

A DISTILLATION COLUMN FOR  
TRAY EFFICIENCY STUDIES

By

MICHAEL DEAN BURNS

Bachelor of Science

University of Arkansas

Fayetteville, Arkansas

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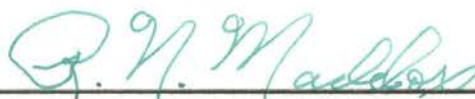
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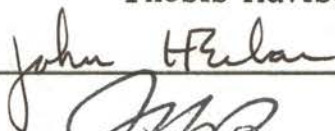
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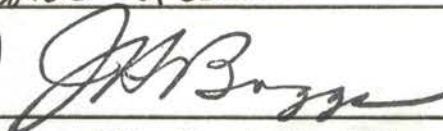
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Thesis Approved:



\_\_\_\_\_  
Thesis Adviser





\_\_\_\_\_  
Dean of the Graduate College

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## CHAPTER I

### INTRODUCTION

Methods are readily available for calculating the behavior of a distillation column on the basis of ideal or equilibrium trays. However, to determine the performance of an operating column, the ideal or equilibrium composition change across a tray must be related to the actual change. The concept of tray efficiencies was introduced to relate the ideal and actual tray.

Determining a tray efficiency is one of the least certain steps in the design of a distillation column. With a growing application of vacuum distillation and the use of more expensive materials of construction, the need for a better understanding of the factors affecting the efficiency is obvious. And, expansion of technology into new and unusual areas has led to demands for an accurate method of predicting efficiencies. For these reasons, tray efficiency research has increased rapidly in recent years.

The object of this work is the design and construction of a distillation column and associated equipment to be used for an experimental investigation of tray efficiencies of both binary and multicomponent mixtures. The column is a 12-inch diameter, ten tray column

with Nutter float valve trays on a 12-inch spacing. It is designed to be run either as a total refluxed column or a continuous feed, non-refluxed stripper. Two systems will be used initially and were the basis for the design. These are the binary system benzene-toluene and the ternary system benzene-toluene-para-xylene. Tray efficiency data will be obtained by sampling the vapor and liquid streams around specific trays.

## CHAPTER II

### TRAY EFFICIENCIES

Prior to the design and construction of the distillation system, the author undertook a complete literature investigation of tray efficiencies. The following is a review of efficiencies as applicable to this thesis. We will be concerned with defining the tray efficiency, areas of research for this type of apparatus, and a review of prior experimental investigations with similar equipment.

#### Definitions

The tray efficiency is a measure of the approach to an ideal tray--an ideal or equilibrium tray being defined as one on which both the vapor and liquid phases leaving the tray are in mutual thermodynamic equilibrium. This concept was first expressed by Murphree (14) in terms of several definitions.

The Murphree vapor efficiency is defined as

$$E_{MV} = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}}$$

Here  $y_n$  is the mole fraction of a particular component in the vapor leaving tray  $n$ ,  $y_{n-1}$  is the mole fraction of the same component in the



vapor entering tray  $n$ , and  $y_n^*$  is the composition of vapor that would exist if the vapor leaving the tray were in equilibrium with the actual liquid leaving the tray,  $x_n$ . In a similar manner the Murphree liquid efficiency is defined as

$$E_{ML} = \frac{x_n - x_{n+1}}{x_n^* - x_{n+1}}$$

The terms  $x_{n+1}$  and  $x_n$  are the compositions of the liquid entering and leaving tray  $n$  respectively, and  $x_n^*$  is the composition of liquid in equilibrium with the vapor leaving the tray,  $y_n$ . Murphree, in this manner, has defined tray efficiency as the ratio of the actual change to that which would have occurred had the tray reached an equilibrium state.

These definitions of tray efficiency will represent a physical situation only in certain cases. Vapor channeling and incomplete liquid mixing are two common conditions which will result in deviations from the Murphree model. On a large diameter tray, the liquid composition can vary across the tray and result in an efficiency of greater than 100 per cent. The point Murphree efficiency, expressed in terms of vapor compositions, describes the degree of approach to equilibrium between the vapor and the liquid at a single point on the tray. It is defined as

$$E_{OG} = \frac{y_n(p) - y_{n-1}(p)}{y_n^*(p) - y_{n-1}(p)}$$

where  $y_{n-1}(p)$  and  $y_n(p)$  are the compositions of the vapor which enter

and leave point  $p$  on the tray and  $y_n^*(p)$  is the composition of the vapor in equilibrium with the liquid on the tray at point  $p$ .

In some cases tray efficiencies are related to the separation efficiency of a whole column (or section of a column). Lewis (12) defined the overall column efficiency,  $E_O$ , as the ratio of actual trays in a column to the number of ideal trays that will yield an equivalent separation. Although this is obviously the simplest approach, this definition suffers from the difficulty that it tries to describe the separation behavior of an entire column with one number even though conditions are changing throughout the column. It also leads to difficulties for multicomponent mixtures, for unless the column efficiencies for all constituents are the same, the ideal and the actual column cannot yield the same product concentrations for non-key components.

Two other definitions of tray efficiency are of interest. No experimental work for these efficiencies has been reported.

Carey, as reported by Nord (15), introduced temperature or thermal efficiencies for the case of heat transfer on a distillation tray. Here, efficiency represents the degree of approach to thermal equilibrium. The vapor temperature efficiency is defined by

$$E_{TV} = \frac{T_n - T_{n-1}}{T_n^* - T_{n-1}}$$

$T_n$  and  $T_{n-1}$  are the temperatures of the vapor entering and leaving the tray respectively, and  $T_n^*$  is the temperature of the vapor in equilibrium with the liquid leaving the tray.

Standart (18) has defined a generalized tray efficiency which is concerned with the change of the extensive properties of a phase across the tray. The states of the entering streams on the tray are kept constant in comparing the actual with the ideal equilibrium tray. They are given by the flow rate, temperature, and composition of the streams. The following constitute Standart's generalized efficiency. The overall material efficiency is given by

$$E = \frac{V_n - V_{n-1}}{V_n^* - V_{n-1}} = \frac{L_n - L_{n+1}}{L_n^* - L_{n+1}}$$

The efficiency for the  $i$ th component of the system is

$$E_i = \frac{V_n y_{n,i} - V_{n-1} y_{n-1,i}}{V_n^* y_{n,i}^* - V_{n-1} y_{n-1,i}} = \frac{L_n x_{n,i} - L_{n+1} x_{n+1,i}}{L_n^* x_{n,i}^* - L_{n+1} x_{n+1,i}}$$

And the enthalpy efficiency is given by

$$E_H = \frac{V_n H_n - V_{n-1} H_{n-1} + r_n Q_n}{V_n^* H_n^* - V_{n-1} H_{n-1} + r_n Q_n} = \frac{L_n h_n - L_{n+1} h_{n+1} + (1-r_n) Q_n}{L_n^* h_n^* - L_{n+1} h_{n+1} + (1-r_n) Q_n}$$

Here,  $V$  and  $L$  are the molal flow rates of the streams around a plate.  $V_{n-1}$  is the rate of the vapor entering tray  $n$  from the tray below and  $L_{n+1}$  is the liquid entering tray  $n$  from the tray above.  $V_n^*$  and  $L_n^*$  are the vapor rate and liquid rate respectively of the equilibrium streams leaving the ideal tray, and  $V_n$  and  $L_n$  are the rates of the vapor and liquid streams which actually leave the tray. The vapor mole fraction and liquid mole fraction for the  $i$ th component are given by  $y_i$  and  $x_i$ .  $H$  is the molal vapor enthalpy and  $h$  is the molal liquid enthalpy. These terms are subscripted and superscripted as the  $V$

and  $L$  terms, where  $n$ ,  $n+1$ , and  $n-1$  indicate the  $n$ th tray, the tray above, and the tray below respectively, and  $*$  denotes equilibrium.  $Q_n$  represents the rate of heat loss from the  $n$ th tray to the surroundings and  $r_n$  is the fraction of that heat lost by the vapor.

