DISTILLATION TRAY EFFICIENCIES -
AN EXPERIMENTAL STUDY
WITH A TERNARY SYSTEM

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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 growing application of vacuum distillation and use of more expensive materials of construction, the need for a better understanding of the factors affecting the efficiency is obvious. Expansion of technology into new and unusual areas has led to demands for an accurate method of predicting efficiencies.

A great deal of experimental work on tray efficiencies has been done and an increasing number of factors affecting efficiencies have been found. These investigations have led to several methods for predicting tray efficiency. Methods in the literature are concerned with correlations for the overall column efficiency or the Murphree tray efficiency averaged to an overall efficiency. These correlations were developed almost exclusively for binary systems, but most industrial distillations involve more than two components.

The purpose of this study was to obtain data from trays in an operating distillation column employing a ternary system and to calculate and evaluate the tray performance with these data. The specific objectives included the following: to review and evaluate the various tray efficiency concepts and prior work; to obtain the experimental data necessary for tray efficiency calculations from an operating column; to calculate the Murphree tray efficiencies for these data and to develop a procedure for and to calculate the generalized tray efficiencies defined by Standart; and to determine if these efficiencies can be correlated as a function of the variables studied.

In order to carry out these objectives, a 12-inch diameter distillation column with 10 valve trays was operated at a steady state, total reflux condition. The ternary system benzene-toluene-para-xylene was used. System composition and column loading were varied. Data from this equipment were analyzed by gas chromatography and reduced by a set of computer programs developed to calculate the tray efficiencies. On the basis of these results, comments and recommendations were made as to the value and applicability of the generalized efficiencies as compared to Murphree efficiencies.

CHAPTER II

TRAY EFFICIENCY CONCEPTS

Tray efficiency in general is considered to be a measure of the degree of 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. Several different definitions of tray efficiency have been developed. Here, we will examine these different concepts and look at their limitations and interrelations. We will present and evaluate a generalized equilibrium tray definition and set of generalized tray efficiencies as conceived by Standart (38). A procedure for evaluating the generalized equilibrium state will be developed.

Review of Tray Efficiency Definitions

The most common and most widely used definition of tray efficiency is the overall column efficiency. Lewis (29) defined overall column efficiency as the ratio of the total number of ideal or equilibrium trays to the number of actual trays in an operating column that will give an equivalent separation. Although this is obviously the simplest approach, this definition implies the same degree of approach to equilibrium for all components in the distillation system. Unless the column efficiencies for all constituents are the same, the ideal and actual column will not yield the same product concentrations for all components. When application of an overall efficiency is made to calculations, the variable is the number of trays required for the same separation with the ideal and actual columns. With individual tray efficiencies, the variable is the degree of separation obtainable with one tray. The latter concept is the only one which permits generalization to multicomponent mixtures.

Efficiencies of individual trays will be considered with the following. Figure 1 is a schematic diagram of a distillation tray and the vapor and liquid streams to and from the tray. The trays are numbered from the bottom of the column up.

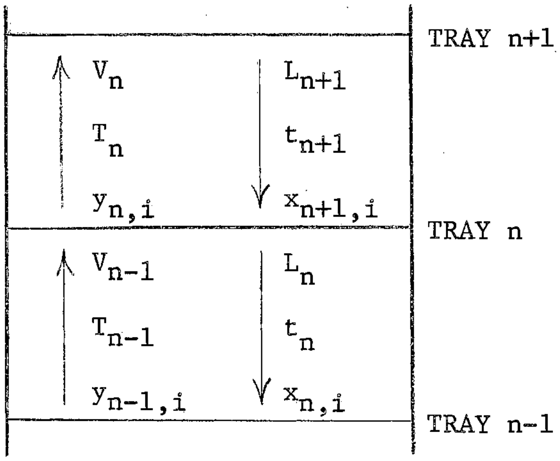


Figure 1

Schematic Distillation Tray

In this figure, V and L represent the total vapor and liquid rates. The mole fractions of the component i are given by y_i and x_i and the temperatures by T and t for the vapor and liquid streams respectively.

Murphree Efficiency

The Murphree tray efficiencies (32) are concerned with the degree of approach to equilibrium on specific trays and involve each component of a mixture individually. They can be defined either for the vapor phase or the liquid phase. The Murphree vapor efficiency is defined as

$$E_{MV_{n,i}} = \frac{y_{n,i} - y_{n-1,i}}{y^*_{n,i} - y_{n-1,i}} \tag{1}$$

Here $(y_{n,i} - y_{n-1,i})$ is the change of composition that actually occurs for component i across tray n and $(y^*_{n,i} - y_{n-1,i})$ is regarded as the change across the corresponding equilibrium tray. The term $y^*_{n,i}$ is the composition of component i in the vapor that would exist if the vapor leaving tray n were in equilibrium with the actual liquid leaving the tray. In a similar manner the less commonly used Murphree liquid efficiency is defined as

$$E_{ML_{n,i}} = \frac{x_{n,i} - x_{n-1,i}}{x^*_{n,i} - x_{n-1,i}} \tag{2}$$

and $x^*_{n,i}$ is the composition of component i in the liquid in equilibrium with the actual vapor leaving the tray.

These definitions of Murphree tray efficiency are for the overall tray and involve the average vapor and liquid composition leaving the tray. This means that both the liquid and vapor leaving a given tray are assumed to be completely mixed and as a consequence no concentration gradient exists across the tray. The assumption of complete mixing is unrealistic except for special cases in very small columns. Incomplete liquid mixing and vapor channeling are two common conditions which will result in deviations from Murphree's assumption.

The Murphree point vapor efficiency describes the degree of approach to equilibrium between the vapor and liquid at a single point on a tray.

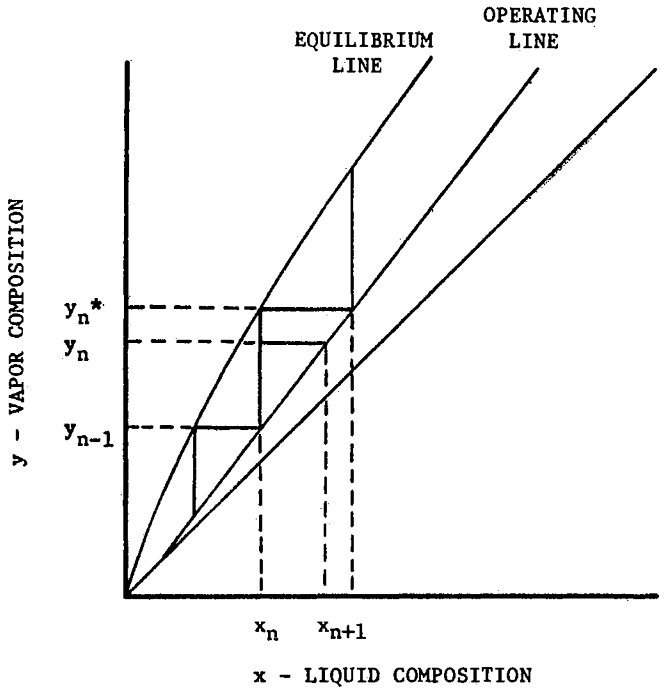
The definitions of the point and overall tray efficiencies are analytically identical except that individual point and average compositions must be used. Since the degree of mixing on the tray will have no effec on the value of the point efficiencies, they will depend entirely on the transport properties of the system.

Several limitations of the Murphree efficiencies have been discussed by Standart (38). Murphree efficiencies are concerned only with mass transfer on the tray and ignore the transfer of heat. Constant molal flow rates along the column were assumed when Murphree defined the efficiencies and application includes this assumption since the variable is mole fraction of each component and not total moles of each component. The vapor and liquid efficiencies are related but not equivalent. Looking at McCabe-Thiele diagrams for a binary system, Figure 2 indicates the differences in the two efficiencies. Figure 2a shows application of the vapor efficiency when calculating up the column. Figure 2b is for application of the liquid efficiency when calculating down the column. The vapor and liquid efficiencies can be related by assuming the vapor-liquid equilibrium relationship is linear over the composition range of the tray. The relationship is given by

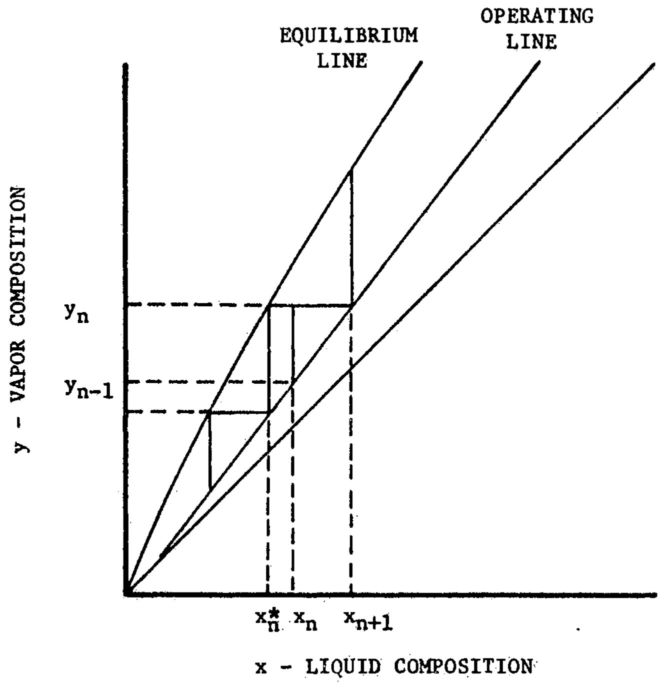
$$\left(\frac{1}{E_{ML}} - 1\right) = \left(\frac{1}{E_{MV}} - 1\right) \frac{L}{kV} \tag{3}$$

where k is the slope of the equilibrium relationship. Therefore, except where the operating and equilibrium lines are parallel, the vapor and liquid efficiencies are not equal.

Criticism has been made of the Murphree efficiencies concerning unsymmetrical definitions and phase saturation in the actual column. Looking again at Figure 2 we see that there is no direct relationship between the equilibrium vapor, y^*, defined for the Murphree vapor efficiency and the equilibrium liquid, x^*, defined for the Murphree liquid efficiency. In other words, these vapor and liquid compositions are not in mutual thermodynamic equilibrium. Or, as stated by Standart (38), "the Murphree definitions



(a) MURPHREE VAPOR EFFICIENCY



(b) MURPHREE LIQUID EFFICIENCY

Figure 2. Murphree Efficiencies and McCabe-Thiele Diagrams

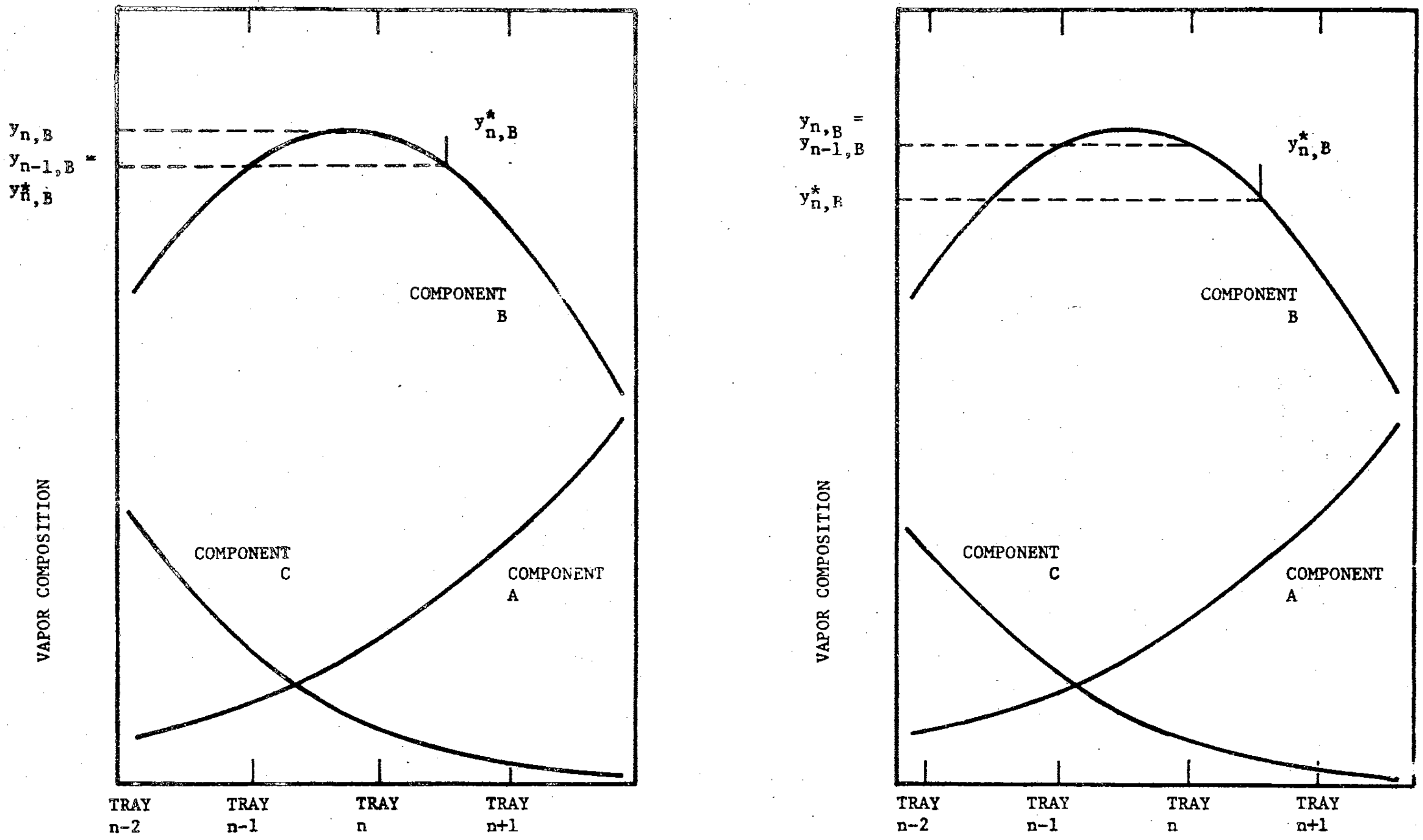
implicitly require that certain of the streams on the actual tray be saturated -- yet the attainment of saturation by a stream leaving the tray depends on which Murphree efficiency we are using." The saturated state refers only to bubble point or dew point compositions - the temperature equilibrium question being ignored.

One of the most serious problems with Murphree efficiencies can be encountered when applied to multicomponent systems. With a system of three or more components, the Murphree efficiency definition is unbounded; values ranging from $-\infty$ to $+\infty$ can be obtained. Examples of this problem are illustrated in Figure 3. A vapor composition profile is illustrated for a ternary system A-B-C. The composition for component B passes through a maximum value and it is in such a case that unbounded values of the Murphree efficiency occur.

An undefined Murphree vapor efficiency is illustrated in Figure 3a. The measured composition change for component B across tray n is some finite value; $y_{n-1,B} < y_{n,B}$. The Murphree vapor efficiency for component B is undefined where the corresponding composition change across the equilibrium tray is zero.

$$\lim_{y^*_{n,B} \to y_{n-1,B}} E_{MV_{n,B}} = \lim_{y^*_{n,B} \to y_{n-1,B}} \left[\frac{y_{n,B} - y_{n-1,B}}{y^*_{n,B} - y_{n-1,B}} \right] = \infty \qquad (4)$$

Figure 3b illustrates a condition where the Murphree vapor efficiency



(a) $E_{MV_{n,B}} = \infty$

(b) $E_{MV_{n,B}} = 0.0$

Figure 3. Ternary Composition Profile

is equal to zero. Here, the measured change of component B across the actual tray is zero. If $y_{n,B}^{*} < y_{n,B} = y_{n-1,B}$ then $E_{MV_{n,B}} = 0$.

If the composition profile for a component passed through a minimum, efficiencies ranging to negative infinity would be obtained by similar arguments. Therefore, the Murphree vapor efficiency is unbounded. The Murphree liquid efficiency is likewise unbounded.

Temperature Efficiency

Cary, as reported by Nord (34), 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

$$E_{TV_n} = \frac{T_n - T_{n-1}}{T_n^{*} - T_{n-1}} \qquad (5)$$

where T_n^{*} is the vapor temperature in equilibrium with that of the liquid leaving tray n, i.e., equal to it. Similarly for the liquid phase, the liquid temperature efficiency is given by

$$E_{TL_n} = \frac{t_n - t_{n+1}}{t_n^{*} - t_{n+1}} \qquad (6)$$

where t_n^{*} is the liquid temperature in equilibrium with the vapor leaving the actual tray n, i.e., equal to it.

These thermal efficiencies were developed analogously to the Murphree efficiencies. As a result, most of the criticisms claimed for Murphree efficiencies analogously apply to Cary's efficiencies. As with the Murphree model, inequality of the thermal vapor and liquid efficiencies, lack of symmetry in the definition of the equilibrium states, and the question of saturation and thermal equilibrium of the phases in the actual column give a forced approach to realism.

Haunsen Efficiency

Haunsen (25) has defined a distillation tray efficiency for binary systems based on a special tray model whereby co-current vapor and liquid contact is assumed. The equilibrium state is determined from an equation of the form

$$Vy_{n-1,i} + Lx_{n+1,i} = Vy_{n,i} + Lx_{n,i} = Vy^*_{n,i} + Lx^*_{n,i} \tag{7}$$

Here constant molal rates are assumed -- denoted by the constant V and L terms -- and the equilibrium state exists between the ideal vapor and liquid compositions leaving the tray independent of the actual vapor and liquid compositions. This concept differs from that of Murphree. Haunsen efficiency can be defined as

$$E_{GL_{n,i}} = \frac{y_{n,i} - y_{n-1,i}}{y^*_{n,i} - y_{n-1,i}} = \frac{x_{n,i} - x_{n+1,i}}{x^*_{n,i} - x_{n+1,i}} \tag{8}$$

The composition of the entering streams is kept constant and the efficiency measures the difference in composition of the ideal and actual streams leaving the tray.

Although the efficiency is symmetrically equal for both phases, and for the equilibrium tray the vapor and liquid phases are in concentration equilibrium, Haunsen still leaves room for criticism. He considers only the composition differences and neglects non-constant molal overflow and thermal effects.

Holland's Efficiencies

Holland (27) defines several modified forms of tray efficiencies claimed to be easily applied to Thiele-Geddes type calculations. A modification of the Murphree vapor efficiency is given by

$$E^{M}_{MV_{n,i}} = \frac{y_{n,i} - y_{n-1,i}}{Y_{n,i} - y_{n-1,i}} \qquad (9)$$

The term $Y_{n,i}$ is not the equilibrium bubble point composition for the actual liquid leaving tray n, but is defined by

$$Y_i = K_i \, x_i \qquad (10)$$

The distribution coefficient, K_i, is evaluated for each component at the actual temperature and pressure of the liquid leaving the tray. The modified vaporization efficiency is defined by

$$E^{o}_{n,i} = \frac{y_{n,i}}{Y_{n,i}} \tag{11}$$

where the $Y_{n,i}$ is again defined by equation (10). And finally, a modified heat transfer efficiency was defined to be

$$E_{HT_n} = \frac{T_n}{t_n} \tag{12}$$

which is the ratio of the temperatures of the vapor and liquid streams leaving a tray.

