions: Total Reflux and Atmospheric Pressure

Wattage in Reboiler, Watts	h_t, In. of Liquid	U_o, ft/sec	Wattage in Reboiler, Watts	h_t, In. of Liquid	U_o, ft/sec
	Benzene			Carbon Tetrachloride	
200	0.44	6.36	250	0.428	8.1
250	0.47	7.94	300	0.470	9.75
300	0.495	9.55	400	0.59	13
350	0.53	11.1	450	0.69	14.6
400	0.58	12.7	480	0.735	15.6
450	0.64	14.3	380	0.577	12.3
500	0.70	15.85	510	0.75	16.6
550	0.775	17.42	430	0.65	14
	Ethanol			Methanol	
250	0.39	6.1	450	0.39	11.5
300	0.4	7.05	490	0.40	12.65
330	0.42	7.98	500	0.41	12.9
400	0.43	9.80	570	0.427	14.7
475	0.48	11.6	600	0.45	15.40
520	0.5	12.7	650	0.47	16.75
520	0.52	12.7	660	0.49	17.0
550	0.55	13.48	690	0.50	17.8
640	0.64	15.55	710	0.51	18.3
650	0.67	15.90	790	0.565	19.80
675	0.672	16.50	800	0.58	20.6
700	0.685	17.05	860	0.494	22.2
750	0.77	18.05	900	0.65	23.6