### Research Considerations

Although tray efficiency research has been extensive in recent years, many factors remain unsolved. Several areas of interest are currently being pursued. Many factors that affect the distillation process on the tray are being studied including foaming, surface tension, entrainment, liquid mixing effects, froth height, and thermal effects. Two areas of particular interest in this work are prediction-correlation studies of tray efficiency and investigations of efficiency of multicomponent systems.

The correlation and prediction of tray efficiency is being investigated from two approaches--empirical and fundamental. The empirical approach offers many possibilities but requires large quantities of accurate data. The fundamental approach involves correlations based on theoretical mass transfer models.

### Empirical Approach

The empirical approach to tray efficiency research involves investigating and correlating any or all of the design, operating, and system property variables encountered on the distillation tray with tray efficiency.



Drickamer and Bradford (5) utilized plant test data from refinery fractionating columns to correlate overall column efficiency with molal average liquid viscosity of the feed. The tests were on bubble-tray towers of over 4-feet diameter and apply only for this type of tray and for hydrocarbon systems. An extension of the Drickamer-Bradford correlation was developed by O'Connell (16). This correlation relates the overall efficiency for fractionating columns to the product of the relative volatility of the key components and the molal average liquid viscosity of the feed. By inclusion of the relative volatility, O'Connell was able to extend the Drickamer-Bradford correlation somewhat. Although these correlations are adequate for the systems on which they were based, they utilize only one or two of the many variables involved.

To more accurately account for the complexity of the problem, more variables were considered. Charyavech and Van Winkle (2) attempted to evaluate the effect of several system property variables on perforated plate efficiencies in small diameter columns. The variables considered were surface tension, relative volatility, viscosity, density, and diffusivity of both vapor and liquid phases. Data obtained from a 1-inch diameter perforated plate column were correlated to give an equation for the Murphree plate efficiency in terms of these variables. English and Van Winkle (8) improved on this by developing a correlation of tray efficiency as a function of the design and operating variables as well as the system property variables that predom-

inately affect the efficiency. These were fractional free area, weir height, reflux ratio, the liquid Schmidt number, and surface tension. Both of these correlations are relatively untested.

### Fundamental Approach

The fundamental approach to tray efficiency research involves the use of modern mass transfer theory to characterize the transfer processes occurring on the tray. The mass transfer relations which have been developed for tray efficiency will not be reviewed in this thesis. These are adequately covered in Smith (17).

The first major effort in this area was a project of the AIChE Research Committee--a five year study of bubble tray efficiencies. The aim of this program was to develop a method of predicting efficiencies for bubble trays used in commercial distillation columns. The result was the publication of a bubble tray design manual (1) incorporating correlations based on fundamental models of the transfer processes occurring on the tray.

The AIChE method (20) follows a development based on the two-resistance concept of mass transfer. This theory, a modification of the two-film theory of Lewis and Whitman, postulates two additive resistances in series. One is the resistance to mass transfer in the vapor phase and the other is the resistance to mass transfer in the liquid phase. Addition of the two resistances gives a total resistance to mass transfer from one bulk phase to the other. Correlations were obtained for each of the two resistances. The variables affecting the

resistances were the physical characteristics of the tray, the vapor and liquid flow rates, and mass transfer characteristics of the fluid phases. The Murphree point efficiency is then calculated from the overall mass transfer resistance.

To relate the point efficiency to the Murphree tray efficiency, a model estimating the degree of liquid mixing on the tray was developed. Here, the variables considered were the distance of liquid travel across the tray, the eddy diffusion coefficient, and the residence time of the liquid on the tray.

The final step in predicting tray efficiency by this method is accounting for the effects of entrainment. The degree of entrainment is estimated and the Murphree tray efficiency is adjusted. Variables involved were surface tension, vapor velocity, and tray spacing.

Two authors have offered modifications for the AIChE method. Strand (19) introduced a model which accounts for the effects of liquid and vapor bypassing on the tray. Eduljee (6, 7) suggested different correlations for the hydraulic behavior and the tray efficiency which give improved results. These correlations were based on additional data than that used for the AIChE correlations.

Application of the AIChE procedure is limited to bubble tray towers and, except for very specific cases, to binary systems. The major usefulness of the method is for systems where no previous experience exists. Yet this work showed what had been felt by many authors for some time--that tray efficiencies can be correlated and

predicted from the more fundamental theories of mass transfer.

### Multicomponent Efficiencies

Of the many studies of tray efficiency, the work has been almost exclusively involved with binary distillation. Although the efficiency picture for binary systems is far from fully defined, one should recognize that most industrial distillations involve multicomponent mixtures. Both theoretical and experimental studies have demonstrated that additional complex problems are involved with more than two components present.

Toor and Burchard (21) have applied the theory of multicomponent diffusion and a gas phase film theory model to describe the mass transfer process on a tray. Through computer calculations they have shown that efficiencies in three component systems can be markedly different from binary efficiencies under the same flow conditions.

Free and Hutchison (10) and later Diener (4) made experimental studies of three component systems in small distillation columns. Free and Hutchison showed that differing diffusivities of components in the system will cause Murphree efficiencies for the components not to be identical. Diener developed and experimentally verified mass transfer relationships for a ternary system that are analogous to those used in correlating binary efficiency data in the AIChE research program.



### Prior Experimental Investigations

There have been numerous investigations of tray efficiency. Many studies have been made on small laboratory equipment built to simulate distillation trays. However, efficiency investigations on distillation test columns and pilot plant equipment are pertinent to this study. The following is a review of some of the literature reporting such studies.

A tray efficiency study by Grohse, et al.(11), was made on an extractive distillation system separating C<sub>4</sub> hydrocarbons with furfural. A 13-inch diameter, ten tray, bubble-cap column was used. The equipment was designed so that varied conditions of flow rate, composition, temperature, pressure, and tray design could be studied. Combination sample taps and thermocouple wells were provided on most of the trays. Liquid samples were removed from under the center of the downcomer and vapor samples were removed from under the center of the tray above. The vapor samples were consistently reliable but the liquid sample compositions were well scattered -- presumably a result of concentration gradients. Here, the liquid compositions were calculated from vapor compositions and heat balance relationships. Average values of tray efficiency were evaluated over a number of trays from a McCabe-Thiele diagram by a trial and error procedure.