These efficiencies were developed for easy computer evaluation of operating column data. With these efficiencies new columns for similar separations could be designed. Most applications involve the vaporization efficiency [Eq.(11)] and these will be reviewed in the following chapter. The vaporization efficiency is bounded for the situation illustrated in Figure 3. However, typical values of this efficiency will be less than unity when the component concentration is increasing up the column (as for component A). For a component whose concentration is decreasing up the column (as for component C), typical vaporization efficiencies will be greater than unity. And where the maximum or minimum profile is encountered (Figure 3), the value will change from less than unity to values greater than unity. These efficiencies offer no physical realism in measuring the degree of approach to ideal tray calculations. No mention is made of corresponding liquid phase efficiencies.

The Generalized Equilibrium Tray and Generalized Tray Efficiencies of Standart

One of the most significant advances in tray efficiency concepts was introduced by Standart (38). This is a generalization of the Haunsen efficiency model and incorporates consideration of both the heat transfer and mass transfer processes occurring on a distillation tray. An equilibrium state whereby the ideal tray can be defined is given by the following equations. An overall material balance gives

$$V_{n-1} + L_{n+1} = V_n + L_n = V_n^* + L_n^* \qquad (13)$$

A balance for each constituent, i, gives

$$V_{n-1}y_{n-1,i}+L_{n+1}x_{n+1,i} = V_n y_{n,i}+L_n x_{n,i} = V_n^* y_{n,i}^*+L_n^* x_{n,i}^* \qquad (14)$$

and, an enthalpy balance gives

$$V_{n-1}H_{n-1}+L_{n+1}h_{n+1}-Q_n = V_n H_n+L_n h_n = V_n^* H_n^*+L_n^* h_n^* \qquad (15)$$

The terms without asterisks are as indicated by Figure 1. H and h denote the molal vapor and liquid enthalpies respectively and Q_n is the rate of heat loss from the nth tray. The assumption is made that the rate of heat loss from the actual and ideal trays is the same. The equilibrium state quantities are denoted by the * values. In addition to the material balance and enthalpy balance relationships, the equilibrium state requires

$$T_n^* = t_n^* \tag{16}$$

and

$$\mu_{V,i}^* = \mu_{L,i}^* \tag{17}$$

for complete definition. These indicate that the temperature of the vapor and liquid streams leaving the ideal tray must be equivalent and for each component the chemical potentials, , of the two phases are equal.

Based on the above definitions a set of generalized tray efficiencies may be defined by the following equations. The overall material efficiency is given by

$$E_n = \frac{V_n - V_{n-1}}{V_n^* - V_{n-1}} = \frac{L_n - L_{n+1}}{L_n^* - L_{n+1}} \tag{18}$$

The efficiency for the ith component is

$$E_{n,i} = \frac{V_n y_{n,i} - V_{n-1} y_{n-1,i}}{V_n y_{n,i} - V_{n-1} y_{n-1,i}} = \frac{L_n x_{n,i} - L_{n+1} x_{n+1,i}}{L_n^* x_{n,i}^* - L_{n+1} x_{n+1,i}} \tag{19}$$

And the enthalpy efficiency is given by

$$E_{H_n} = \frac{V_n H_n - V_{n-1} H_{n-1} + r_n Q_n}{V_n^* H_n^* - V_{n-1} H_{n-1} + r_n Q_n} = \frac{L_n h_n - L_{n+1} h_{n+1} + (1-r_n) Q_n}{L_n^* h_n^* - L_{n+1} h_{n+1} + (1-r_n) Q_n} \tag{20}$$

Here r_n is the fraction of the heat lost on tray n by the vapor stream. One can show that the overall material efficiency can be expressed as the weighted mean of the component efficiencies. Therefore it is a dependent variable, leaving equations (19) and (20) as the generalized efficiencies.

No assumptions are made in these definitions concerning the equilibrium tray, constant molal overflow, etc. Both mass transfer and heat transfer aspects are considered. The efficiencies for the vapor and liquid phases are symmetrical and equal. However, the component efficiencies, $E_{n, i}$, can exhibit unrealistic behavior for situations with multicomponent systems as illustrated in Figure 3. Where maximums or minimums are encountered in a composition profile, the component efficiency is unbounded.

Calculational Procedure for Evaluation of the Generalized Equilibrium State

Standart has presented discussion on both the evaluation of the efficiencies from operating data and the calculation of an actual column given values of the generalized efficiencies. Since this study is concerned with measurement of these generalized efficiencies, a calculation method was developed for determining the generalized equilibrium state for a tray from data on an actual operating tray.

Experimental data completely describing the vapor and liquid streams to and from the actual tray were obtained. These data include the measured temperatures and compositions of the vapor and liquid streams to and from the tray. The vapor and liquid molal rates to and from the actual tray are calculated. These are obtained from material and enthalpy balances and the measured reflux rate. The heat leak term, Q_n, given in equation (15) is assumed negligible.

With the operating data, equations (13) through (17) may be solved for the unknown equilibrium values. Use is made of the distribution factor, K_i, for each component. Vapor-liquid equilibrium for each constituent gives

$$y_i^* = K_i \; x_i^* \tag{21}$$

This is an approximation to equation (16) requiring identical chemical potentials of vapor and liquid constituents at equilibrium. The distribution factor, K_i, and the pure component enthalpy values can be obtained as functions of the equilibrium state temperature, $T^* = t^*$. Including the relationship that the sum of the mole fractions of each phase equals unity gives enough independent equations to solve for the unknown state quantities. These include temperature, mole fractions of the components in each phase, and the total stream rates leaving the equilibrium tray. For a binary system a trial and error solution is quite simple. An equilibrium temperature is assumed, the compositions and rates calculated directly, and the assumed temperature checked by the enthalpy balance of equation (15). Convergence is obtained after several trials. For more than two components, the complexity of solving for the compositions and stream rates increases with an increasing number of components. The solution involves systems of nonlinear algebraic equations.

In order to simplify the problem associated with multicomponent systems, a simple but rigorous approach was developed. Assume that the vapor and liquid streams entering the equilibrium tray are completely mixed and leave in an equilibrium state. The completely mixed state on the tray would be analogous to an equilibrium flash calculation where a feed is introduced into a vessel and flashed to equilibrium vapor and liquid streams. By treating the calculation as an equilibrium flash with heat balance, the generalized equilibrium state can be calculated for any number of components without changing the complexity of the calculations.

The procedure is summarized as follows: The vapor and liquid streams to the tray are combined and a total rate and mixing cup composition calculated. An equilibrium temperature is assumed. For the first trial, a temperature corresponding to the average of the temperatures of the vapor and liquid streams entering may be assumed. The problem then is a simple flash calculation for the separation that is obtained at a given temperature (and pressure) -- the trial and error procedures are available in any distillation text book. Results obtained are the compositions and quantities of the vapor and liquid streams. To check the assumed temperature, the enthalpy balance given by equation (15) is employed. Converging to a solution involves repetitive calculations based on successively better temperature estimates until the enthalpy balance checks within a predetermined limit.

With the generalized equilibrium state determined, the generalized efficiencies may be evaluated from equations (19) and (20).

CHAPTER III

LITERATURE REVIEW

Almost all research on tray performance in distillation columns is directed to the Murphree tray efficiency and/or the overall column efficiency. Geddes (19) asserted that at least thirteen independent variables affect the distillation process on a tray. Many of these variables have been studied. These include foaming, surface tension, entrainment, liquid mixing effects, froth height, thermal effects, and composition effects. Only recently have research efforts been extended to multicomponent systems. Rather than attempt a review *in toto* of this voluminous literature, attention will be directed to areas pertinent to this study. A review of experimental investigations of tray efficiencies will be given. Examination of the various correlation-prediction studies for efficiency will be made. Other pertinent investigations will be discussed.

Prior Experimental Investigations

There have been numerous experimental investigations of tray efficiency. Many studies have been made on small laboratory columns or equipment built to simulate distillation trays. Some studies have been made on larger test columns, pilot plant equipment, and industrial columns. Some of these studies will be detailed here -- others briefly described.

Early studies were made by several investigators on small tray equipment as reported by Robinson and Gilliland (36). Gadwa (36) studied six binary systems on a small four-tray column with one bubble cap per tray. Mixtures of benzene-carbon tetrachloride, methanol-isobutanol, methanol-n-propanol, isobutanol-water, n-propanol-water, and methanol-water were used. Samples were taken of liquid on the trays and Murphree efficiencies calculated. He concluded that the efficiency was substantially independent of the concentration and vapor velocity. Lewis and Smoley (36), using an 8-inch diameter, ten tray column with bubble caps found average plate efficiencies of 60 per cent for benzene-toluene mixtures and 75 per cent for the benzene-toluene-xylene system. Using the same column, Carey and co-workers (36) reported an average efficiency of 70 per cent for the benzene-toluene system and 50 to essentially 100 per cent for an ethanol-water mixture in a 6-inch, single-tray, bubble-cap unit. Total reflux operation was employed in each of these investigations and either liquid or liquid and vapor samples were taken.

Operation of an 11-inch diameter ten tray column with bubble caps is reported by Huffman and Treybal (36). Liquid samples were taken from each tray near the downcomer and vapor samples were removed from six inches above the tray. Studies of the carbon tetrachloride-toluene system were made. The column could be operated at total reflux or with feed on any tray.

Several early studies were made by investigators using industrial size equipment. Brown (36) and Guiness (36) independently found Murphree tray efficiencies of greater than 100 per cent for large commercial gasoline stabilizers. Lewis and Wilde (36) found an average tray efficiency of 65 per cent for the rectification of naphtha in a ten tray column 9-feet in diameter.

Efficiency experiments have been conducted by several investigators using Oldershaw columns. Collins and Lantz (8) and Berg and James (3) studied several binary systems. No tray samples could be taken but the overall column efficiency was studied. They found the efficiency nearly independent of thruput and reflux rate.

A tray efficiency study by Grohse, et. al. (22) was made on an extractive distillation system separating C_4 hydrocarbons with furfural. A 13-inch diameter, ten-tray, bubble-cap column was used. Varied conditions of flow rate, composition, temperature, pressure, and tray design were studied. Liquid samples were removed from under the center of the downcomer and vapor samples were withdrawn from under the center of the tray above. The vapor samples were consistently reliable but the liquid sample compositions were scattered. The authors felt this to be a result of concentration gradients. The liquid compositions were calculated from vapor compositions and heat balance relationships. Average values of Murphree tray efficiency were evaluated over a number of trays from a McCabe-Thiele diagram.

From their studies in transient distillation, Armstrong and Wilkinson (1) reported a constant Murphree efficiency of 72 per cent. These efficiencies were determined on a four inch, 21-tray column for varying feed compositions of the benzene-carbon tetrachloride system.

The first major coordinated effort in tray efficiency research was a five year study of bubble tray efficiencies by the AIChE Research Committee (4, 43). The test column employed in these studies was two feet in diameter with five trays. A variety of bubble tray designs was used in several different studies. 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 taken 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. Murphree tray efficiency calculations were made graphically by McCabe-Thiele diagram. Several operating variables for the two systems were studied including operating pressure, vapor rate, and liquid rate through the column.

Of interest in the AIChE report (43) 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. Liquid composition and temperature data for each of the trays were reported for a variety of operating and tray design conditions.

Manning and co-workers (31) have studied various tray designs in a 5-foot diameter test column. The column was 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 column could be operated at total reflux or with feed at several points. The system used for most of the studies was iso-octane and toluene. Liquid samples were removed from the downcomer. Murphree tray efficiencies were calculated.

Van Wijk and Thijssen (44) studied the effect of composition on Murphree tray efficiency for the n-heptane-methylcyclohexane system. An 8-tray sieve plate column 1 1/2-inches in diameter was used. Liquid samples were taken from the tray during total reflux operation. Efficiency was found to drop sharply at composition extremes.

At the International Symposium on Distillation held in 1960, three experimental studies of interest were reported. Haselden and Sutherland (24) studied fractionation of ammonia-water in a 3-inch column with four perforated trays. Composition and reflux ratio were widely varied. Liquid samples and vapor and liquid temperatures were obtained. Murphree tray efficiencies were calculated.

Conclusions drawn included the fact that thermal effects in distillation should be studied. Ellis and Shelton (16) measured efficiencies for the binary system methanol-water in a 4-inch diameter column containing six bubble cap trays. Vapor samples were taken from under the bubble cap and liquid samples from the edge of the downcomer. Tray efficiencies were calculated directly from the equations for the Murphree vapor and liquid efficiency. Efficiencies of greater than 100 per cent were obtained over certain composition ranges and attributed to neglect of thermal effects in the measurements. Free and Hutchison (18) made experimental studies of two ternary systems -- acetone-methanol-ethanol and acetone-benzene-chlorobenzene. A 4-inch diameter, four tray bubble cap column was employed. Total reflux operation was used and liquid samples from each tray obtained. Results showed that differing diffusivities of the components in the system can yield different Murphree efficiencies for each component on a tray.

Hay and Johnson (26) investigated sieve tray efficiencies for the methanol-water system. An 8-inch diameter five tray column was operated at total reflux. Liquid samples were taken from the downcomers. Vapor velocity and concentration gradients were studied. Foaming was visually examined. The Murphree efficiencies were calculated and ranged from 82 to 105 per cent.

Charyavich and Van Winkle (7) studied the effects of system properties on tray efficiency. A 1-inch diameter column with a perforated plate was used. The unit was operated at total reflux. Several binary systems were used in order to obtain a variety of system property effects. These included: propanol-water, octane-toluene, acetone-butanol, methanol-dioxane, ethanol-dioxane, and carbon tetrachloride-propanol. Murphree tray efficiency was found to be a function of relative volatility, surface tension, viscosity, density, and diffusivity.

A uniquely designed column was employed by Liang and Smith (30) for the study of thermal effects in distillation. Using an inverted pear-type wetted wall column, liquid samples and liquid and vapor temperatures were obtained. Both the cyclohexane-toluene and methanol-water systems were employed at total reflux operation. Of particular interest was the fact that the measured vapor phase temperatures were less than the saturation temperatures for several of the "trays". Thermal effects were demonstrated to play an important role in the value of the Murphree tray efficiency.

Dale et. al. (9) report on a study of tray efficiency in a 12-inch diameter valve tray column. This column is almost identical to that used by this author. Total reflux operation was studied and vapor samples were taken from alternate trays. The overall column efficiency was determined and compared at various operating conditions.

A single tray bubble cap column was used by Bakowski (2) to investigate efficiency of several binary systems. The column was a 4-inch diameter unit with total reflux operation. Liquid samples to and from the tray were taken as well as temperature measurements. The systems methanol-water, acetone-water, trichloroethylene-toluene, benzene-toluene, and water-acetic acid were investigated. Murphree efficiencies were calculated for each system over a range of compositions and vapor rates.

Dynamic distillation column studies were reported by Murrill (33). He operated an 8-inch by 20-inch column with five sieve trays employing the benzene-acetone system. Initial steady-state, total-reflux data were obtained including samples of both vapor and liquid around the middle tray. Liquid samples were taken by syringe and vapor samples were collected in polyethylene bags. Efficiency of the middle tray was determined by an overall efficiency calculation.

An experimental study was made by Diener (13) on Murphree tray efficiencies for the ternary system acetone-methanol-water and the three corresponding binary systems. A two-tray, rectangular, split-flow, sieve-tray column was employed with 5 by 6-inch dimensions. Total reflux operation was used and liquid samples were removed from the downcomers and from the reflux line. Results of the study indicated that where ternary diffusional interactions must be considered, the Murphree efficiency of each component would differ, but where the three binary diffusion coefficients were equal an average binary efficiency would approximate the ternary efficiencies.

The analysis of the performance of an industrial methanol distillation apparatus is discussed by Gelbin (20). The column was

2.9 meters in diameter and employed bubble cap trays. The performance of the 15 trays below the feed tray were studied. Liquid samples and temperature measurements were taken from the trays. Five components were involved -- methanol, ethanol, n-propanol, isobutanol, and water -- and the Murphree efficiencies evaluated for each. The Murphree vapor efficiencies varied from 60 per cent for water to 150 per cent for isobutanol. Measured values of the Murphree liquid phase efficiency were reported to be meaningless for column calculations.

Hartman and co-workers (23) report on an experimental study of the performance of turbogrid trays in a 6-inch diameter column. Three to five trays were employed. Two binary mixtures were used: methanol-water and methanol-isopropanol. Pressure probes were mounted below each tray. Samples and temperatures were taken of each stream. A covered sample probe located under the tray above was used for vapor sample withdrawal. Liquid samples were taken from on the tray. Operating and design variables such as free plate area, plate spacing, vapor velocity, and reflux ratio were studied. Conclusions were made that the Haunsen type efficiency should be used rather than the Murphree efficiency.

In a recent study, Kastanek and Standart (28) discuss an experimental evaluation of several types of distillation trays in a large column at total reflux. Tray types included bubble cap, sieve, Uniflux, APV-West, Ripple, and Turbogrid. A 39-inch diameter test column was used with up to five trays. The methanol-water system was used. Except for the top and bottom trays, samples and temperature measurements were taken for both the vapor and liquid streams around each tray. Liquid samples were taken from the downcomer or just under a slot or hole in the tray. A great deal of effort was spent developing a vapor sample device which would centrifically remove any entrained liquid. Hence "dry" vapor samples were obtained. The performance of the various trays (versus vapor velocity) was studied. Both Murphree vapor efficiencies and the generalized Haunsen type efficiencies are reported. Though very little detail was given, the latter type was interpreted by this writer to be the generalized component efficiency of interest in this dissertation.

In summary, the experimental studies on efficiencies involve a wide range of equipment, systems, and techniques for investigation. The early experiments were involved with attempts at obtaining efficiencies for various systems. Bubble cap trays were used almost exclusively. As more and more of the factors affecting tray performance were recognized, experimental programs investigating these particular factors were conducted. The AIChE program was the first large scale program designed to study all the known factors affecting efficiencies and with industrial size equipment. As a result of the AIChE program, many new experimental programs were initiated. Some were attempted improvements of the AIChE work, others were investigations of points where the AIChE work was incomplete such as other tray types and multicomponent efficiencies. A large volume of experimental data has been taken in attempts to develop a more general empirical correlation for efficiencies. The thermal effects and surface tension factors have been of interest. In general, all this work has been directed toward the Murphree efficiency concept. The experimental work was done for the purpose of evaluating or arriving at Murphree efficiencies. In several very recent articles, questions of the value of using the Murphree concept have been raised. Here, finally seems to be the realization that Murphree's approach was either incomplete or inadequate.

Correlation -- Prediction Studies

Correlation -- prediction studies for tray efficiency have been approached from two concepts: the empirical and the fundamental. Both methods of attack have been directed toward the overall column efficiency and/or the Murphree tray efficiency. The empirical approach involves correlating any or all of the design, operating, and system property variables encountered on the distillation tray as they affect efficiency. The fundamental approach incorporates the use of mass transfer theory and liquid mixing concepts to characterize the tray performance.

Walter and Sherwood (46) developed one of the most important of the early efficiency correlations. This fundamental correlation was based on the derivation of the Murphree equation and centered around the resistance to mass transfer. Walter and Sherwood (46) separated the overall mass transfer resistance into liquid and vapor film resistances. This correlation was developed for bubble cap tray columns.

Drickamer and Bradford (14) utilized plant test data from refinery fractionating columns to empirically 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. In their work they found that the length of the liquid path across the tray was important.

An extension of the Drickamer-Bradford correlation was developed by O'Connell (35). This correlation relates the overall efficiency for fractionating columns to the product of the relative volatility of the key componenets and the molal average liquid viscosity of the feed. The use of relative volatility implies that the plate efficiencies of various components in a multicomponent system are not the same.

The AIChE Research Committee sponsored a five-year study of bubble tray efficiencies. The aim of the program was to develop a method of predicting efficiencies for bubble trays in commercial distillation columns. The result was the publication of a Bubble Tray Design Manual (4) incorporating correlations based on fundamental models of the transfer processes occurring on the tray.