The AIChE tray efficiency study (20) contains two experimental

investigations of interest. The test column employed was 2-feet in diameter with five trays on a 24-inch spacing. The column shell was made of flanged pieces to facilitate tray design changes. A variety of bubble tray designs was used in several different studies. The column was controlled at constant pressure by varying condensate level within the vertical condenser. This reportedly gave close pressure control over a wide range of operating conditions. Numerous sample taps and thermowells were employed to allow measurement of vapor and liquid temperatures on each tray. The liquid samples were removed from the downcomer and the vapor samples from under the center of the tray. Liquid samples were also removed from five points on one of the trays to study liquid mixing effects. Both the acetone-benzene system and the pentane-xylene system were used. In both cases analyses of samples were by refractive index. Tray efficiency calculations were made graphically by a McCabe-Thiele diagram. Several operating variables for the two systems were studied. These include operating pressure, vapor rate, and liquid rate through the column.

Of interest in the AIChE report (20) is a section containing data from a tray efficiency study by Fractionation Research, Inc. This data is for the cyclohexane-n-heptane system in a 4-foot diameter test column with ten bubble-cap trays on a 24-inch spacing. Liquid composition and temperature data for each of the trays were reported for a variety of operating and tray design conditions. No information

was given on obtaining the tray composition data.

Manning, Marple, and Hinds (13) have studied various tray designs in a 5-foot diameter test column. The column is 32-feet high and equipped with sight glasses in the wall for visual and photographic observation of tray action. Thermowells, sample connections, and taps for hydraulic studies were available at each tray location. The system design was such that rapid equilibration and steady operation were realized. The column could be operated at total reflux, as a rectifying section, and as a stripping section. Constant pressure operation was maintained either by controlling the condensate level in the vertical condensers or by controlling the pressure of an inert gas bleed from the overhead accumulator. The system used for most of the studies was iso-octane and toluene. Liquid samples were removed from the downcomer, cooled, and collected. Analysis was by refractive index. Tray efficiency was calculated from the equation as defined by Murphree.

Ellis and Shelton (9) measured efficiencies for the binary system methanol-water in a 4-inch diameter column containing six trays. The trays contained one bubble-cap and a downcomer and were spaced 12-inches apart. Vapor samples were removed from under the bubble-cap and liquid samples from the edge of the downcomer. The samples were analyzed by weight. Tray efficiencies were calculated directly from the equations for Murphree vapor and liquid efficiency.

Dale, Dove, and Huntington (3) report on a study of tray efficiency in a 12-inch diameter valve tray column. The trays have a 12-inch spacing and are part of a package unit. This column is almost identical to that used by the author. Total reflux operation was studied. Vapor samples were taken from alternate trays, condensed, and collected. Analyses of samples were by refractive index. The overall column efficiency was determined and compared at various operating conditions.

## CHAPTER III

### DESCRIPTION OF EQUIPMENT

The distillation system which was designed and constructed for this study consisted of the following: the distillation column; the associated equipment including the reboiler, condenser, pumps, and tanks; the instrumentation and control apparatus; and the vapor and liquid sampling systems. A detailed flow diagram is presented in Figure 1.

The system is designed for operation both at total reflux and as a non-refluxed stripper with continuous feed on the top tray. For total reflux operation there is no feed, no bottoms product, and no distillate product removed from the system. The overhead vapor is totally condensed and the feed-reflux pump is used to pump the condensate from the distillate accumulator back onto the top tray of the column as reflux. For operation as a non-refluxed stripper, feed is pumped from tanks by the feed-reflux pump onto the top tray of the column. Bottoms product is pumped from the reboiler to a product tank and the distillate product is drained from the distillate accumulator to another tank by gravity.

This equipment is located on the concrete pad directly behind

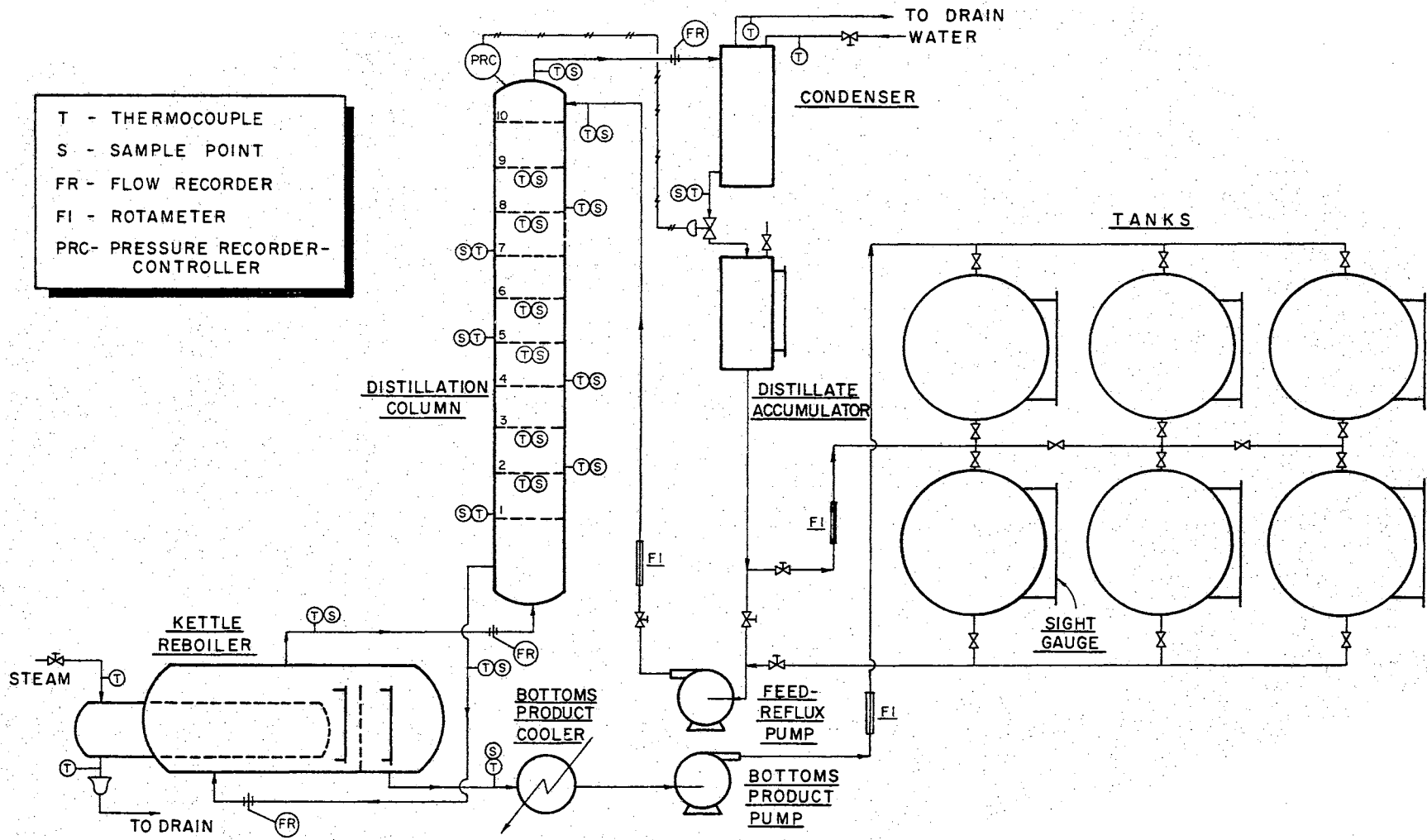


Figure 1  
Schematic Flow Diagram

the Engineering North building on the Oklahoma State University campus. Utilities were provided directly at the installation and include water, electricity, 50 psig steam, and 50 psig air.

### Distillation Column

The distillation column is a 12-inch diameter column equipped with ten Nutter float valve trays on a 12-inch tray spacing. A detailed diagram of the column and the tray assembly is shown in Figure 2. The column is 14-feet in length and constructed from 12-inch Schedule 40 steel pipe. The top of the column is flanged for removal of the tray package. The trays have a 2-inch weir height, a 1 1/2-inch downcomer escape height, and a 0.0702 sq ft downcomer area. There are six Nutter float valves per tray. The top tray is equipped with an entrance baffle for the feed or reflux stream. The downcomer for the bottom tray has a seal pot as shown. The vapor return from the reboiler passes up through the bottom of the column while the liquid to the reboiler is removed from the side. Sample taps are provided for removing both liquid and vapor samples on each tray. Pressure and temperature nozzles are also provided as shown.

The tray package is an independent unit within the column shell and rests on a bottom support in the vessel. It can be removed through the flanged top by means of a crane. Each tray has a floating metal seal ring around the circumference which seals the tray to the tower wall. The trays are assembled as a package unit by means of

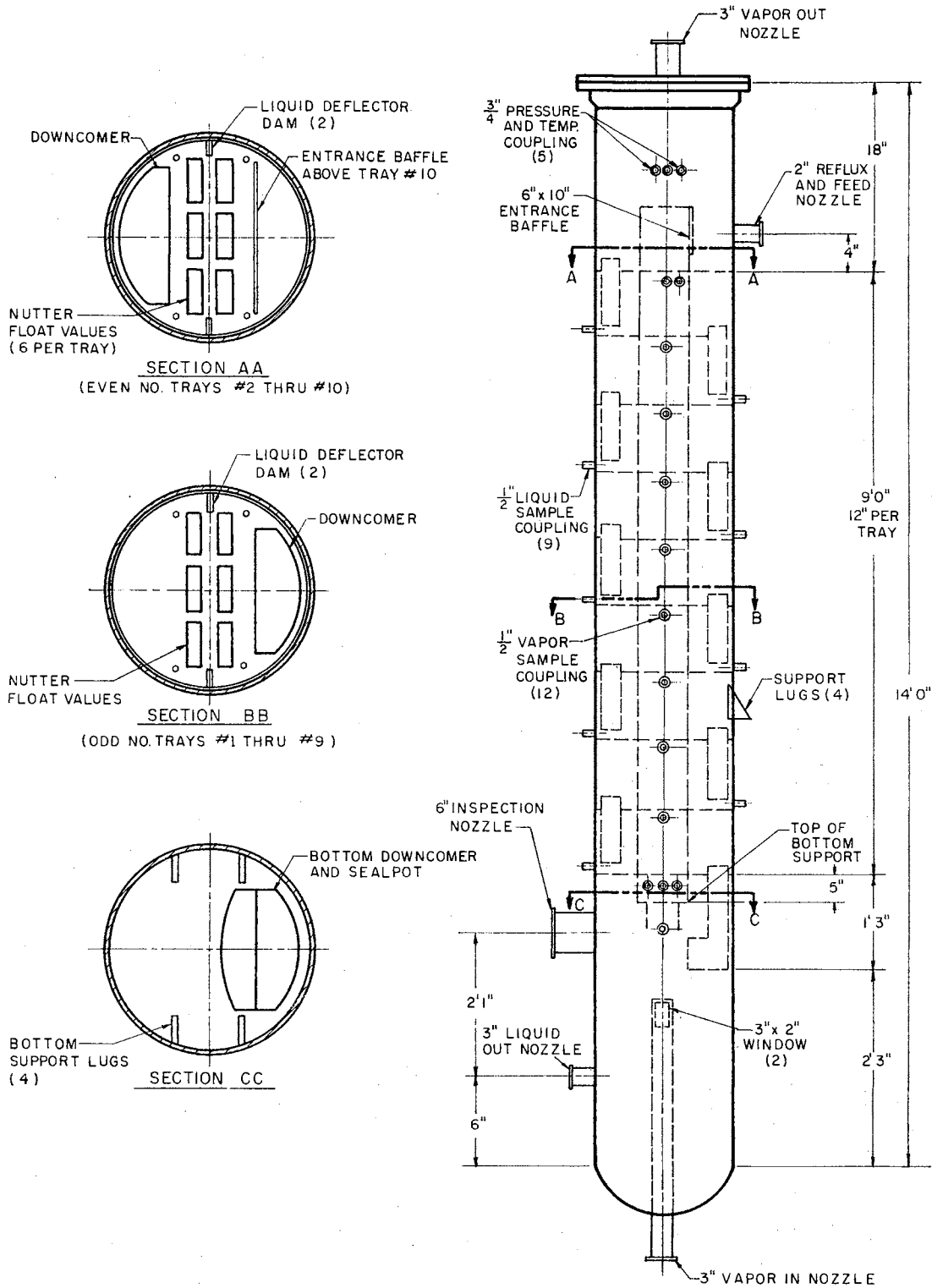


Figure 2  
Detailed Diagram of Column



four verticle support rods.

A photograph of the column mounted in this structure is shown in Figure 3. This structure is built of six 22-foot upright lengths of heavy wall 2 1/2-inch pipe. Three platforms above the ground level were installed on cross bracing. These were spaced at five foot intervals and made of steel grating. A 5-foot high overhead cross piece was welded to the top of the main structure for use in installing the column and for mounting the overhead condenser. The column itself is mounted in the structure by means of three support lugs located midway down the column.

#### Associated Equipment

The process equipment associated with the distillation column consists of: the reboiler, the condenser, the feed-reflux pump, the bottoms product pump, a bottoms product cooler, the distillate accumulator, and six tanks for feed and products. Specifications for the equipment are as follows:

1. Reboiler

U-tube kettle reboiler by Western Supply Company. The tube bundle consists of twelve 3/4-inch steel tubes with a tube surface heat transfer area of 23 sq ft. The kettle is 20-inches in diameter and over 6-feet in length. A weir is provided for bottoms product removal. 50 psig steam provides heat. Glass gauge connections for both