The AIChE method (4, 43) 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. Addition of the resistance in the vapor phase and the resistance in the liquid phase gives a total resistance to mass transfer from one bulk phase to the other. Correlations were obtained for each of the two resistances. 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 (39) introduced a model which accounts for the effect of liquid and vapor bypassing on the tray. Eduljee (15) suggested different correlations for the hydraulic behavior and the tray efficiency which give improved results. These correlations were based on additional data to that used for the AIChE correlation.

Chatyavech and Van Winkle (7) developed an empirical correlation for system property effects on tray efficiency in small diameter columns. The variables considered were surface tension, relative volatility, viscosity, density, and diffusivity of both vapor and liquid phases. The correlation was effective for selected sets of literature data.

English and Van Winkle (17) 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 predominately affect efficiency. These were fractional free area, weir height, reflux ratio, the liquid Schmidt number, and surface tension. Data from binary systems were employed.

Bakowski (2) derived an equation for predicting tray efficiency in bubble cap columns and verified its applicability for experimental and some published data. Mass transfer rate was assumed to depend on concentration, vapor pressure, rate of renewal of liquid surface, and interfacial area. Only binary systems were considered.

While these methods are helpful in attempts to predict the column performance where no previous experience exists, the limitations are obvious. Except for specific cases, the methods are applicable only to binary systems and most of the correlations apply only to bubble cap trays. Many effects only recently studied were not included. And all work points to mass transfer considerations and the Murphree or overall efficiency. Though the AIChE program showed that tray efficiency can be correlated and predicted from the more fundamental approach, some authors still contend that the problem is too complex for this type attack. Some physical properties used in the correlations cannot be predicted or measured conveniently. The empirical methods are either untested or unreliable except for systems for which they were developed.

Other Work

Toor and Burchard (42) 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 Murphree efficiencies in ternary systems can be markedly different from the binary efficiencies under the same flow conditions. Minor diffusional interaction effects changed the efficiency only slightly while strong interaction effects exhibited by one of the three components resulted in an efficiency for the component that varied by as much as 60 per cent from its corresponding binary efficiency.

Walsh (45) commented on multicomponent efficiencies. He proposed that when two components are similar and one if different, the efficiencies of the similar components will be low. The efficiency of the dissimilar component will be close to that of the binary efficiency.

Gerster (21) reflected on the use of the AIChE tray efficiency method for multicomponent mixtures. He reported that the method could be used in certain instances. If each of the binary pairs in a multicomponent mixture has about the same gas-phase diffusivity, then the multicomponent efficiencies will equal the binary efficiencies. In another case, if two components comprise nearly all the mixture, then the two components will also have an efficiency equal to their binary efficiencies. In the general case where each of the binary pairs have unequal gas-phase diffusivities, the computation is quite complex.

Holland and co-workers (11, 12, 27, 40) have developed methods for determining the modified Murphree vapor efficiency and the vaporization efficiency from operational data on multicomponent systems. The vaporization efficiency is claimed to be superior from a computational point of view. The vaporization efficiencies were determined from field tests for several operating conditions. From these efficiencies, the efficiencies at any intermediate set of operating conditions could be obtained by interpolation or correlation. Hence new columns could be designed from efficiencies obtained on similar units.

The vaporization efficiency as given in equation (11) is evaluated by breaking the efficiency into a component factor and a plate factor. The component factor is essentially a mass transfer function (analogous to a Murphree point efficiency). The plate factor accounts for all the remaining effects necessary to yield the calculated component vaporization tray efficiency.

CHAPTER IV

DESCRIPTION OF EQUIPMENT

The distillation system 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; the vapor and liquid sampling systems; and the utilities. A gas chromatograph was used for sample analysis.

The distillation system is shown in the schematic flow diagram presented in Figure 4. It is designed for operation both at total reflux and as a non-refluxed, stripper with continuous feed on the top tray. The experimental runs were made exclusively at total reflux and the equipment required for the non-refluxed, stripping operation will not be detailed here. For total reflux operation there is no feed and no bottom product or distillate product is removed from the system. The overhead vapor is totally condensed and the reflux-feed pump is used to pump the condensate from the distillate accumulator, through the reflux-feed preheater, and onto the top tray of the column. The tanks, bottoms product cooler, and bottoms product pump are not employed for total reflux operation.

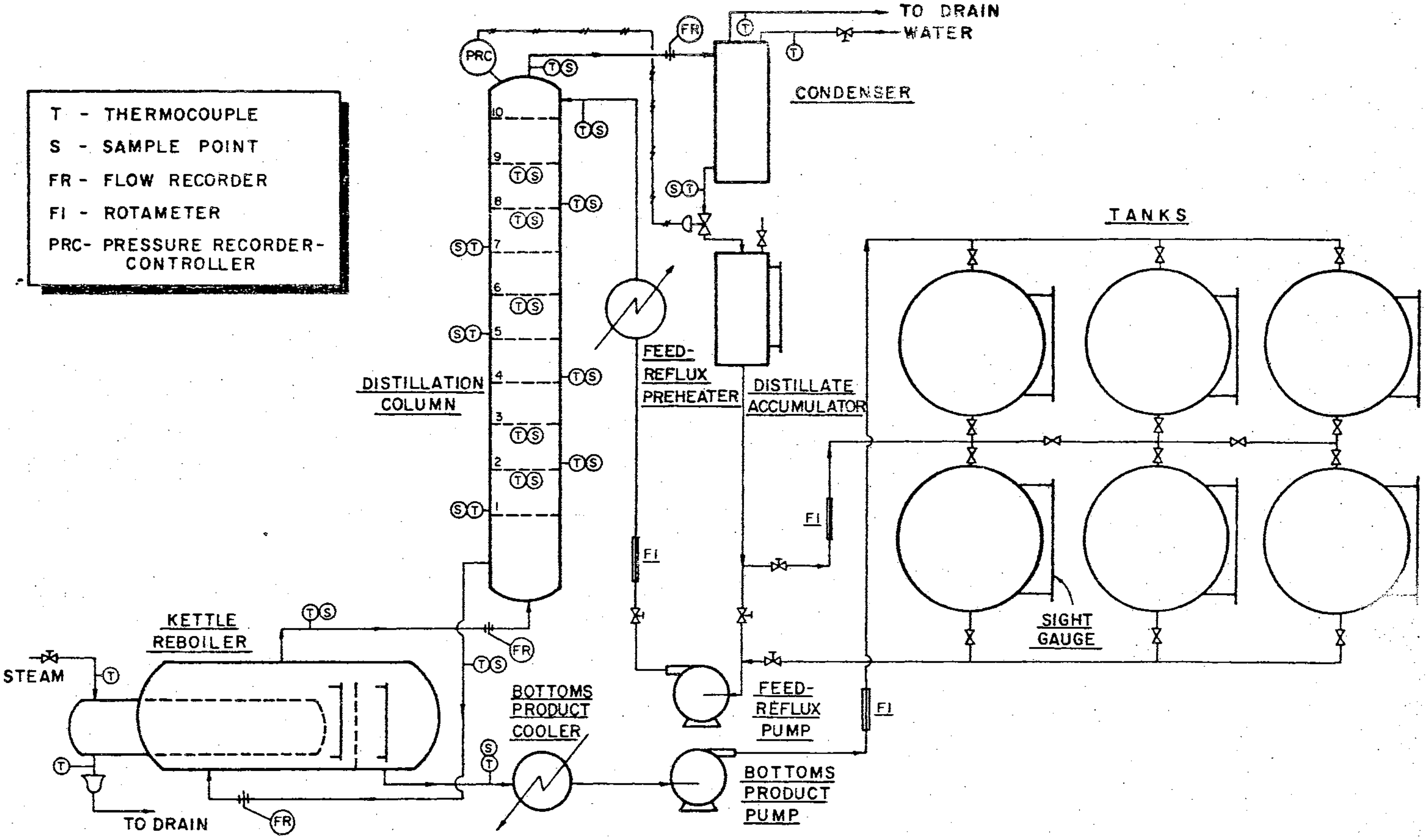


Figure 4. Schematic Equipment Flow Diagram

Distillation Column

The distillation column is a 12-inch diameter column equipped with ten Nutter float valve trays on 12-inch tray spacing. A detailed diagram of the column and the tray assembly is shown in Figure 5. 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 are single-cross-flow type and have a 2-inch weir height, 1-1/2 inch downcomer escape height, and 0.0702 sq. ft. downcomer area. There are six Nutter valves per tray. The top tray is equipped with an entrance baffle for the reflux stream. The downcomer for the bottom tray has a liquid 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. A sight gage was installed to monitor the liquid level in the bottom of the column. 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

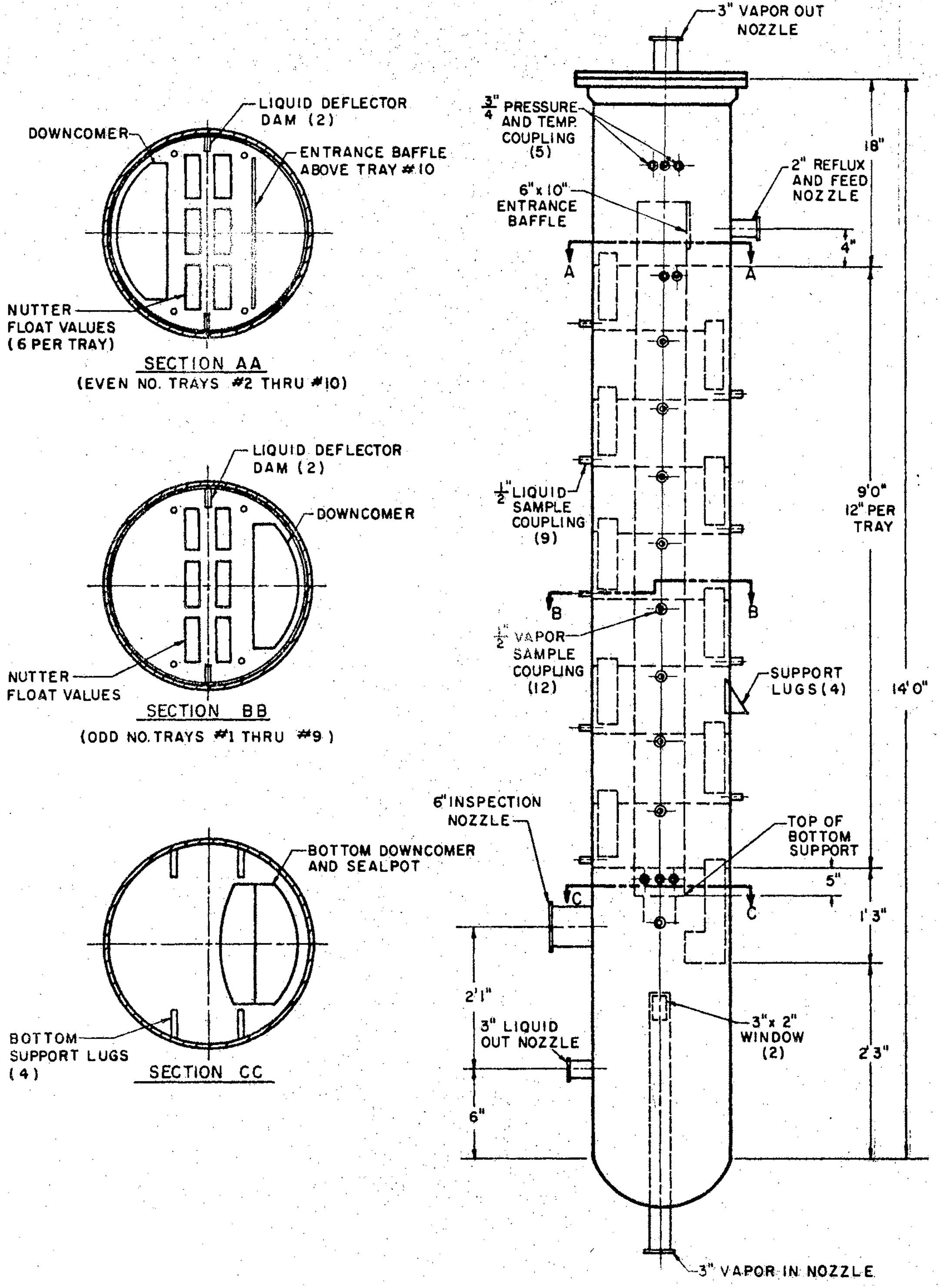


Figure 5. Detailed Diagram of Column

four verticle support rods.

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

The column, reboiler, and associated piping were insulated for conservation of heat. Two-inch thick, 85 per cent magnesia was used for the column shell and piping. Two-inch thick fiberglass material was used for the reboiler. All of the insulation was jacketed with aluminum for weatherproofing.

Associated Equipment

The equipment required for operating the distillation column at total reflux consists of: the reboiler, the condenser, the reflux-feed pump, the reflux-feed preheater, and the distillate accumulator. The bottom product pump and cooler and the tanks are not used during the actual operation but serve for startup, shutdown, and composition change purposes. Specifications for the equipment are as follows:

1. The reboiler is a U-tube kettle type exchanger 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. Gauge glass connections for both sides of the weir, pressure gauge connections, and thermowells are provided as are inlet and outlet nozzles. Saturated steam is used as the heat source.

2. The condenser is a Ross BCF603 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. The reflux pump is a two-stage Eastern centrifugal pump model 2J34D 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. The reflux preheater is a Whitlock type HT-4-B-CI shell and tube exchanger of brass and bronze construction. The heater is vertically mounted with reflux on the tube side and condensing steam on the shell side. The tube side is four pass with 5/8-inch by 24-inch tubes. The heat transfer area is 6.5 sq. ft.

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

Details on the remaining equipment have been presented previously (5, 6).

Instrumentation and Controls

The instrumentation and controls for the distillation column are also indicated schematically in Figure 4. The flow rates and temperatures of the streams around the column were measured and recorded. The pressure drop across the ten trays in the column was measured. Temperatures of the vapor and liquid streams within the column were measured. Column pressure was controlled automatically and recorded while the other controls were manually operated.

Both rotameters and orifice meters were used to monitor and measure stream rates. The reflux rate was measured by an in-line rotameter as shown in Figure 4. A Fisher-Porter rotameter was employed and the calibration is discussed in Appendix C. Similar rotameters were available for continuous feed operation to measure distillate and bottom product rates. The vapor stream from the reboiler to the bottom of the column and the liquid stream from the column to the reboiler were monitored by orifice plates flanged in the lines. These were coupled to American Meter Company disk chart recorders by the use of seal pots with ethylene glycol as the sealing liquid. The recorders were mounted in the control panel. Accurate calibration attempts were never very successful (5). Since these two stream rates could be obtained by heat and material balance calculations, the orifice equipment was used only to monitor for a steady rate.

The pressure drop across the column trays was measured during operation. This was done by measuring the gauge pressure from the two manometers attached to the column as shown in Figure 4. Two 6-inch diameter seal pots with ethylene glycol as the intermediate fluid were used for connecting the manometers to the column.

Thermocouples were provided for temperature measurement at the points indicated on Figure 4. All of these assemblies were Conax, copper-constantan, bare-wire thermocouples with a stainless steel sheath. Thermocouple calibrations are given in Appendix B. Copper-constantan lead wire was employed up to the monitoring and measurement recorders. Employing these leads up to the measurement instrument prevents the formation of extraneous EMF at the thermocouple head. A thermocouple switching panel with ice bath reference junction was used. The circuitry is shown schematically in Figure 6 for two of the 24 thermocouples monitored. The switching panel allowed the thermocouples to be monitored on a multipoint recorder or measured on a potentiometer or milivolt recorder. Twelve of the temperature points could be monitored continuously by a Honeywell-Brown Electronic 24-point multipoint temperature recorder. With this instrument each of the 12 monitored temperature points could be measured and printed on roll chart every 30

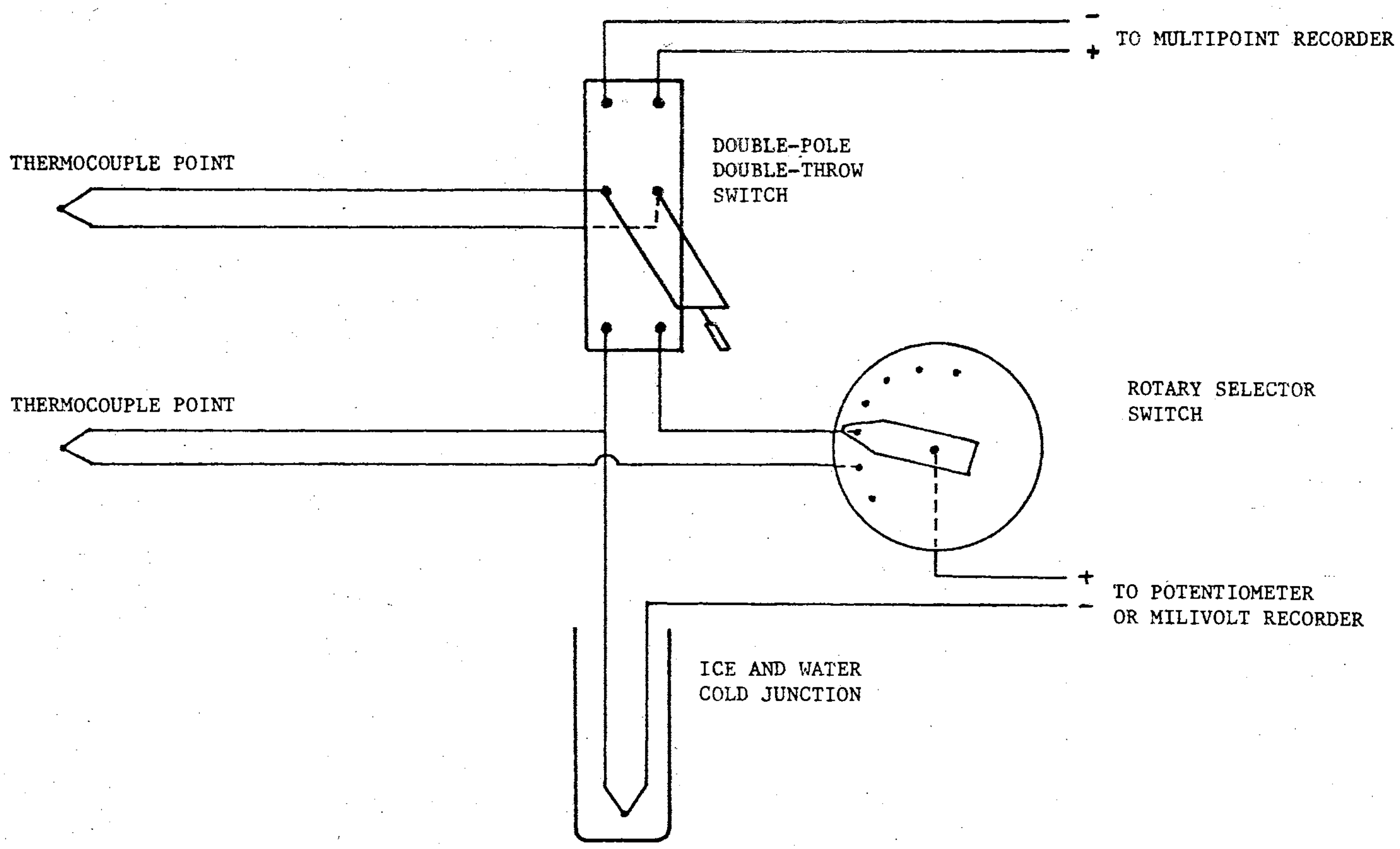


Figure 6. Schematic Thermocouple Circuit Diagram

seconds. This recorder had a temperature range of 0 to 400 degrees F. It was located in the control panel. For the actual temperature measurements a Leeds and Northrup model 8686 millivolt potentiometer was used. The thermocouple switching panel allowed each thermocouple to be individually switched to the potentiometer circuit for measurement. For continuous monitoring of individual temperature points a Bristol Dynamaster Recorder was used. This was a 5-range millivolt chart recorder. The EMF of individual thermocouples could be recorded on chart paper over a variable range scale of from 0-1 millivolt to 0-5 millivolt. This instrument allowed study of the dynamic behavior and temperature fluctuations in the column. The unit was coupled into the thermocouple circuit in place of the potentiometer when used.

The control system for the column includes automatic control of the pressure and manual operation of the other points of control by valves. These are represented schematically in Figure 4. A Honeywell Disk Chart pressure recorder-controller and an air-driven Masonneilan diaphragm control valve provided automatic control of the column pressure. The controller has a 0-25 psig range and is coupled to the column via the ethylene glycol seal pot system located at the top of the column. The manual points of control are: the reflux rate to the column, the steam rate to the reboiler and to the reflux preheater, and the water rate to the overhead condenser. The reflux rate and steam rate points were controlled with 1-inch blunt needle valves. For very fine adjustment of the reflux rate a 1/2-inch needle valve on the pump bypass line was used.

Control Panel

The instrumentation and control equipment is located in or adjacent to the control panel. The control panel is 8 ft. high, 8 ft. wide, and 2 ft. deep and provides space for most of the instrumentation and control equipment used with column operation. These include the flow recorders and the pressure recorder-controller; the thermocouple circuitry; the thermocouple switching panel; the multipoint temperature recorder; the solenoid control switches; and the pump controls. A portable cart is positioned in front of the panel with the potentiometer and the Bristol recorder.

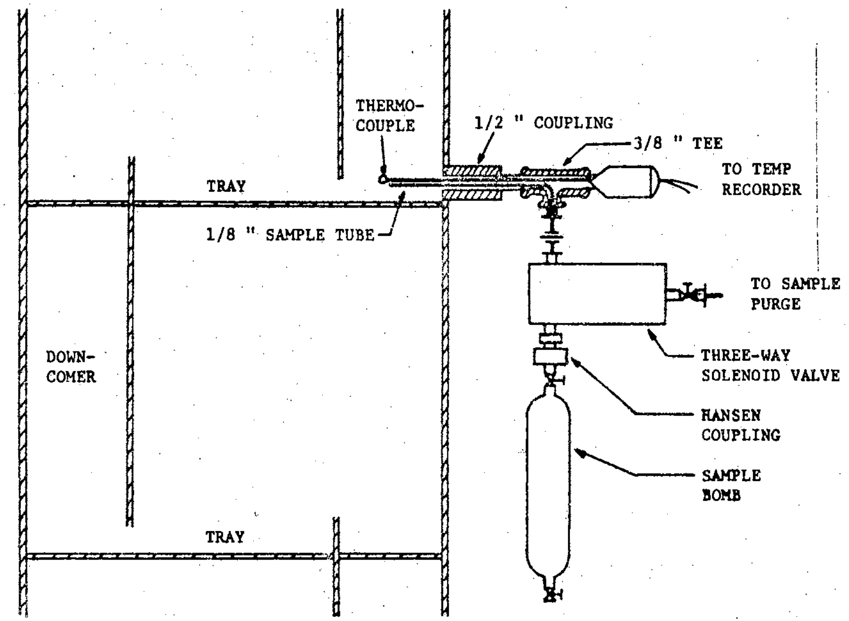
Sampling Systems

Samples for composition analysis are taken from both the vapor and liquid streams around specific trays. The sample points indicated in the diagram in Figure 4 are concerned with trays 2, 5, and 8. Also, for use in the description of the column operation, samples of the main streams around the column were taken.

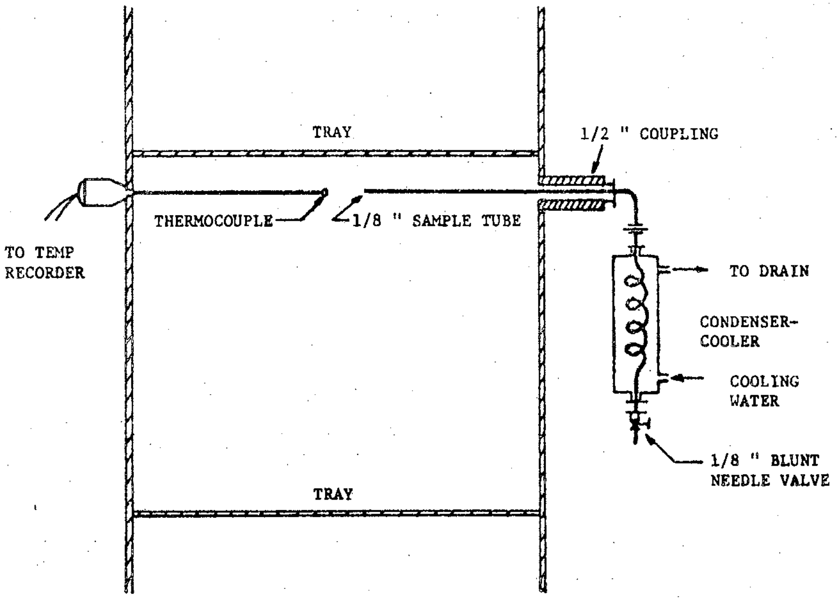
Two types of sample systems were employed. For external points around the column and for the liquid samples taken from the trays, a solenoid valve-sample bomb system was used. For the vapor samples removed from between the trays, a small condenser-cooler system was used with a collection vial. These systems are shown schematically in Figure 7. The liquid sample is taken from directly under the downcomer at the same point the thermocouple is employed. The sample of the vapor leaving a tray is taken directly from under the tray above and in the center of the column. A thermocouple is directly opposite the vapor sample inlet. These systems allow sampling of both vapor and liquid streams into and leaving the trays of interest without any noticeable upset of column operation.

The sampling tubes were constructed of 1/8-inch ID stainless steel tubing and Swagelok tubing fittings. The solenoid valve-sample bomb system used Asco number 8314A-75 explosion-proof solenoid valves, Hansen push-tight couplings, and an evacuated sample bomb. The sample bombs were cast aluminum with a 380 milliliters capacity. A bleed bypass line of 1/8-inch copper tubing and a needle valve for control were provided for constant purging of the sample line. The purge stream was introduced back into the column two trays below the sample point or into the reboiler. The solenoid valve controls are located on the control panel described previously. A switch controls the current to the solenoid valves and toggle switches are provided to place the individual valves on or off the sampling circuit.

The condenser-cooler sample system was designed to allow one to remove vapor samples from the column and collect them



(a) SOLENOID VALVE - SAMPLE BOMB SYSTEM



(b) CONDENSER - COOLER VAPOR SAMPLE SYSTEM

Figure 7. Detail of Sample Systems

as subcooled liquid. The small condenser-cooler was constructed from 1-inch diameter rigid copper tubing and caps and a coiled 1/8-inch copper tube. The sample passed through the tube and water was used as coolent on the shell side in countercurrent fashion. A 1/8-inch needle valve was used to control the sample rate. The system was designed for a very small sample holdup -- found to be less than 3 cubic centimeters.

Utilities

Utilities required for the operation include electricity, water, 50 psig air, and steam. The electricity, both 110 and 220 volt, was available at the installation. Ample cooling water was available from city sources. The pressured air was available at the installation. Two steam sources were employed. The installation site had 50 psig steam available. To supplement this, a 54 killowatt Model RHC54 Reimers Electric Steam Boiler was installed. This unit was capable of providing up to 184,000 BTU per hour of saturated steam over a pressure range up to 100 psig. The boiler was a package unit equipped with controls and condensate return system including receiver tank and pump.

Figure 8 is a schematic diagram of the steam manifold system and steam boiler. The available 50 psig steam, the boiler steam, or both can be used in any combination for operation of the reboiler and reflux preheater. A condensate collection vessel

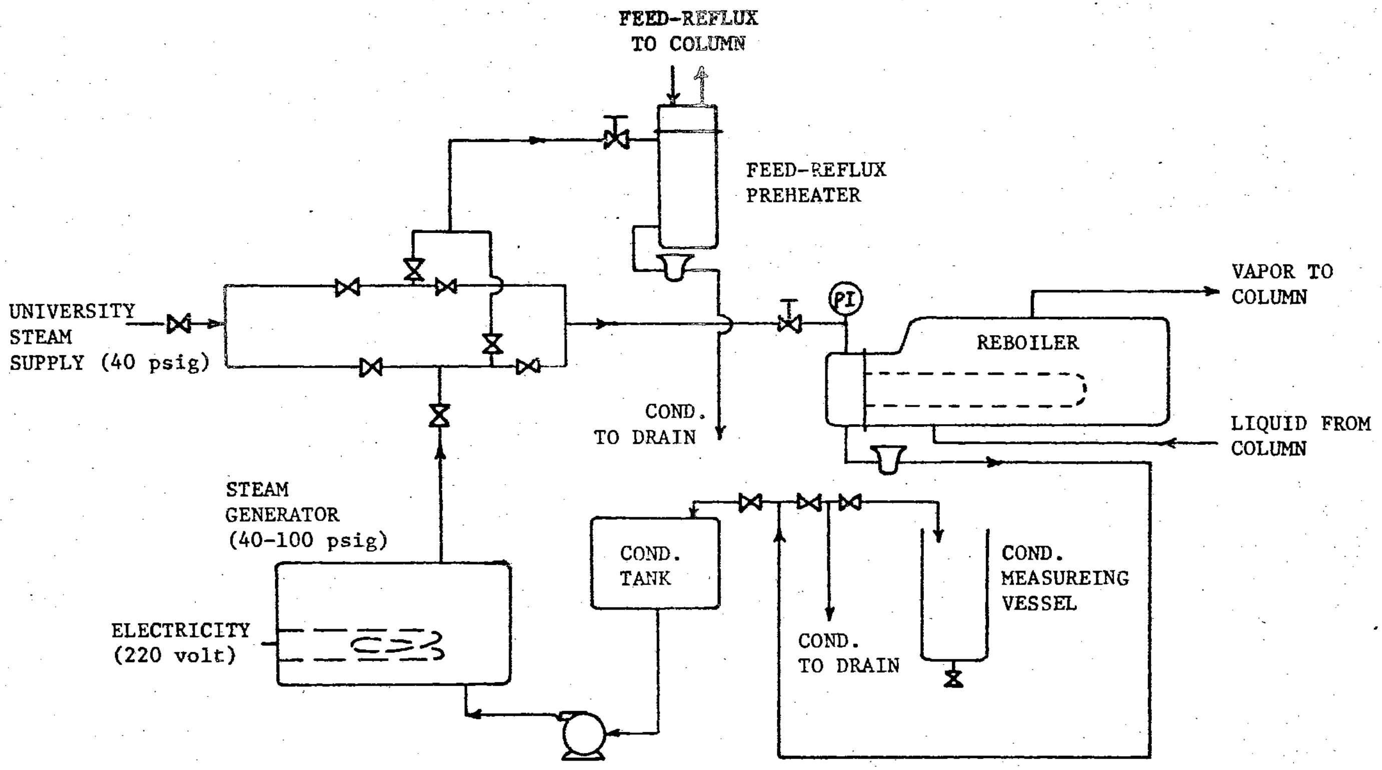


Figure 8. Schematic Steam Manifold System Diagram

is also indicated. This is a 5-foot length of 6-inch pipe with a calibrated volume of 3.165 gallons. With this vessel and a timer, the condensate rate from the reboiler can be measured for boil-up rate calculations and heat balance checks.

Gas Chromatograph

The samples from the experimental runs were analyzed on an F and M Model 500 Programmed High Temperature Gas Chromatograph incorporating a Perkin-Elmer Model D2 Electronic Integrator and a Honeywell-Brown Electronic Recorder. The calibration and discussion is given in Appendix D. The recorder was used only to monitor the analysis. The integrator operates on the principle of voltage to frequency conversion. Output voltage from the chromatograph serves as input to the integrator. The output frequency from the integrator is proportional to the input voltage and these output pulses are fed into a seven-digit decade counter. These counts are stored in the counter until they are read out and printed by a Kienzle Digital Printer.

CHAPTER V

EXPERIMENTAL PROCEDURE

The experimental procedures for this study consist of the following: the startup and operation of the distillation apparatus, the techniques for obtaining samples and measuring the temperatures and other variables, and the chromatograph operation for sample analysis.

Column Startup and Operation

The startup procedure consisted of first pumping sufficient material into the reboiler for operation. The material in the reboiler was then circulated via the reflux pump onto the top tray of the column in order to assure that the trays were wet. Cooling water was circulated to both the condenser and the bottom product cooler -- the latter being employed only during the startup pump around procedure. A bleed valve at the top of the structure between the condenser and the column was opened to allow non-condensable gases to escape. The pressure controller was set to a predetermined value -- 3 to 5 psig, and the steam to the reboiler was turned on. The multipoint temperature recorder was used to monitor the vapor temperatures in the column. When the vapor overhead temperature began to rise and condensable material began to flow through the non-condensable vent, the column was shut-in by closing this bleed valve. The pressure then began to build up in the column. This in turn activated the pressure control valve and distillate began to fill the distillate accumulator. When the level in the accumulator reached about 1-foot, the reflux pump was switched so as to pump from the accumulator and the reboiler was shut-in to total boil-up. The reflux rate was set at the minimum operable reflux rate (found to be about 40 per cent of maximum) to allow the column pressure to build up to that desired and to prevent pumping the accumulator dry. Steam was slowly introduced to the reflux preheater. Cooling water to the bottom product cooler was turned off. The vapor spaces in the top of the seal pots were bled to remove trapped noncondensables. The desired column pressure was usually obtained rapidly but temperatures and rates were unsteady. At this point the column was in a non-steady state, total reflux condition.

Column operation was adjusted to the desired conditions and the tower was allowed to line out to a steady state. The reflux rate was adjusted to the desired rate by adjusting the steam rate to the reboiler. The steam rate controls the amount of distillate produced overhead. By using the reflux control valve to maintain a constant level in the distillate accumulator, the reflux rate can be controlled to equal the rate of the vapor overhead. A minimum level (about 4-inches) was maintained in the accumulator to assure minimum holdup of reflux. The liquid level in the bottom of the column was maintained between the top of the vapor pipe and the top of the liquid line to the reboiler. These adjustments in quantity of material in the system as well as any composition adjustments were made by appropriately adding some material to the column with the reflux stream or bleeding material out of the reboiler or distillate accumulator. In either case the rates of withdrawal or addition were made very slowly so as to minimize any upset.

Approximately an hour from start-up the rates and temperatures began to approach constant values. Fine adjustments were made in the steam rate to the reboiler to reach the desired reflux rate, and the steam rate to the reflux preheater was gradually increased. The reflux temperature was adjusted until it approached, but was below its bubble point when introduced back into the column.

After approximately two hours of operation the reflux rate and the liquid and vapor rates to and from the reboiler indicated constant values. The column pressure and temperatures indicated constant values. The column was then allowed to operate at this steady condition an additional two to five hours before samples were taken.

Three points should be covered concerning problems in column operation. Two problems are very easy to initiate: superheating the reflux with the preheater so that it flashes on the top tray; and when operating at a minimum condition, falling below the minimum operable rate. Both of these problems lead to a downcomer loading problem. The liquid backs up in the downcomers and then dumps in cycles. Also, one or more dry trays were obtained while attempting runs. These were detected by negligible temperature changes in the vapor stream passing through the tray. All of these problems require cutting the steam and reinitiating the startup procedure.

Obtaining Experimental Data

During the two to five hour operation following line out, water was turned on to the cooler-condenser sample systems and flow through each of the sample bleed lines initiated at a very slow rate. The multipoint recorder was continuously used to monitor the temperatures and the flow rates, liquid levels, and column pressures were checked. Although not employed on each run, the Bristol recorder was used to study temperature fluctuations during operation. The recorder was used to measure fluctuations in EMF for individual thermocouples.

The potentiometer was placed in the thermocouple circuit and measurements of each temperature point was started. A measurement of the rate of steam condensate from the reboiler was taken. After two or more measurements of each temperature point had been made, the reflux rate and the top and bottom column pressure were recorded, and the sampling procedure was started. While continuing to measure the temperatures, samples were taken in the following order: vapor samples 8 and 1 were collected in vials; liquid samples 2, 3, 5, 6, 8, and 9 and the vapor overhead, the reflux, and the liquid to and vapor from the reboiler were sampled simultaneously with the solenoid system; vapor samples 8, 7, 5, 4, 2, and 1 were collected in vials; and again the liquid samples 2, 3, 5, 6, 8, and 9 and the vapor overhead, the reflux, and the liquid to and vapor from the reboiler were sampled simultaneously with the solenoid system. In taking the vapor samples from the points employing the condenser-cooler system, each sample point was flushed until a volume of five to ten cubic centimeters had been removed. Then the sample was collected and capped in the half dram vials. To take the remaining samples with sample bombs, the evacuated bombs were coupled to the solenoid valve, the bomb valves opened, the switch tripped, the bomb valves closed, and the sample bombs removed. The reflux rate and the top and bottom column pressures were recorded, and another measurement of the steam condensate rate from the reboiler was taken. The column was then shut down.

The samples collected in vials were stored in a freezer at 10 degrees F. until analyzed. The samples in the bombs were packed in ice for a 30 minute period and air was bled into the bombs. Essentially complete condensation occurred and the samples were transferred as liquid to 1 dram vials and capped. These, too, were stored in the freezer until analyzed.

Chromatograph Operation

The chromatograph and the integrator were both turned on and the chromatograph was allowed to reach thermal steady state. About four or five hours were required before a drift-free, steady operation was obtained. Both the recorder and the balance needle on the integrator were used to determine when the chromatograph was at equilibrium. The samples were removed from the freezer as required and placed in an ice water bath. A two microliter portion was injected into the chromatograph and the results recorded by the digital printer. Each sample was analyzed three or four times. The output from the printer was recorded as frequency counts which were proportional to the amount of each component analyzed. Details of the chromatograph calibration and weight and mole fraction calculation from the chromatograph output are given in Appendix D.

CHAPTER VI

PRESENTATION OF RESULTS

Experimental data were obtained for the total reflux distillation of the benzene-toluene-para-xylene system. A series of 15 experimental runs were made at just above atmospheric pressure. Four "compositions" were employed and for each column "composition", three or four runs were made, each at a different reflux rate. Vapor and liquid samples and temperature measurements were taken from around tray numbers 2, 5, and 8 in the column.

The column operating conditions, the experimental data, and the calculated intermediate and final results are detailed for each run in Appendix E. The results are presented in the remainder of this chapter. A summary of the column operating conditions and the composition range involved for each run is given in Table I. The experimental tray conditions and the calculated results are presented in Tables II and III. These results include both the Murphree efficiencies and the generalized tray efficiencies.