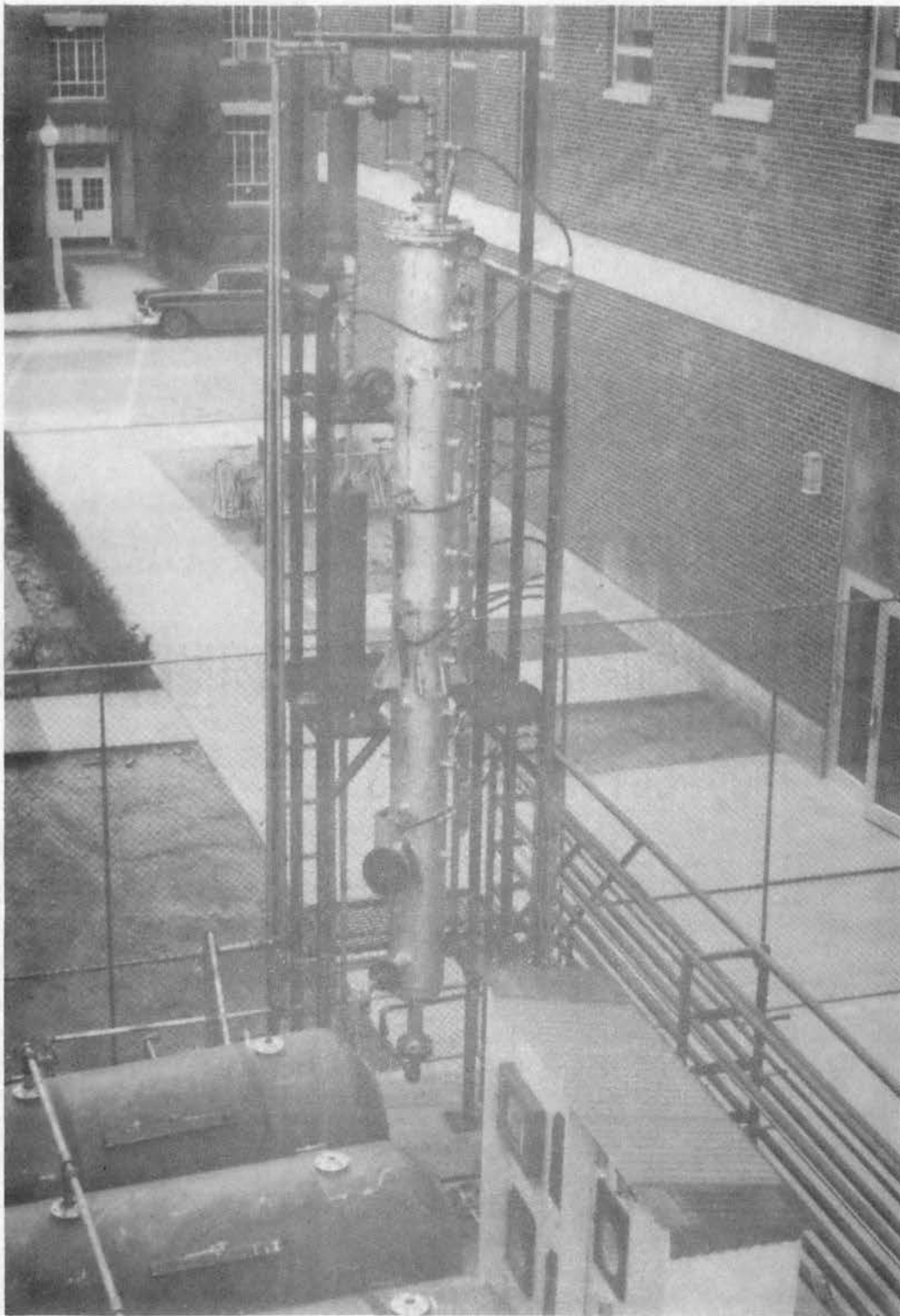


Figure 3  
Photograph of Column and Structure

sides of the weir, pressure gauge connections, and thermowells are provided as well as the inlet and outlet nozzles.

2. Condenser

Ross BCF 603 copper and brass exchanger. It is vertically mounted with condensation on the single pass shell side. The tube side is two pass. Water is the cooling medium. The exchanger contains 116 tubes, 5/8-inch in diameter and 31.5 -inches long. The heat transfer area is 8.6 sq ft.

3. Feed-reflux pump

Two stage Eastern centrifugal pump model 2J-34D of cast iron construction. A mechanical seal is used. A 3/4 HP explosion proof motor gives the pump a capacity of 8 gpm at 60-feet of head.

4. Bottoms product pump

Single stage Eastern centrifugal pump model F-34-B. The pump is constructed of cast iron with a mechanical seal. A 1/3 HP explosion proof motor is used for a 5 gpm capacity at 35-feet of head.

5. Bottoms product cooler

Water cooler of bronze construction. The cooler is horizontally mounted with water on the tube side and product on the shell side. The tube side is two pass.

#### 6. Distillate accumulator

The accumulator was constructed from a 4-foot length of 8-inch steel pipe. Sight glass connections, inlet and outlet nozzles, and a vent were added. The volume is approximately 10 gallons.

#### 7. Tanks

Six horizontal cylindrical tanks provide tankage for the feed and the distillate and bottoms products. The tanks are 250 gallon capacity each and constructed of aluminum. They are mounted in a tank rack as shown in Figure 1. Sight gauge connections are provided. The piping is such that the tanks are integrally connected for ease of material transfer.

### Instrumentation and Controls

The instrumentation and controls for the distillation column are shown schematically in Figure 1. The flow rates and temperatures of the streams around the column are measured and recorded. A temperature profile through the column is also obtained. The column pressure is controlled automatically and the other controls are manually operated. The recording and control equipment is housed in an operator's panel.

The flow rates of several of the streams are determined by measuring the pressure drop across an orifice. Others are measured

by in-line rotameters. The overhead vapor stream between the top of the column and the condenser, the liquid stream from the column to the reboiler, and the vapor stream from the reboiler to the bottom of the column are all measured by orifice plates in the line and recorded on disk chart recorders mounted in the control panel. The feed or reflux stream to the top of the column is measured by a rotameter in the line. During continuous feed operation the flow of the distillate product stream and the bottoms product stream are measured by similar rotameters.

Temperatures are taken at the points indicated on Figure 1. These temperatures are recorded by a Honeywell 24-point temperature recorder. With this instrument, each temperature point is measured and printed on roll chart every minute. The recorder has a temperature range of 0 to 400 degrees F. It is located in the control panel. Copper-constantan thermocouples are used.

The control system for the column includes automatic control of the column pressure and manual operation of the other points by globe valves. A pressure recorder-controller and an air driven diaphragm control valve automatically control the column pressure. Here, for a constant heat load in the reboiler, a constant column pressure will be maintained. The points of manual control are: feed or reflux to the column, bottoms product from the reboiler, distillate product from the accumulator, steam to the reboiler, and water rate to both the overhead condenser and the bottoms product cooler.

An instrument and control panel was built to house the recording and control equipment. A photograph of the panel is shown in Figure 4. The equipment shown includes the multipoint temperature recorder, the flow recorders, the pressure recorder-controller, and the pump switches and solenoid valve switches.

### Sampling Systems

Samples for composition analysis are taken from both the vapor and liquid streams around specific trays. Also, for use in the description of the column operation, samples of the main streams around the column are made. The sample points are indicated in the diagram in Figure 1.

The samples taken of the vapor and liquid streams around a tray will be used for tray efficiency calculations. Figures 5 and 6 show the design of the sample systems and how the samples are removed. The sample of the vapor leaving a tray is taken directly from under the tray above and in the center of the tray. A thermocouple is directly opposite the vapor sample inlet. The liquid sample is removed from directly under the downcomer. As shown, the sample inlet consists of the annular region between the sample tube and the thermocouple. For a complete description of the composition change on a tray, the vapor streams into and leaving the tray are sampled as well as the liquids entering and leaving the tray.