TABLE 1

SUMMARY OF COLUMN OPERATING CONDITIONS

Run No.	Internal Liquid Rate moles/hr	Top Pressure psia	Temperature Range - °F		Column Composition Range - Mole Percent					
					Reboiler			Vapor OH		
			Reboiler	Vapor OH	Benz.	Tol	P-xyl	Benz.	Tol	P-xyl
303	11.01	35.3	256.7	211.4	-	.816	.184	.618	.382	-
304	6.73	35.4	254.4	215.1	-	.823	.177	.567	.433	-
305	8.64	35.1	254.7	197.8	.004	.814	.183	.835	.165	-
306	8.04	36.6	257.2	201.7	.003	.810	.187	.810	.190	-
307	12.26	34.3	253.7	190.9	.007	.822	.170	.904	.096	-
308	10.42	34.9	254.3	193.3	.005	.819	.176	.869	.131	-
309	10.32	35.1	255.4	215.0	-	.814	.186	.518	.482	-
310	8.58	34.7	254.6	214.4	-	.811	.189	.522	.478	-
311	11.42	33.4	249.8	183.2	.028	.814	.157	.966	.034	-
312	7.76	36.0	253.9	189.5	.012	.809	.178	.946	.055	-
313	11.25	35.9	253.5	187.4	.021	.792	.187	.965	.035	-
314	8.95	34.8	251.8	185.9	.026	.794	.180	.970	.030	-
315	10.30	34.5	266.9	220.1	-	.605	.395	.379	.621	-
316	8.18	35.4	268.3	221.0	-	.597	.403	.412	.588	-
317	7.78	36.7	269.7	220.9	-	.597	.403	.476	.524	-

TABLE II

SUMMARY OF EXPERIMENTAL TRAY CONDITIONS

	Rates		Temperatures				Compositions					
Run & Tray No.	L_n & V_{n-1}	L_{n+1} & V_n	t_n	t_{n+1}	T_n	T_{n-1}	X_n, Benz	X_n, Tol	X_n, P-Xyl	X_{n+1} Benz	X_{n+1} Tol	X_{n+1} P-Xyl
303-1, 2	10.85	10.92	246.2	244.4	246.2	248.5	-	.888	.112	.004	.922	.074
5	10.98	10.98	240.4	238.0	239.5	241.4	.020	.954	.026	.042	.943	.015
8	10.98	11.01	229.3	221.7	228.0	233.7	.148	.847	.005	.253	.747	-
304-1, 2	6.62	6.65	247.0	245.1	247.0	249.0	-	.878	.122	.003	.911	.086
5	6.70	6.72	241.4	240.0	240.4	242.2	.013	.959	.028	.025	.959	.016
8	6.72	6.73	233.8	227.2	231.2	235.6	.091	.903	.006	.178	.822	-
305-1, 2	8.36	8.40	244.8	242.7	244.9	247.3	.008	.885	.108	.014	.915	.070
5	8.45	8.46	236.8	232.0	235.2	239.0	.062	.914	.024	.117	.869	.014
8	8.54	8.64	217.5	207.4	214.0	222.5	.324	.670	.005	.496	.504	-
306-1, 2	7.80	7.84	247.7	245.6	247.7	250.2	.006	.887	.107	.012	.917	.071
5	7.88	7.90	240.1	236.0	238.6	242.1	.050	.926	.024	.094	.892	.014
8	7.95	8.04	222.6	212.9	218.8	227.5	.278	.717	.005	.446	.554	-
307-1, 2	11.72	11.77	241.6	239.2	242.3	244.7	.017	.889	.094	.032	.906	.062
5	11.82	11.90	229.2	222.5	228.4	234.0	.130	.850	.021	.240	.749	.011
8	12.13	12.26	204.0	197.2	203.3	211.8	.532	.468	-	.644	.347	.009
308-1, 2	10.00	10.03	243.8	241.2	243.8	246.5	.013	.887	.100	.025	.910	.065
5	10.09	10.13	233.3	226.9	231.5	226.9	.099	.879	.022	.185	.803	.012
8	10.28	10.42	209.7	200.8	207.6	216.5	.452	.544	.004	.616	.384	-
309-1, 2	10.19	10.24	245.2	243.3	245.5	247.9	-	.888	.112	.004	.923	.073
5	10.30	10.33	240.0	238.4	239.5	241.4	.015	.959	.026	.031	.954	.015
8	10.31	10.32	231.2	224.7	230.8	234.9	.109	.886	.006	.195	.805	-
310-1, 2	8.45	8.49	244.7	242.8	244.9	247.2	-	.888	.112	.004	.920	.076
5	8.55	8.56	239.6	237.8	238.8	240.6	.014	.959	.027	.029	.956	.015
8	8.57	8.58	231.8	225.3	229.7	234.0	.098	.897	.006	.187	.813	-
311-1, 2	10.59	10.63	235.4	229.9	235.8	240.4	.071	.847	.082	.129	.821	.052
5	10.83	10.99	212.2	202.8	209.9	219.9	.387	.599	.014	.549	.444	.007
8	11.34	11.42	189.6	186.1	188.8	193.8	.821	.179	-	.872	.128	-
312-1, 2	7.31	7.34	243.8	240.2	243.2	246.8	.031	.876	.093	.056	.882	.062
5	7.40	7.45	227.1	218.6	224.7	232.5	.207	.775	.018	.334	.657	.010
8	7.65	7.76	201.0	194.0	198.8	206.2	.633	.367	-	.778	.222	-
313-1, 2	10.44	10.47	240.1	234.7	240.6	234.7	.065	.851	.084	.116	.830	.054
5	10.60	10.78	216.6	207.5	215.1	225.2	.354	.631	.015	.521	.471	.008
8	11.12	11.25	193.9	190.2	193.5	198.7	.790	.210	-	.875	.125	-
314-1, 2	8.27	8.30	238.4	232.5	238.0	243.1	.069	.845	.087	.122	.823	.055
5	8.44	8.57	214.4	204.4	212.2	222.0	.372	.613	.014	.549	.444	.007
8	8.86	8.95	192.3	188.6	191.2	196.1	.806	.194	-	.889	.111	-
315-1, 2	9.95	10.06	250.5	247.3	251.2	255.7	-	.736	.264	.003	.809	.188
5	10.21	10.28	241.0	239.4	240.5	243.0	.139	.918	.068	.027	.931	.041
8	10.33	10.30	233.8	228.6	232.6	235.0	.089	.895	.016	.147	.843	.009
316-1, 2	8.73	8.82	252.4	249.1	253.0	257.6	-	.733	.267	-	.808	.192
5	8.95	8.99	242.8	240.7	242.3	244.7	.009	.916	.075	.018	.937	.045
8	9.02	8.18	235.4	230.2	234.3	237.4	.067	.917	.016	.130	.961	.009
318-1, 2	7.54	7.58	255.4	251.1	255.0	259.9	-	.733	.027	-	.799	.201
5	7.71	7.75	245.2	243.1	244.1	247.1	.010	.914	.076	.020	.934	.045
8	7.78	7.78	237.4	231.8	235.4	239.3	.074	.910	.016	.147	.844	.009

TABLE III

SUMMARY OF CALCULATED FRACTIONAL TRAY EFFICIENCIES

	BENZENE			TOLUENE			PARA-XYLENE			
Run & Tray No.	$E_{MV_{n,i}}$	$E_{ML_{n,i}}$	$E_{n,i}$	E_{MV}	E_{ML}	E_{Si}	E_{MV}	E_{ML}	E_{Si}	E_{H_n}
303-1, 2	-	-	-	.588	.470	.883	.665	.512	.736	.286
5	.879	.940	.973	.990	1.795	.468	.790	.635	.832	.378
8	.736	.828	.904	.716	.786	.782	-	-	-	.611
304-1, 2	-	-	-	.536	.407	.790	.585	.434	.670	.308
5	.687	.831	.891	.139	-.053	-.236	.758	.596	.808	.223
8	.882	.936	.975	.854	.876	.816	-	-	-	.630
305-1, 2	.668	.818	.879	.671	.478	.928	.670	.519	.743	.318
5	.764	.866	.922	.768	.982	.746	.751	.578	.803	.544
8	.809	.839	.934	.799	.814	.849	-	-	-	677
306-1, 2	.637	.797	.864	.696	.459	.935	.645	.492	.730	.288
5	.741	.854	.916	.739	.999	.658	.746	.575	.796	.445
8	.836	.872	.956	.825	.847	.850	-	-	-	.629
307-1, 2	.643	.798	.868	.646	.363	1.151	.644	.485	.717	.295
5	.841	.900	.957	.848	.948	.838	.778	.594	.818	.558
8	.551	.502	.735	.593	.600	.702	-	-	-	.446
308-1, 2	.664	.813	.876	.666	.427	.976	.665	.510	.738	.384
5	.799	.879	.938	.805	.945	.804	.755	.572	.803	.575
8	.757	.754	.893	.748	.733	.820	-	-	-	.641
309-1, 2	-	-	-	.610	.489	1.158	.676	.525	.733	.172
5	.821	.909	.958	.960	-28.076	.132	.767	.606	.807	.170
8	.760	.855	.921	.732	.800	.761	-	-	-	556
310-1, 2	-	-	-	.557	.429	.865	.618	.463	.696	.272
5	.873	.938	.971	1.094	-3.281	.118	.826	.689	.860	.276
8	.855	.919	.964	.828	.862	.810	-	-	-	616
311-1, 2	.653	.785	.863	.645	3.047	.704	.655	.484	.728	.525
5	.733	.744	.876	.734	.768	.821	.703	.437	.750	.665
8	.519	.367	.682	.519	.367	.610	-	-	-	.453
312-1, 2	.598	.760	.840	.726	.189	1.799	.621	.460	.698	.392
5	.713	.788	.891	.713	.820	.781	.717	.506	.767	.561
8	.819	.760	.955	.819	.760	.857	-	-	-	536
313-1, 2	.639	.776	.857	.642	17.722	.660	.637	.468	.711	.501
5	.772	.790	.919	.775	.819	.827	.705	.447	.747	.539
8	.750	.624	.912	.750	.624	.806	-	-	-	.410
314-1, 2	.636	.773	.860	.628	9.543	.584	.641	.473	.710	.461
5	.807	.816	.936	.810	.843	.853	.738	.479	.776	.599
8	.777	.650	.915	.777	.650	.829	-	-	-	.495
315-1, 2	-	-	-	.600	.511	.804	.627	.527	.711	.297
5	.715	.847	.904	.752	.438	1.482	.733	.577	.789	.194
8	.597	.741	.821	.587	.785	.745	.699	.512	.768	.669
316-1, 2	-	-	-	.610	.513	.801	.610	.513	.699	.314
5	.794	.894	.942	.745	.535	1.488	.760	.614	.808	.262
8	.811	.833	.800	.791	.942	.757	.757	.581	.806	.547
318-1, 2	-	-	-	.538	.442	.731	.538	.442	.639	.420
5	.818	.907	.952	.741	.533	1.563	.766	.623	.812	.241
8	.882	.934	.976	.897	1.001	.804	.761	.580	.806	550

CHAPTER VII

DISCUSSION

In this chapter analyses and discussions are given for: the column operation and degree of approach to steady state obtained; the temperature measurements recorded; the vapor and liquid samples obtained; the chromatographic analysis of these samples; and the calculation of the results. A comparison and evaluation of the Murphree and the generalized tray efficiencies is made. Finally, a discussion of probable error is given.

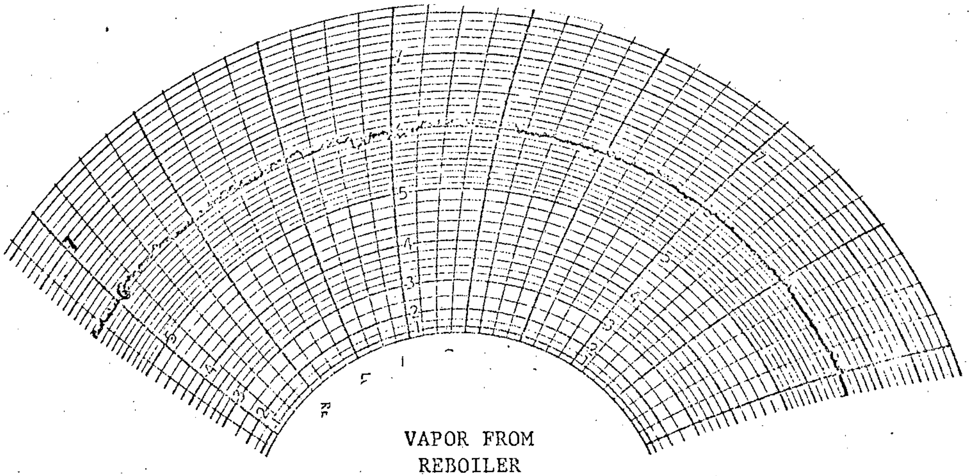
Column Operation

Precise column operation was desirable to assure as complete approach to steady state as possible. The objective during the experimental run was to obtain and maintain a constant column pressure, constant flow rates, and a constant temperature profile. After the line-out period, column pressure was held constant within one- to two-tenths inch of mercury. Flow rates were constant when time-smoothed. The reflux rate was constant within approximately two per cent based on variations about the mean reading. Some of this fluctuation was due to pump vibration, but the major variation was a function of, and could be predicted from, column pressure behavior. The chart recorders employed on the lines for reboiler streams fluctuated consistently with the steam trap dumps. However, constant average readings were maintained.

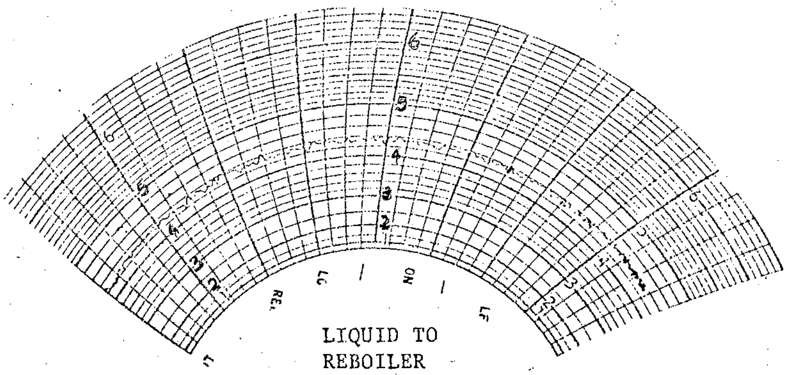
Figure 9 shows a chart recording of the monitored vapor and liquid rates around the reboiler. Figure 10 gives a typical temperature and composition profile. The two sets of liquid samples indicated were taken about 15 minutes apart. These show that the column operation was essentially a steady state one. The data in Appendix E give the measured temperatures and both sets of sample compositions.

Heat balance calculations were also made to evaluate steady column operation. The measured and calculated reboiler duties are compared and summarized in Table IV. The calculated duty was equivalent to the pseudo-condenser duty which was determined by measurement of the vapor overhead temperature and the liquid reflux temperature and rate. The measured duty was determined from the measured steam condensate rate from the reboiler. The equipment used for measured duty determination was added after runs 303 and 304 were made. Except for runs 311 through 313, the heat balance was very good. There is no explanation for the poor heat balance check on these three runs. All other parameters indicated acceptable column operation.

The most consistent problem encountered was change in steam



VAPOR FROM
REBOILER



LIQUID TO
REBOILER

Figure 9. Disk Chart Recorder Traces of Monitored Flow Rates To and From Reboiler

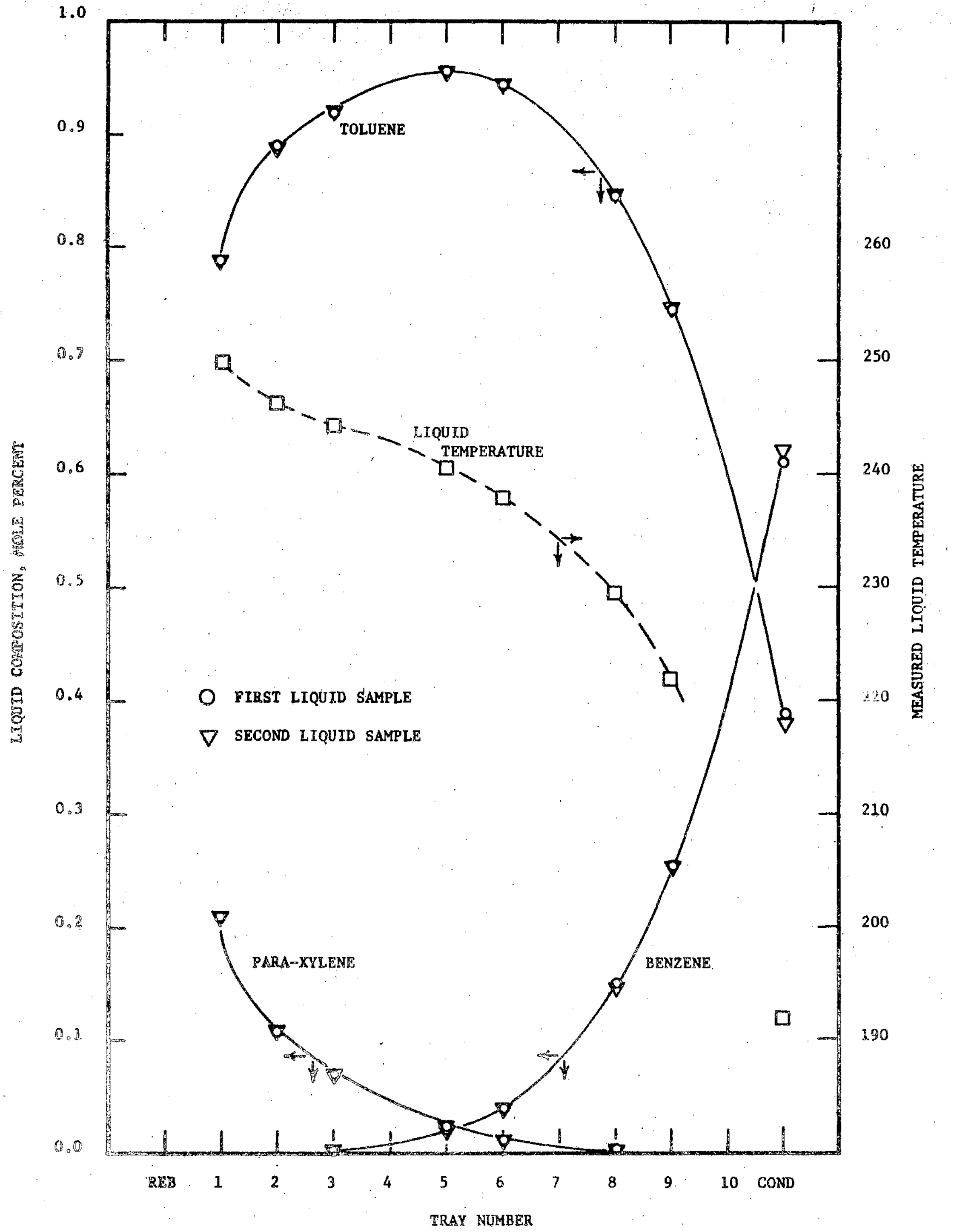


Figure 10. Temperature and Composition Profile

TABLE IV

REBOILER HEAT BALANCES

Run No.	Steam Pressure, psig	Measured Duty, BTU/Hr	Calculated Duty, BTU/Hr	Error, %
303	-	-	155700	-
304	-	-	95100	-
305	40.5	120500	120000	-0.4
306	49.0	116500	111700	-4.1
307	47.0	171500	168000	-1.7
308	49.0	143000	143500	+0.4
309	48.0	144000	146500	+1.7
310	44.0	125000	121500	-2.8
311	44.0	183000	152600	-16.6
312	41.5	140000	104700	-25.3
313	46.5	138000	149800	+8.6
314	42.5	120000	118900	-0.9
315	57.5	143500	146200	+1.9
316	56.0	128000	127900	-0.1
318	55.5	110200	110000	-0.1

pressure available at the reboiler and reflux heater. This occurred when the University-supplied steam was employed alone or in conjunction with generated steam. The column pressure and the reflux rate changes were generally initiated by these steam pressure changes. Steam pressure changes as large as five psi were experienced during some runs.

Temperature Measurements

Accurate temperature measurements were desired. The vapor and liquid temperatures were used in heat and material balance calculations to determine internal column rates and also for calculation of the generalized equilibrium tray state. As discussed in Appendix B the thermocouples were calibrated and found accurate to within 0.4 degree F.

Temperature measurements of liquid streams were consistent and accurate. With respect to the measured compositions of the corresponding liquid samples, the measured temperatures were slightly below the calculated bubble point temperatures. This would be expected for a less than ideal distillation tray operation.

Vapor temperature measurements were anomalously below the calculated dew point of the vapor stream. The tables of data in Appendix E show the measured vapor temperature behavior and the liquid temperature measurements compared with their respective calculated dew and bubble point temperatures.

Difficulty in measuring vapor temperatures in distillation apparatus has been experienced by several investigators. In particular, temperature measurements below the calculated dew point of a vapor stream have been reported in Chapter 3. Two possible factors are readily apparent.

1. Tray weeping could splash the thermocouple with liquid from the tray above. The temperature of this liquid would normally be lower than the vapor stream.
2. Condensation of the vapor on the tip due to heat loss by conduction could lower the measured temperature.

A laboratory study was made concerning vapor temperature measurements and is detailed in Appendix G. The results of this study are that in a total-refluxed still, the measured vapor and liquid phase temperatures are equal when pure benzene is used and unequal when a benzene-toluene mixture is employed. The measured vapor temperature for the mixture was about 4.7°C. lower than the liquid phase temperature. Condensation was observed on the thermocouple. Fractional condensing would give a saturated vapor with a lower temperature.

Fluctuations were encountered for both vapor and liquid temperature measurements. These are illustrated in Figure 11 which shows recording EMF traces of several of the thermocouples made with the Bristol recorder. The fluctuation span is equivalent

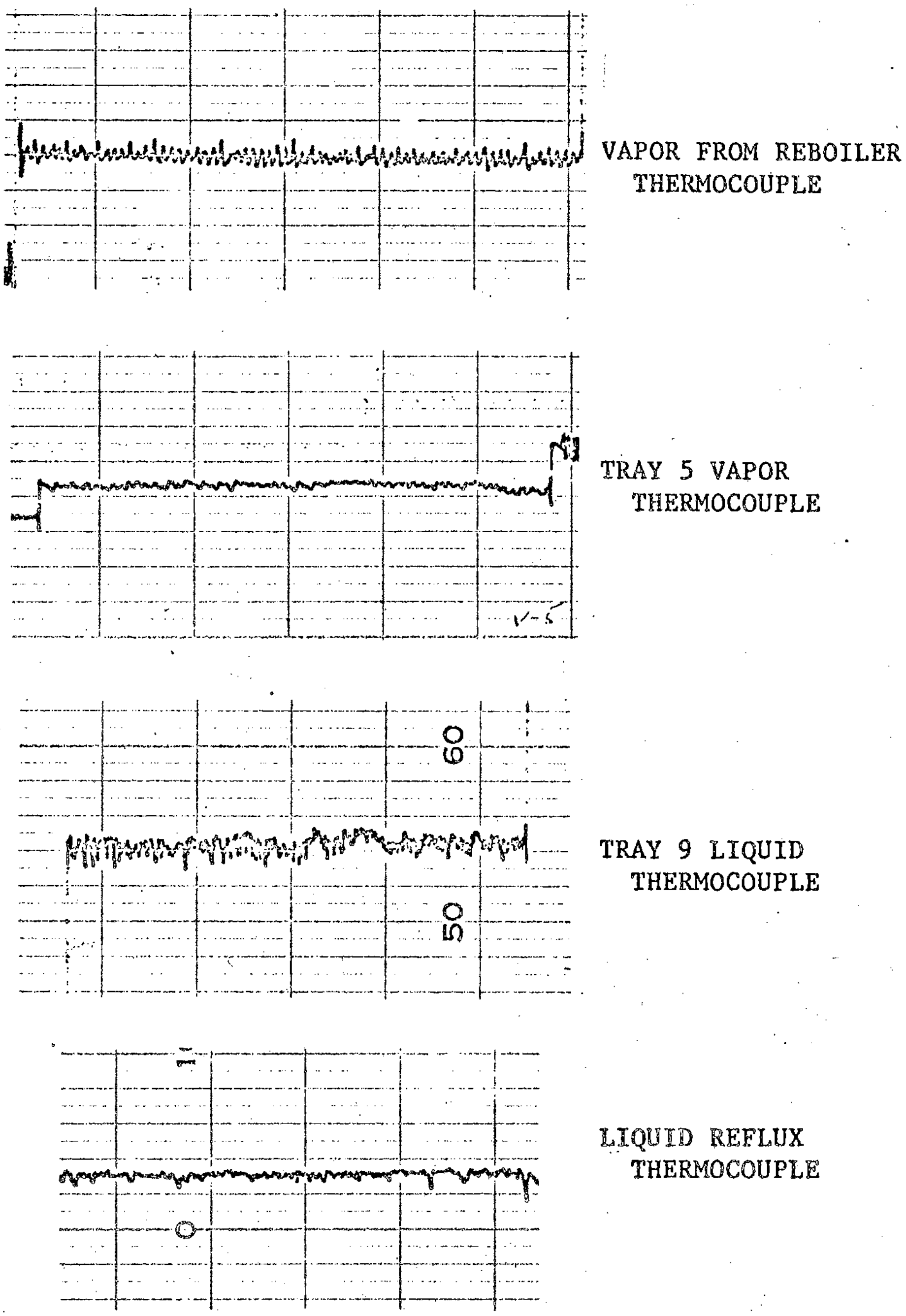


Figure 11. Strip Chart Recorder Traces of EMF Outputs from Thermocouples Monitored During Run 309

to less than one degree F. Similar fluctuation was observed using the total-refluxed still with a benzene-toluene mixture. These fluctuations could be caused by condensation and dripping and by liquid splashing at the bottom of the downcomer.

Samples

The most important phase of an experimental run was collecting representative samples. Two sets of the liquid samples were collected over a fifteen minute period to verify steady column operation. Some vapor samples were collected to check the liquid sample results.

The liquid sample systems and the solenoid devices were reliable and satisfactory samples were obtained. Laboratory sampling of liquid aromatics with this sample bomb apparatus was demonstrated to give representative and reliable results and is discussed in Appendix H. Error introduced by this sampling procedure is less than the expected analytical error.

The vapor sample compositions were consistent but not reliable. They were not compatible with the compositions of the corresponding liquid samples. In every case more of the lower boiling material (benzene or benzene and toluene) was present in the vapor sample. Variations between the liquid samples and corresponding vapor samples are shown in the data in Appendix E.

Possible causes for poor vapor samples include entrainment, foaming, weeping, and condensation. Entrainment or foaming problems would produce vapor sample deviations in the opposite direction - more higher boiling material. Also, entrainment and foaming would not be suspect with the properties of this system and the low flow rates employed. Weeping of a lower boiling mixture onto the sample probe could explain these results. If liquid from the tray above showered onto the sample probe and was swept into the tube, poor samples would result. Fractional condensation of the vapor could result in a vapor sample with more lower boiling material. Heat loss thru the column wall, the sample probe, and the thermocouple would be heat sinks for condensation.

For the majority of runs, the samples of the vapor overhead and the reboiler vapor exhibited the same problem - a higher concentration of lower boiler material than the corresponding liquid reflux and liquid to the reboiler samples. For these four samples, a solenoid valve-sample bomb apparatus was employed. This would indicate no fault in the design and/or operation of the condenser-cooler vapor sampling system.

Chromatograph Analyses

The chromatograph calibration and operation have been detailed in Appendix D and Chapter V respectively. Consistent and reliable analyses were obtained within the accuracy of the instrument.

The sample compositions given in the data in Appendix E are average results of three or four analyses of each sample. For each analysis the mass and mole fractions were calculated and normalized from the chromatograph count fraction. Then the average values of the count, mass, and mole fractions were calculated and normalized. A computer program with the calibration regression fits described in Appendix D was used for these calculations. Table V gives an example of the results for a typical sample.

An evaluation of the accuracy of the instrument was made during calibration. Table XI shows a standard deviation of 0.0005 count fraction or less. The maximum and minimum count fraction deviation was determined and found to be .001 count fraction or less. Some error is also encountered in the regression analyses of the calibration data. The expected standard deviation was estimated for the regression fits and found to be about 0.001 count fraction or less.

The sample composition data given in Table II and Appendix E shows concentrations for some components changing from a finite value to zero across a tray. The zero concentrations shown are not necessarily correct but are the result of a limitation of the digital integrator coupled to the chromatograph detector. The chromatograph detected the low component concentrations but did not trip the digital integrator. Table XI shows that standard sample J contained 0.0014 wt fraction benzene but did not register through

TABLE V

TYPICAL CHROMATOGRAPH RESULTS

SAMPLE NUMBER 1 OF TRAY NUMBER 6
LIQUID FOR RUN 304

	AREA FRACT	MASS FRACT	MOLE FRACT
BZ	0.01931	0.02108	0.02483
TOL	0.96671	0.96009	0.95885
XYL	0.01398	0.01883	0.01632
BZ	0.01916	0.02094	0.02466
TOL	0.96700	0.96037	0.95914
XYL	0.01384	0.01869	0.01620
BZ	0.01948	0.02124	0.02502
TOL	0.96635	0.95974	0.95849
XYL	0.01417	0.01902	0.01649
BZ	0.01952	0.02129	0.02507
TOL	0.96659	0.95997	0.95868
XYL	0.01389	0.01874	0.01624

STREAM AVERAGE

	AREA FRACT	MASS FRACT	MOLE FRACT
BZ	0.01937	0.02114	0.02490
TOL	0.96666	0.96004	0.95879
XYL	0.01397	0.01882	0.01631

the integrator. Mole fractions of 0.005 could be detected but the calibration at this concentration is questionable.

Calculation of Results

The physical properties used for the benzene-toluene-para-xylene system are discussed in Appendix D. The calculations involved heat and material balances, dew point and bubble point calculations, and the flash calculation procedure for the generalized equilibrium state as discussed in Chapter II. The assumption of no heat loss from the equipment was made. The convergence limitations set for dew point and bubble point calculations were well within the analytical accuracy of the experimental data. The convergence limits for the enthalpy balance on the generalized equilibrium state flash calculation was 0.01 per cent. This, too, was well within the accuracy of the experimental and physical property data. These convergence limits were "tight" enough to allow valid evaluations of the generalized equilibrium state.

Results were calculated for each run with the vapor compositions set equal to the corresponding liquid composition. Since two sets of liquid samples were obtained, the calculations were made for each sample set independently. These are presented in Appendix E.

The calculated efficiencies are not reported in Table II for the situation where a component concentration changes from a finite value to zero across a tray. However, the efficiencies are reported as calculated in Appendix E.

Comparison and Evaluation of Results

Table III gives the Murphree vapor, the Murphree liquid and generalized component efficiencies for each component. The generalized enthalpy efficiencies are also presented. The intermediate and final results are detailed for each run in Appendix E.

The Murphree and generalized component efficiency values were generally as expected. The values were mostly less than unity, indicating the tray performance at less than 100 per cent. However, several of the calculated toluene efficiencies were evaluated at or near the maximum in the toluene composition profile. This was illustrated in Figure 10. This is one of the composition regions where the Murphree efficiency and the generalized component efficiency values can be unbounded as discussed in Chapter II. The calculated values of these particular toluene efficiencies are summarized in Table VI. The measured liquid composition data and the calculated equilibrium data are also reported. In this unbounded region the Murphree vapor toluene efficiencies varied from 0.14 to 1.1, the Murphree liquid toluene efficiencies varied from -28.1 to 17.7, and the generalized toluene efficiencies varied from -0.24 to 1.8. Because of extremely small composition changes, part of the reason for these values can be

TABLE VI

SUMMARY OF TOLUENE EFFICIENCIES CALCULATED ABOUT THE COMPOSITION PROFILE MAXIMUM

			Murphree Equil.		Generalized Equilibrium		Efficiencies		
Run and Tray No.	$x_n = y_{n-1}$	$x_{n+1} = y_n$	Bub. Pt. y_n^*	Dew Pt. x_n^*	y_n^*	x_n^*	E_{MV_n}	E_{ML_n}	E_n
303-1, 5	.954	.943	.943	.949	.944	.953	.990	1.795	.468
304-1, 5	.959	.959	.957	.954	.960	.958	.139	-.053	-.236
307-1, 2	.889	.906	.916	.858	.915	.880	.646	.363	1.151
309-1, 2	.888	.923	.945	.852	.937	.876	.610	.489	1.158
309-1, 5	.959	.954	.954	.954	.955	.958	.960	-28.076	.132
310-1, 5	.959	.956	.956	.955	.957	.958	1.094	-3.281	.118
311-1, 2	.846	.821	.807	.879	.821	.846	.645	3.047	.704
312-1, 2	.876	.882	.885	.847	.890	.869	.726	.189	1.799
313-1, 2	.851	.830	.818	.831	.832	.849	.642	17.722	.660
314-1, 2	.845	.823	.810	.825	.824	.844	.628	9.543	.584
315-1, 5	.918	.931	.936	.901	.936	.913	.752	.438	1.482
316-1, 5	.916	.937	.944	.898	.942	.911	.745	.535	1.488
318-1, 5	.915	.934	.942	.897	.940	.910	.741	.533	1.563

attributed to analytical limitations. However this phenomenon is real and these values are believed to approximate the calculated toluene efficiencies for these conditions. The corresponding component efficiencies for benzene and para-xylene are given in Appendix E. These values are less than unity and are representative of the actual tray performance.

The value of the Murphree efficiencies is uniquely a function of the measured compositions. While these compositions depend on many tray variables, only the compositions determine the calculated efficiency. Non-constant molal flow rates and variations in the measured temperatures will not affect their calculated values.

The generalized component efficiencies concern the change in total quantities of a component across a tray. Heat and material balance calculations are involved in the generalized equilibrium state calculation. As a result, thermal effects are considered in the component efficiencies. This is illustrated by example in Table VII. Here, Run 303-1 is calculated using both the measured vapor temperature and the corresponding dew point temperature for the calculations. The dew point temperatures were higher and the effects on the generalized equilibrium state and resulting efficiencies are shown. The temperature differences were 3 to 4 degrees F. The change in calculated values of the generalized efficiencies ranged from negligible to almost 50 per cent.

TABLE VII

THERMAL EFFECT ON GENERALIZED EFFICIENCY CALCULATIONS

RUN 303-1

		TRAY 2	TRAY 5	TRAY 8
MEASURED VAPOR TEMP				
T_N		246.24	239.49	227.96
T_{N-1}		248.49	241.37	233.68
Equil Vap - V_N^*		10.80	10.84	11.85
T_N^*		248.17	242.30	231.67
$y_{i,N}^*$	B	.003	.044	.268
	T	.936	.944	.731
	X	.061	.012	.001
Equil Liq - L_N^*		10.96	11.12	11.14
t_N^*		248.17	242.30	231.67
$x_{i,N}^*$	B	.001	.019	.134
	T	.875	.953	.862
	X	.124	.028	.004
Efficiency - $E_{i,N}$	B	1.415	.973	.904
	T	.883	.486	.782
	X	.736	.832	1.370
$E_{H,N}$		.286	.380	.610
DEW POINT TEMP USED FOR VAPOR TEMP				
T_N		249.04	242.56	232.19
T_{N-1}		252.06	244.49	237.25
Equil Vap - V_N^*		10.84	10.89	10.90
T_N^*		248.18	242.35	231.70
$y_{i,N}^*$	B	.003	.044	.267
	T	.936	.944	.732
	X	.061	.013	.001
Equil Liq - L_N^*		10.84	11.01	11.00
t_N^*		248.18	242.35	231.70
$x_{i,N}^*$	B	.001	.019	.134
	T	.875	.953	.862
	X	.124	.028	.004
Efficiency - $E_{i,N}$	B	1.409	.965	.891
	T	.783	.704	.833
	X	.743	.840	1.376
$E_{H,N}$		.321	.490	.721

The effect of non-constant molal flow can also affect the values obtained for the generalized component efficiency. This is shown by the last three efficiencies listed in Table VI (Runs 315-1, 5, 316-1, 5 and 318-1, 5). Toluene efficiencies greater than unity were calculated because of the different total molal rates around the tray.

No relationship between the two efficiencies was apparent. Even with the assumption of constant molal flow rate, a relationship does not exist. The equilibrium state definitions for the two efficiencies could not be related in any way.

The effect of column loading on the component efficiencies was examined. Figures 12, 13, and 14 show a comparison of the Murphree liquid, and the generalized component efficiencies for benzene, toluene, and para-xylene respectively. These are shown as a function of the molar vapor rate. (The toluene points in Table VI are excluded.) The molar vapor rates ranged from about 6.5 to 12 moles per hour. The operating range was almost twice the minimum rate. Higher rates were not obtainable because of reboiler steam limitations.

The most general indicator of column loading is the F-factor. This is based on the superficial vapor velocity for the free cross-sectional area (ft/sec) times the square root of the vapor density. The operating range for this study corresponds to an F-factor range of 0.35 to 0.60.