The sample apparatus consists of the sampling tube, a three

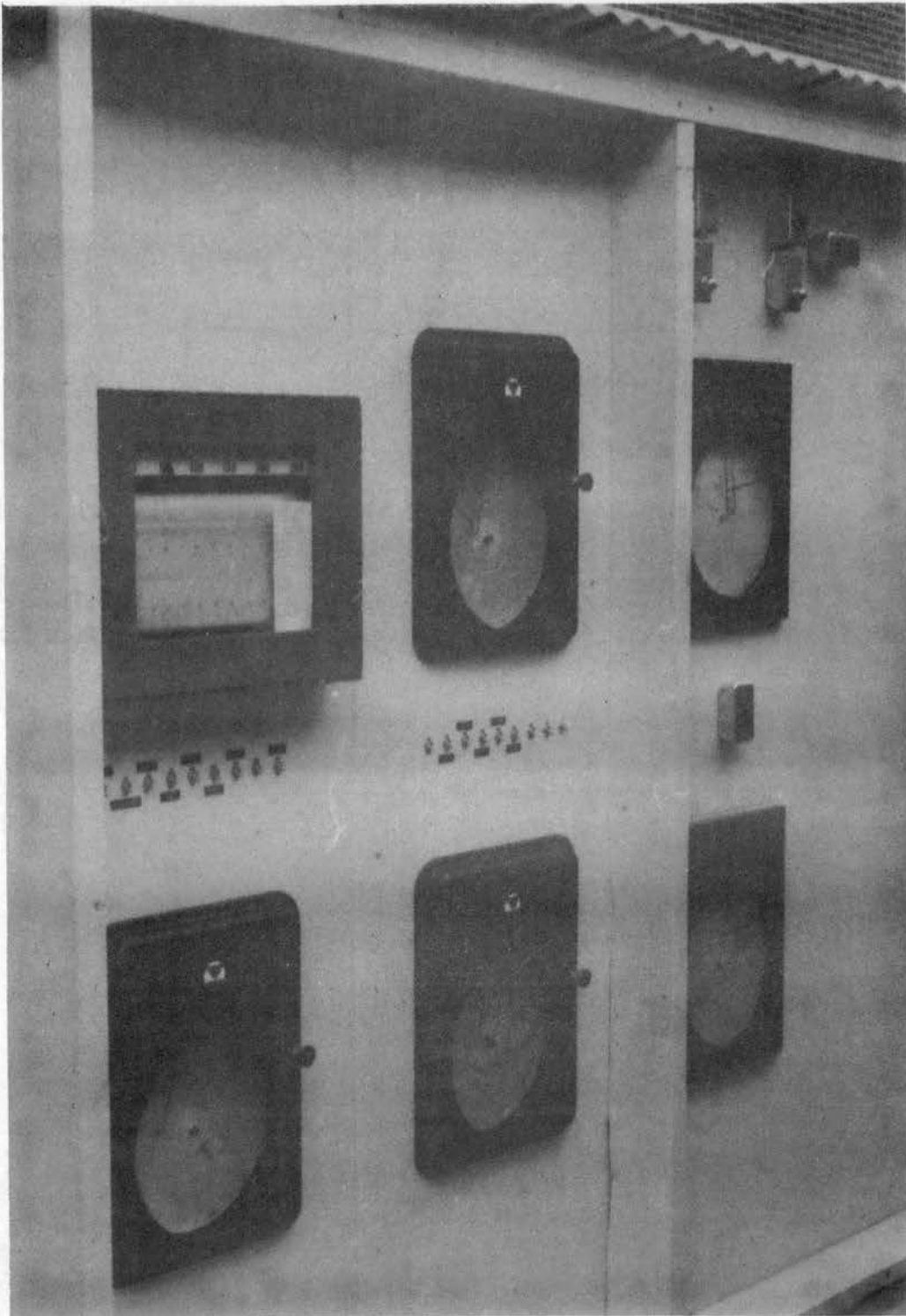


Figure 4  
Photograph of Control Panel