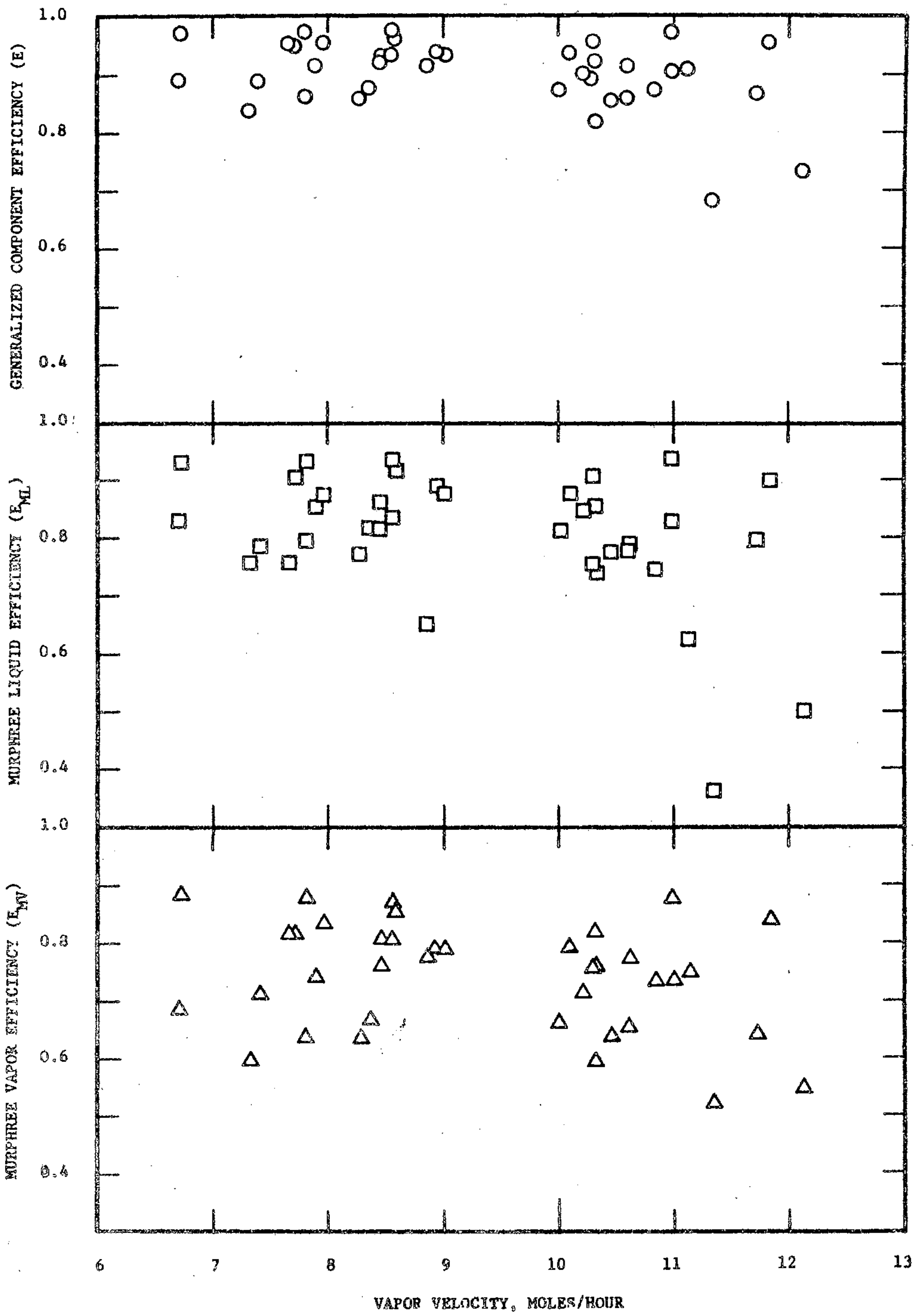


Figure 12. Vapor Rate Effects on Benzene Efficiencies

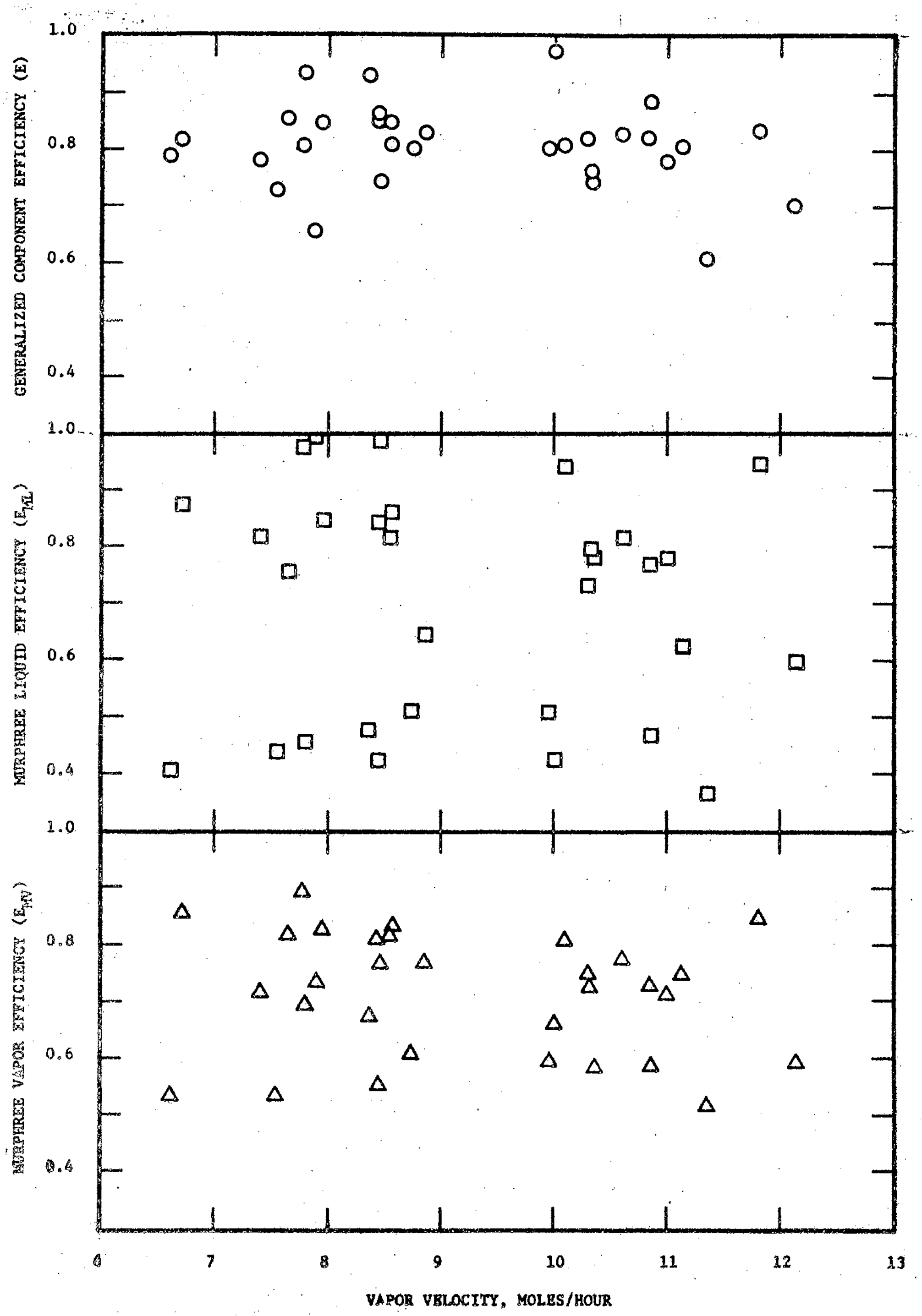


Figure 13. Vapor Rate Effects on Toluene Efficiencies

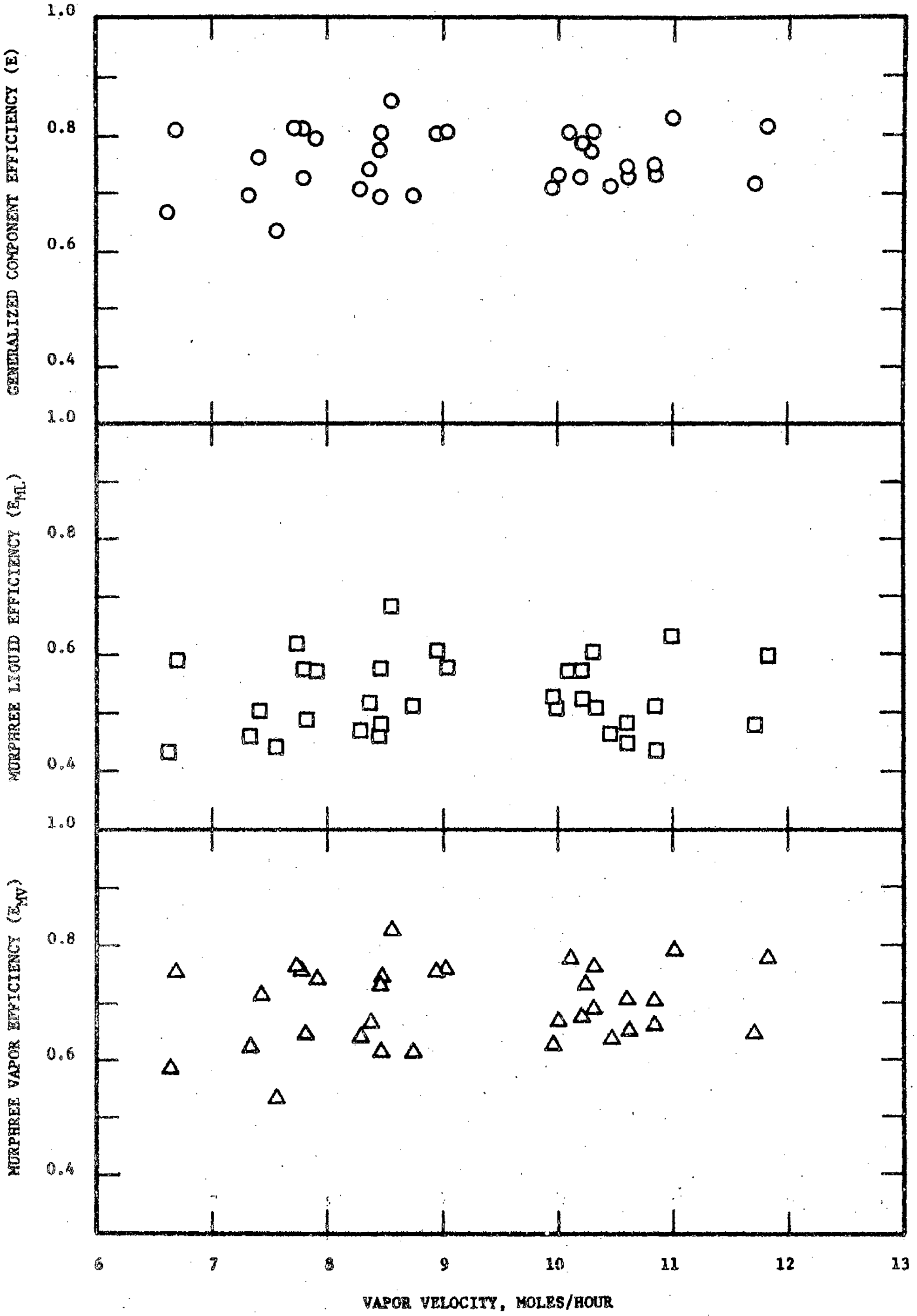


Figure 14. Vapor Rate Effects on Para-Xylene Efficiencies

For benzene, the Murphree vapor efficiencies ranged from about 60 to 90 per cent, most of the Murphree liquid efficiencies ranged from 70 to 95 per cent, and the generalized component efficiencies were in the 80 to 100 per cent range. The toluene efficiencies had a slightly lower range. These Murphree vapor efficiencies ranged from about 50 to 90 per cent, the Murphree liquid efficiencies from 40 to 100 per cent, and the generalized component efficiencies from about 70 to 90 per cent. For para-xylene, the Murphree vapor efficiencies ranged from about 60 to 80 per cent, the Murphree liquid efficiencies from 40 to 70 per cent, and the generalized component efficiencies from 70 to 85 per cent.

The plots exhibit some scatter, particularly for the Murphree efficiencies. The vapor velocity does not give any skewing effects. In fact, there appears to be no trend other than fairly consistent performance over the range of column loading studied. Consistent tray performance over broad operating ranges is characteristic of valve tray columns. To see if the generalized component efficiency behavior is similar to that of the Murphree efficiencies, the operating range should be extended to higher vapor rates. The Murphree efficiencies should drop significantly when high entrainment and incipient flooding occur.

The effect of component composition on the efficiencies was examined. This is shown in Figures 15, 16, and 17 for benzene,

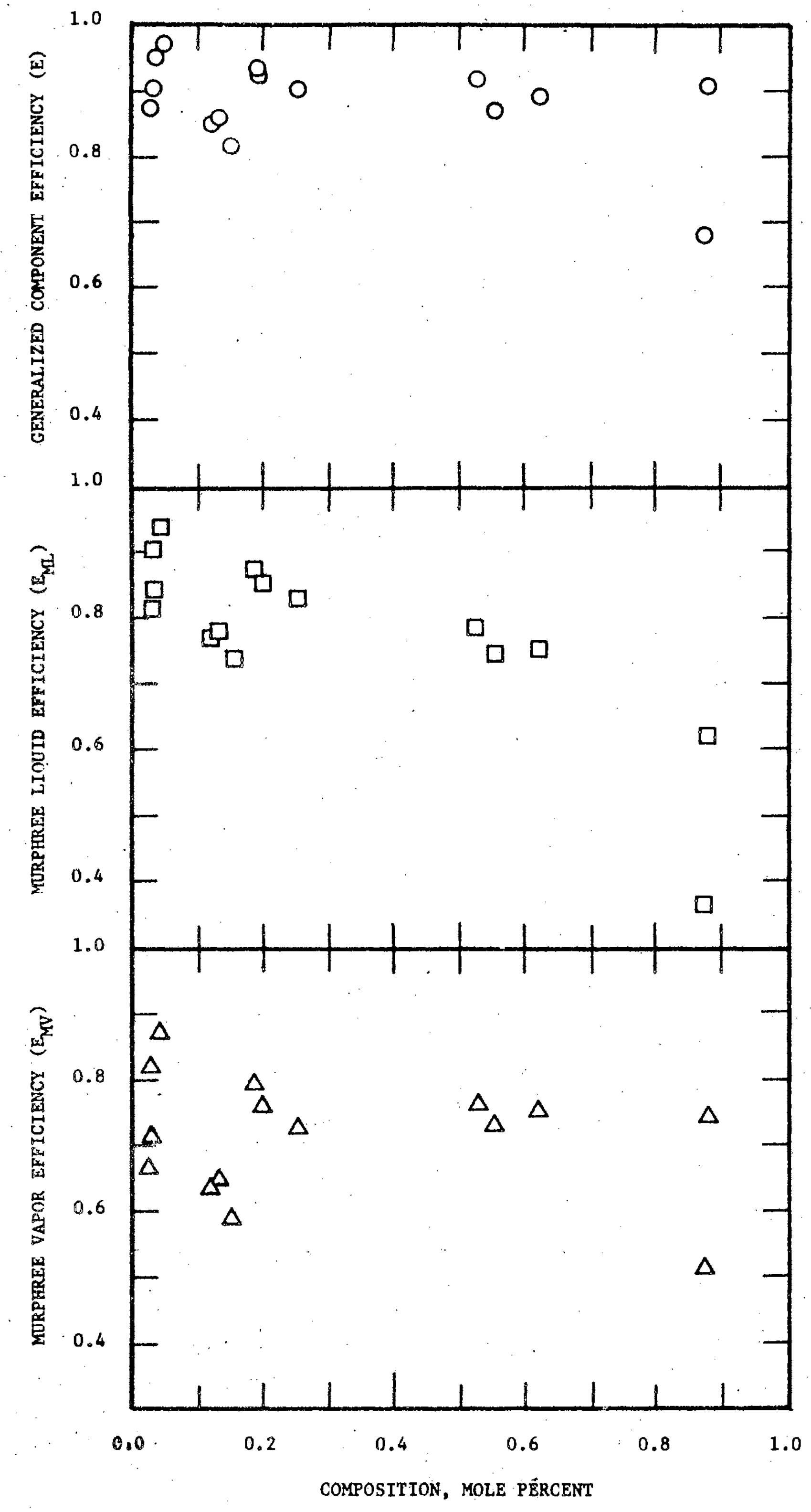


Figure 15. Composition Effects on Benzene Efficiencies

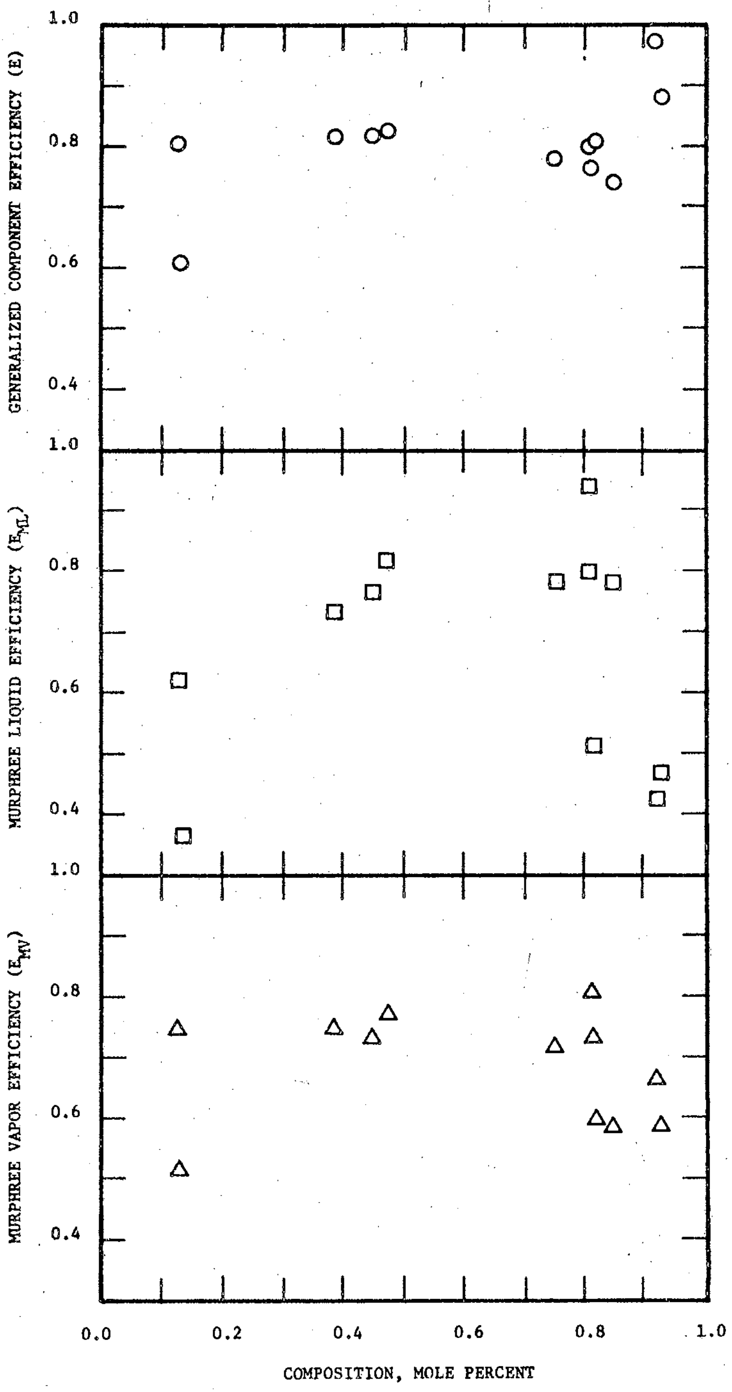


Figure 16. Composition Effects on Toluene Efficiencies

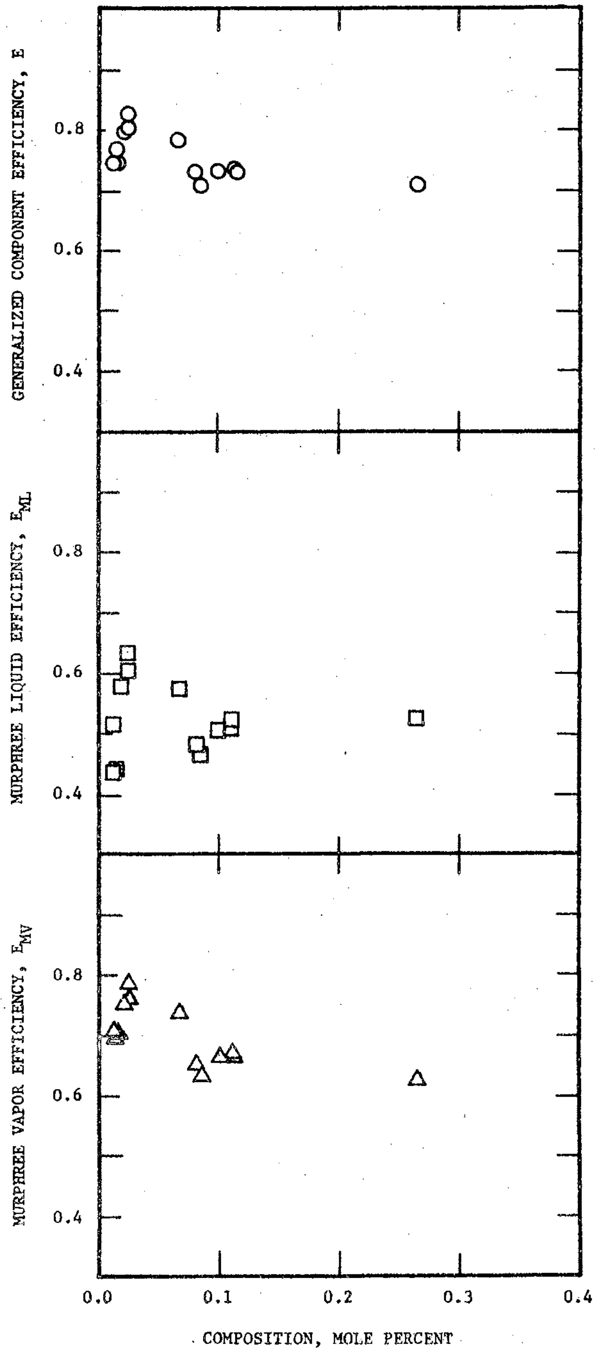


Figure 17. Composition Effects on Para-Xylene Efficiencies

toluene, and para-xylene respectively. These efficiencies are plotted from runs where the column loading was essentially constant - a narrow range of 10 to 11 moles per hour. (Appropriate toluene points in Table 6 were excluded.)