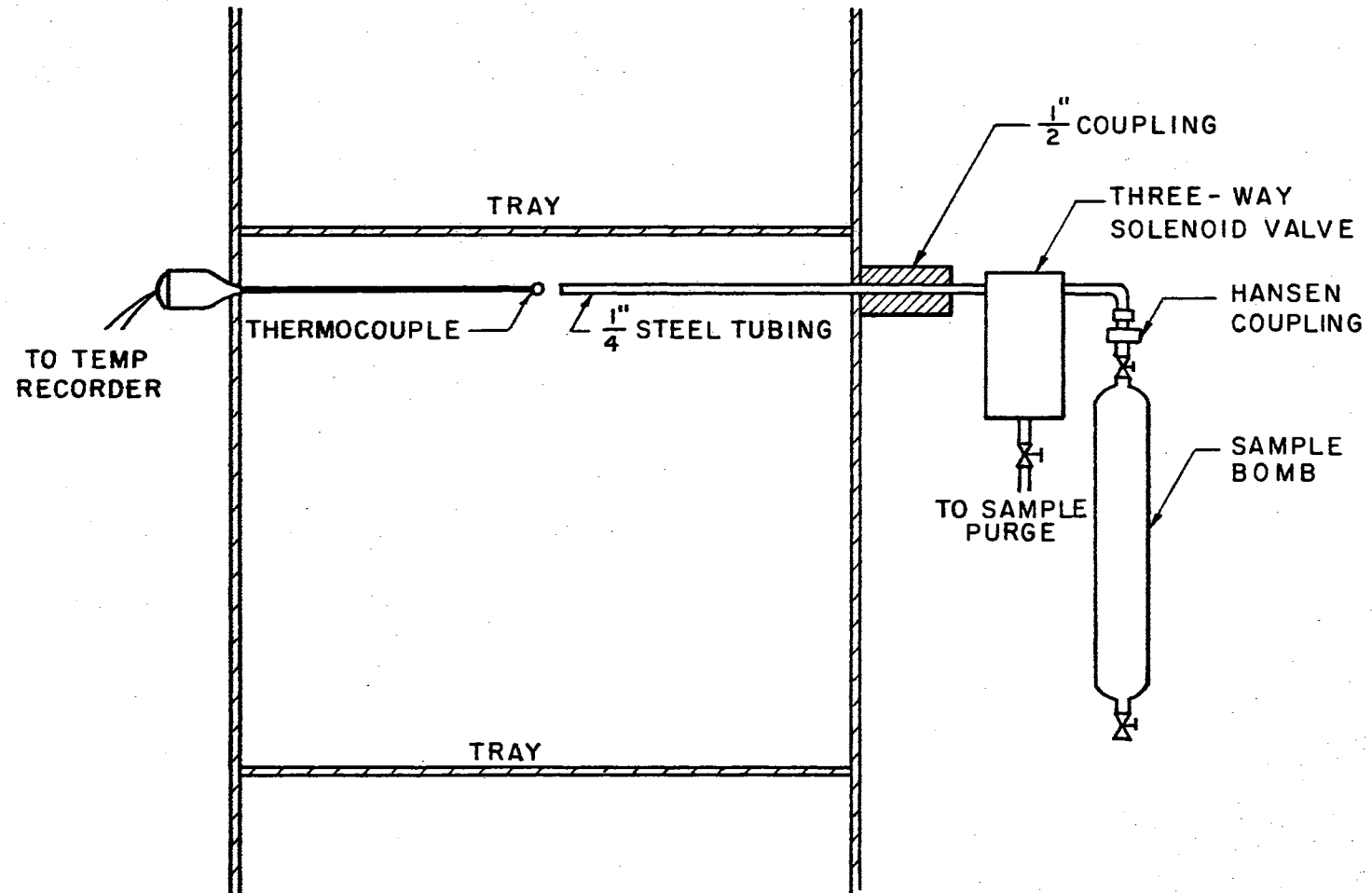


Figure 5  
Detail of Vapor Sample System



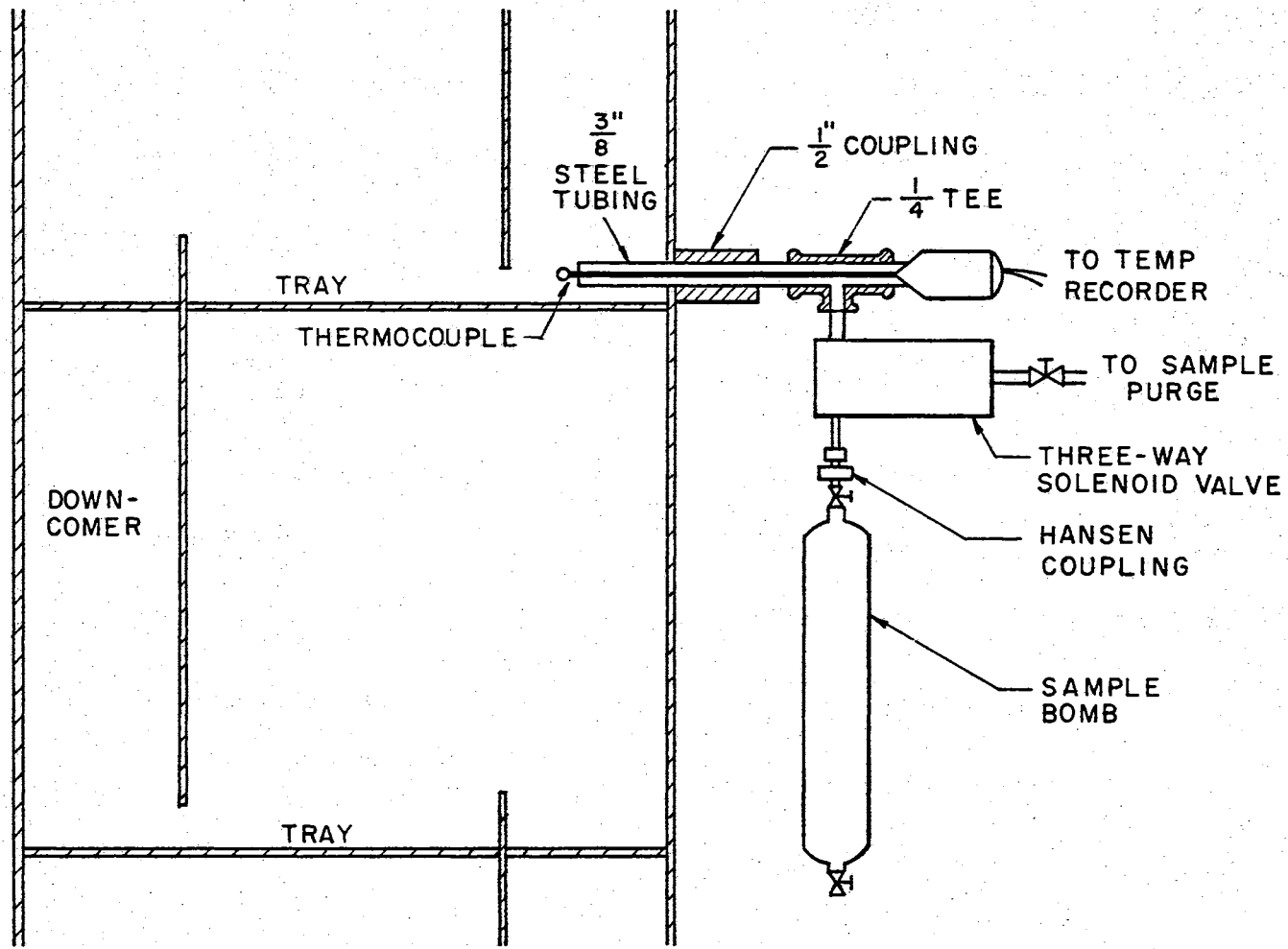
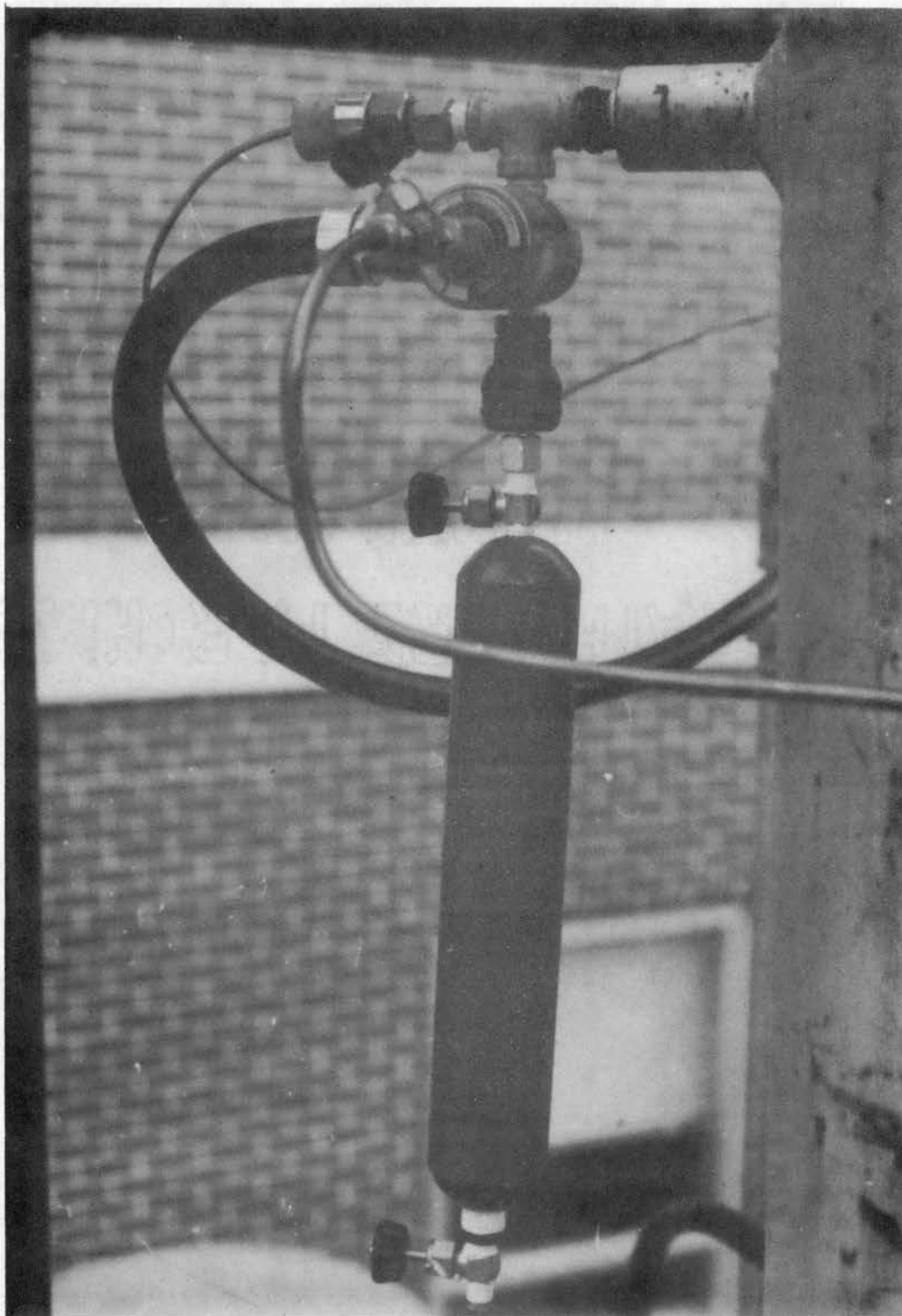


Figure 6  
Detail of Liquid Sample System

way solenoid valve, a bleed line and needle valve for purging the sample line, the sample bomb connection fittings, and the sample bomb. For the vapor systems, the sample tube was constructed from 1/4-inch steel tubing and pipe fittings. The liquid sample tube is made of 3/8-inch steel tubing. The solenoid valves are explosion proof. The controls are located on the control panel described in the previous section. A switch controls the current to the solenoid valves and toggle switches are available to place individual valves on or off the sampling circuit. Hansen push-tight fittings are used to attach the sample bombs to the system. The sample bombs were made from surplus oxygen bottles and will hold approximately 75 milliliters. Figure 7 is a photograph of the installed sampling system for the liquid leaving tray 9. The black hose connected to the solenoid valve is neoprene-jacketed, weatherproof electrical conduit.

The sample composition will be determined by chromatographic analysis. An F and M Model 500 programmed temperature gas chromatograph will be used.



**Figure 7**  
**Photograph of Tray #9 Liquid Sample System**

## CHAPTER IV

### DISCUSSION

#### Design Considerations

The systems to be used initially in the studies with this column are the binary system benzene-toluene and the ternary system benzene-toluene-para-xylene. For the binary system and with column operation at a positive pressure of 15 psig, the existing 50 psig steam will be sufficient. To run with the ternary system at the same pressure, facilities for obtaining 100 psig steam must be installed. Operation of the column at a 15 psig or greater positive pressure is required for proper column control and operation of the sampling systems.

The removable tray package gives this testing unit a major advantage over most others reported in the literature. Most prior investigations used flanged pipe sections to construct the column. The trays were mounted between the flanges. With the removable tray package, the tray type or tray design can be changed with much less difficulty. The procedure would be as follows: disconnect the overhead vapor line, remove the flanged top, disconnect the sample systems and thermocouples, and lift out the tray package. It is not

required that the insulation be stripped off and the entire column be disassembled. Consideration has been given to a series of tests with a sieve tray package as well as the existing valve trays.

Considerations in the design of the instrumentation and controls were as follows. Steady state operation with accurate constant pressure control is necessary for reliable data. Also, a range of operating conditions must be investigated. For a particular run a constant boil-up rate will be set. The instrumentation will provide a complete description of operating conditions. This is desirable both for a study of the effect of operating variables on the tray efficiency and for checking the measured compositions by heat and material balances.

A final factor which influenced this design is the proposed alternate use of the column for studies of distillation transients as well as the proposed tray efficiency investigation.

#### Data Considerations

Tray efficiency data will be obtained in the following fashion. For the specific trays which are being studied, vapor compositions and temperatures both entering and leaving the tray will be measured. Also, the liquid compositions and temperatures entering and leaving the tray will be determined. From this information, the Murphree vapor and liquid tray efficiencies can be determined. Assumptions made are that the vapor leaving a tray is of uniform composition and

that the liquid concentration is uniform at the point where sampled.

These are known to be reliable assumptions, particularly on small diameter columns.

Reliable stream samples will be required. The sample purge line described in the preceding chapter is to assure that the material sampled is representative of the material at the sample point. By operating the bleed by-pass, the sample tubes will be purged and a continuously fresh sample will be available at the sample bomb nozzle. These lines will be purged for approximately 30 minutes after steady state operation is obtained. The rate of purge will be very small so as not to affect the tray material balance. Samples will be taken in duplicate to check their reliability. The second samples will be taken approximately 30 minutes following the first. To compare the reliability of the vapor and liquid samples, the overhead vapor stream and the condensed distillate product stream will be sampled. Analysis should give identical compositions. The accuracy of the tray efficiency data will be determined for binary distillation by checking the results against efficiencies calculated from a McCabe-Thiele diagram. Identical accuracy should be realized for the ternary distillation.

Also of interest is the prospect of investigating the generalized tray efficiency defined by Standart. As a complete description of the tray operation will be measured or calculable, data for the Standart efficiency can be obtained.

## CHAPTER V

### CONCLUSIONS AND RECOMMENDATIONS

A 12-inch diameter distillation column for tray efficiency studies was constructed. It was designed for operation at steady state as a total refluxed column or a non-refluxed stripper. Based on results reported for tray efficiency investigations with similar equipment, the column will maintain a steady state operation and accurate tray efficiency data can be obtained.

The following expansion and modification of the project carried out thus far may be made:

1. Install 100 psig steam facilities.
2. Insulate the column, reboiler, and piping. Two inch thick, 85 per cent magnesia would be used with an aluminum jacketing for weatherproofing.
3. Obtain a tray package with sieve trays for comparative studies with valve trays.
4. Obtain other systems for study in addition to the present benzene-toluene and benzene-toluene-para-xylene systems.

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## VITA

Michael Dean Burns

Candidate for the Degree of

Master of Science

**Thesis: A DISTILLATION COLUMN FOR TRAY EFFICIENCY STUDIES**

**Major Field: Chemical Engineering**

**Biographical:**

**Personal Data:** Born in Wewoka, Oklahoma, May 2, 1941, the son of Kenneth H. and Doris L. Burns. Married to Linda Lou Cox, Eldorado, Arkansas, in June, 1963.

**Education:** Attended elementary and junior high school in Eldorado, Arkansas; graduated in 1959 from Eldorado High School; received the Bachelor of Science degree from the University of Arkansas, Fayetteville, in May, 1964; completed the requirements for the Master of Science degree in May, 1966.

**Membership in Scholarly or Professional Societies:** Tau Beta Pi, Theta Tau, Pi Mu Epsilon, Omega Chi Epsilon, American Institute of Chemical Engineers.

**Professional Experience:** Employed as a Summer Engineer by Shell Oil Company during the summer of 1961; employed during the summer of 1962 as a Production Engineer by Humble Oil and Refining Company; employed by the Research and Development Division of the Ethyl Corporation during the summer of 1963; employed as a Process Design Engineer by Esso Research and Engineering Company during the summer of 1964.