Some composition effect is noticeable for benzene efficiencies. No peak composition effects were present and a wide composition range occurred. Trends show a "tailing-off" of the efficiencies at low and high concentrations. The trends were similar for the Murphree and generalized values. The toluene efficiencies are too scattered to discern any trends. The xylene composition range is too narrow for conclusions.

Analytical limitations play an important part in efficiency calculation at extremely low compositions. This is illustrated for the benzene and xylene efficiencies calculated from composition values of 2 to 4 per cent. Ten to twenty per cent variation in these efficiencies occurred for essentially constant composition and column loading.

The generalized enthalpy efficiency is a measure of the degree of approach to perfect heat transfer between the entering streams on a tray. The enthalpy efficiency values, as given in Table III, range from about 0.15 to 0.70. Figure 18 shows the effect of the tray temperature on these efficiencies. The tray temperatures ranged from 190 to 255 degrees F. No meaningful trend is apparent for the effect of absolute temperature measurements. Figure 19

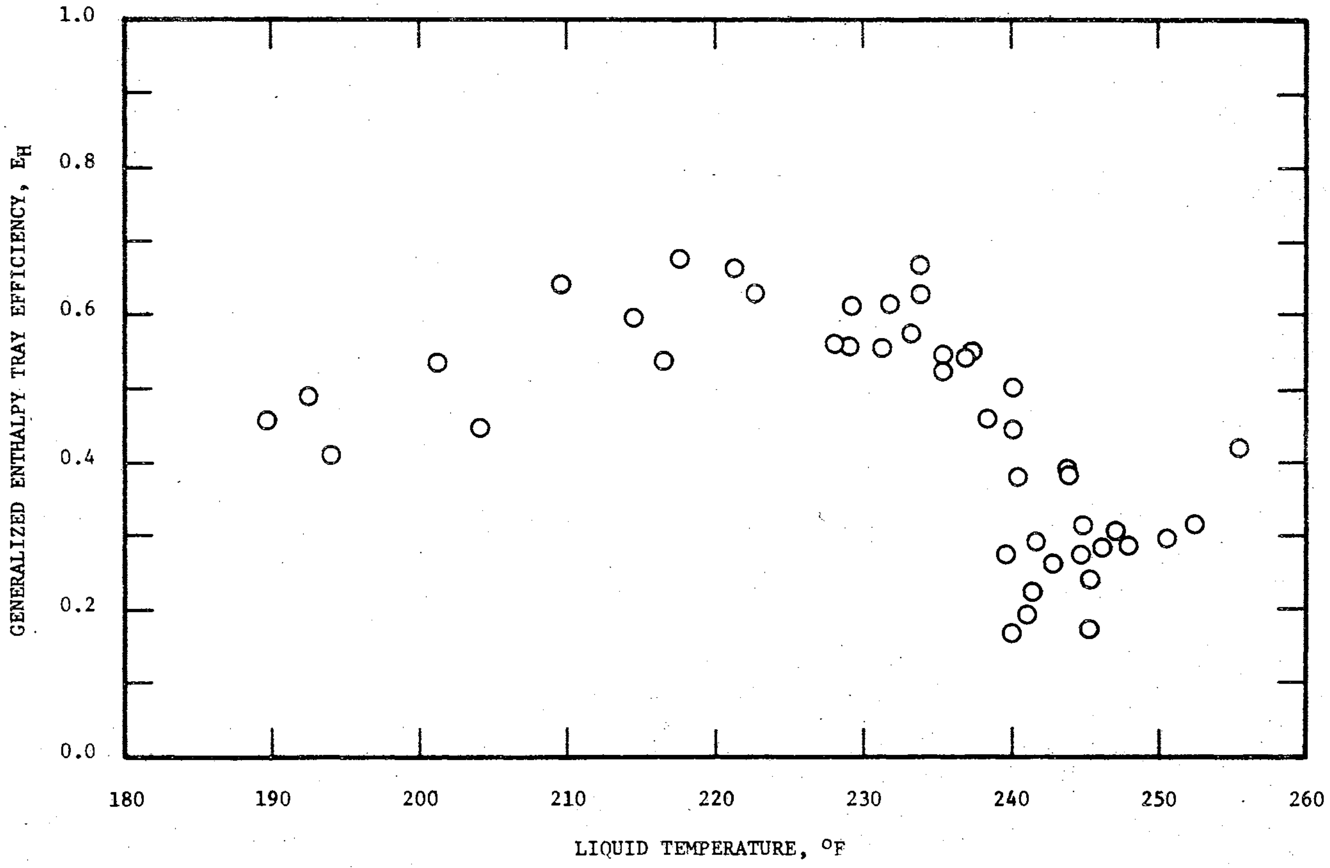


Figure 18. Effect of Tray Temperature on Generalized Enthalpy Efficiency

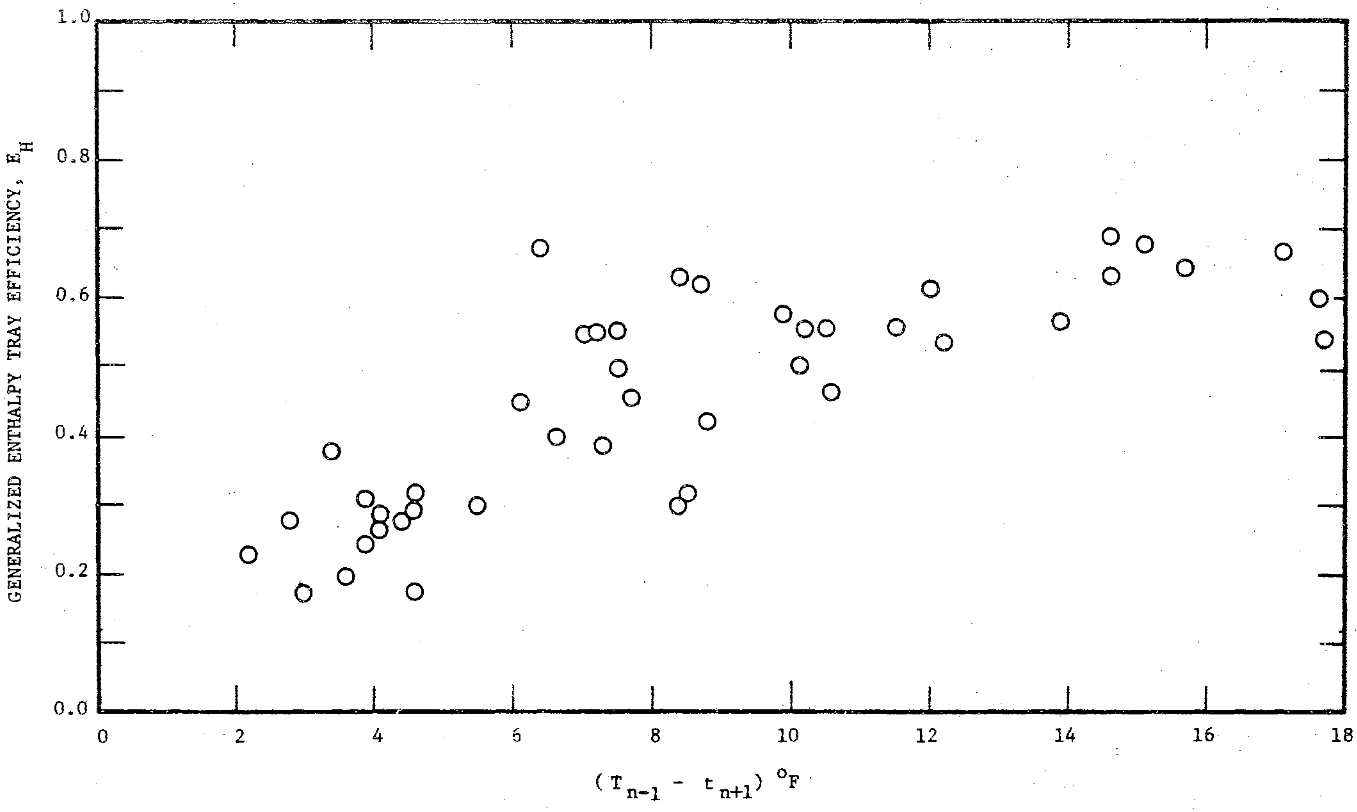


Figure 19. Effect of Temperature Driving Force on Generalized Enthalpy Efficiency

shows the effect of temperature difference of the entering streams on the enthalpy efficiencies. The temperature difference or driving force ranged from 2 to 18 degrees F. The enthalpy efficiency values increased for increasing temperature difference. The data indicates an increasing trend up to about 0.70 and then an apparent leveling off.

Possible factors affecting the enthalpy efficiency include heat loss and inaccurate temperature measurements. The heat loss thru the column shell, Q_n in equations (15) and (20), was assumed zero. This assumption was verified by heat balance for most runs. A sizeable column heat loss would give lower enthalpy efficiency values. Temperature measurements can affect the efficiency values as was shown in Table VII. An increase in the value of the vapor temperature gives higher efficiencies because the equilibrium state has been changed and the temperature difference increased.

Probable Error

Sources of error for the calculated efficiencies are obvious. These are: error associated with the physical property data; unsteady column operation; inaccurate temperature measurements; bad samples; and chromatograph analytical error.

Error in the calculated ideal equilibrium distribution coefficients would appear in all the calculated component efficiency values. No estimate was made of this effect. Error in the enthalpy data would therefore appear as error in the generalized component efficiencies and the generalized enthalpy efficiency. No estimate was made of this error.

The column operation, the temperature measurements, and the sampling procedure have been discussed previously. The column operation was steady and held essentially constant for several hours before the data was taken. Heat balance checks were good for most of the runs. Consistent temperature measurements were obtained. The effect of the questionable vapor temperature measurements on the calculated efficiencies has been illustrated in Table VII and in the previous discussion. The questionable vapor samples were not used in calculating the results - the corresponding liquid samples were employed. Bad liquid samples would, of course, give efficiency errors. However, the liquid samples were found to be reliable and essentially constant with time.

The effect of analytical error on the calculated efficiency values is as follows. The chromatograph calibration analyses and regressions (Appendix D) gave an estimated standard deviation of 0.001 mole fraction or less. The absolute analytical error in composition is 0.1 per cent. The relative analytical error for a component with low concentration is higher - if component concentration is 5.0 per cent the relative error is 0.050 ± 0.001 or ± 2.0 per cent. An analysis of the effect of analytical error is detailed in Appendix F. Because the efficiencies are defined as ratios of composition differences, the error is most significant when the difference or change in component composition across a tray is small. Where the largest composition changes occur, the calculated efficiency should be accurate within one per cent. For smaller composition changes the calculated efficiency values may be ± ten per cent. At extremely low concentrations (as occurred for benzene and xylene) or for high toluene concentrations on the composition profile maximum, the calculated efficiencies can be in error by 30 per cent.

CHAPTER VIII

CONCLUSIONS AND RECOMMENDATIONS

The objectives of this work were to obtain experimental data from an operating distillation column employing a ternary system, and to calculate and evaluate the tray performance with these data. These objectives were met. A series of 15 experimental runs were made. Tray temperatures were measured and tray samples were collected. The samples were analyzed by gas chromatography, Murphree efficiencies and the generalized component efficiencies were calculated.

Conclusions drawn from this study are:

1. The generalized equilibrium state and the generalized efficiencies introduced by Standart can be evaluated from experimental data.
2. The generalized component efficiency values are similar to and behave very much like the Murphree efficiencies.
3. The Murphree efficiencies and the generalized component efficiencies can be unbounded functions where maximums (or minimums) in component composition profiles exist. The toluene efficiencies in this study exhibited

such behavior.

4. Non-constant molal flow and temperature measurements were shown to affect the generalized efficiency values but not the Murphree efficiencies.
5. The generalized enthalpy efficiency can be correlated as a function of the measured temperature differences of the entering vapor and liquid stream to a tray.
6. Over the operating range of this study, no column loading effects were detected. No conclusions on the effect of composition on component efficiencies were made. At composition extremes, the effect of analytical error on the calculated component efficiency values is significant.
7. The experimental equipment performs well and good data was obtained. Minor modifications are desirable to increase the operating range and to reduce the degree of manual operation required.
8. The vapor temperature measurements and the vapor samples were not representative of the expected tray behavior. Condensation and/or tray weeping were suspect.

For future studies the following recommendations are made:

1. Questions on the validity of the vapor temperature measurements and the vapor samples should be

resolved. A laboratory study with a single distillation tray, should be made. Vapor temperature measurements and vapor samples of entering and leaving streams should be studied.

2. The experimental equipment should be modified as follows: Additional, constant pressure steam service should be made available to increase the column operating range and to operate at higher column pressure. A precise liquid-level controller setting an automatic reflux control valve should be installed on the reflux accumulator. Additional sample points should be added to sample all the liquid streams. A differential manometer or transducer should be employed for column pressure drop measurements.
3. Additional experimental efficiency data should be obtained with the modified equipment. This should include operation over wider ranges of column loading and operation with other systems - both binary and multicomponent.
4. A general tray-by-tray distillation calculation procedure should be developed incorporating the generalized efficiencies.

NOMENCLATURE

Major Symbols

A	Component reference, Antoine coefficient, or enthalpy regression coefficient.
a	Chromatograph calibration regression coefficient.
B	Component reference, Antoine coefficient or enthalpy regression coefficient.
b	Chromatograph calibration regression coefficient.
C	Component reference, Antoine coefficient, or enthalpy regression coefficient.
c	Chromatograph calibration regression coefficient.
E_n	Generalized overall material efficiency defined by Eq. (18).
$E_{n,i}$	Generalized component efficiency defined by Eq. (19).
E_{H_n}	Generalized enthalpy efficiency defined by Eq. (20).
$E^o_{n,i}$	Modified vaporization efficiency defined by Eq. (11).
E_{HT_n}	Modified heat transfer efficiency defined by Eq. (12).
$E_{GL_{n,i}}$	Haunsen efficiency defined by Eq. (8).
$E_{ML_{n,i}}$	Murphree liquid-phase tray efficiency defined by Eq. (2).
$E_{MV_{n,i}}$	Murphree vapor-phase tray efficiency defined by Eq. (1).
$E^M_{MV_{n,i}}$	Modified Murphree vapor tray efficiency defined by Eq. (9).

E_{TL_n}	Liquid temperature efficiency defined by Eq. (6).
E_{TV_n}	Vapor temperature efficiency defined by Eq. (5).
H	Molar enthalpy of saturated vapor.
h	Molar enthalpy of saturated liquid.
K_i	Equilibrium distribution coefficient.
k	Slope of the vapor-liquid equilibrium curve.
L	Molar liquid flow rate.
P	System pressure.
P^o	Component vapor pressure.
Q_n	Rate of heat loss from tray n.
r_n	Fraction of Q_n lost by vapor phase.
T	Vapor-phase temperature.
t	Liquid-phase temperature.
V	Molar liquid flow rate.
w	Rotameter fluid flow rate, lbs/hr.
$x_{n,i}$	Liquid composition, mole fraction.
$Y_{n,i}$	Vapor composition defined by Eq. (10).
$y_{n,i}$	Vapor composition, mole fraction.

Greek Symbols

ρ	Density.
σ	Standard deviation.
μ	Chemical potential.

Subscripts

i	Component i.
L	Liquid phase.
n	Tray n.
V	Vapor phase.

Superscript

$*$	Denotes equilibrium value.

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APPENDIX A

PHYSICAL PROPERTIES

PHYSICAL PROPERTIES

Equilibrium Distribution Coefficients

Ideal vapor-liquid equilibrium distribution coefficients, K_i, were calculated from the equation

$$K_i = \frac{P_i^o}{P} \qquad (22)$$

The component vapor pressures, P_i^o, were calculated from the Antoine equation

$$\log_{10} P_i = A - \frac{B}{C + T} \qquad (23)$$

Vapor pressure is psia and T is in degrees F. The coefficients A, B, and C are experimentally determined coefficients from API Research Project 44 (37) and given below in Table VIII.

TABLE VIII

ANTOINE VAPOR PRESSURE CONSTANTS

	A	B	C
Benzene	5.19204	2179.859	365.422
Toluene	5.24103	2420.640	363.068
P-xylene	5.27691	2616.174	355.553

From the measured column pressure drop, an average drop per tray was determined. An individual tray pressure was employed in the distribution coefficient calculation rather than assuming an average column pressure.

Calculations were made to determine the deviation from ideality. Both liquid activity coefficients and an imperfection pressure correction were evaluated and applied to the equilibrium distribution coefficients. The ideal K-values deviated by less than one per cent and were used in the calculations for this study.

Enthalpies

The ideal vapor enthalpy data used in this study are from the API Research Project 44 (37, 41). The liquid enthalpies were calculated by subtracting the heat of vaporization from the vapor enthalpy at a given temperature. The heats of vaporization were given in the API Technical Data Book (41).

Both the vapor and liquid enthalpies were curve-fitted as a function of absolute temperature by linear regression. The resulting enthalpy equation is of the form

$$\text{Enthalpy} = A + BT + CT^2 \tag{24}$$

Enthalpy is BTU/lb mole and T is °Rankin. The coefficients are given in the Table following.

TABLE IX

ENTHALPY REGRESSION COEFFICIENTS

	A	B	C
Bz Vapor	14873.040	394.23407	160.13672
Tol Vapor	16182.117	577.34830	192.96094
Xyl Vapor	17541.495	743.12951	226.71094
Bz Liquid	-3436.2775	593.89251	255.41016
Tol Liquid	-4006.1295	732.39456	294.19922
Xyl Liquid	-5179.7625	1151.4322	303.00782

The enthalpy data is reported (41) to be in error by less than two per cent.

APPENDIX B

THERMOCOUPLE CALIBRATIONS

THERMOCOUPLE CALIBRATIONS

Twenty-one thermocouples were employed in the experimental equipment. These were copper-constantan, bare-wire thermocouples installed in a 1/8-inch diameter stainless steel sheath. The thermocouples were purchased from the Conax Company, Buffalo, New York.

These thermocouples were guaranteed within the desired accuracy of ± 1/2 degree F. However, the standard copper constantan calibration was checked over the temperature range encountered in the experimental work.

The atmospheric boiling point temperatures of absolute ethanol, deionized water, and 99.98 per cent octane were used. These corresponded to 173, 212, and 258 degrees F. respectively.

A total refluxed still was used for the calibration measurements. This was assembled from a double-necked, round-bottom flask, a water-cooled condenser and an electric heating mantel and powerstat. The thermocouple was placed in the vapor phase of the still with the tip about an inch above the boiling liquid. The calibration potentiometer was the same used in the experimental work -- a Leeds and Northrup model 8686 millivolt potentiometer.

After each thermocouple was placed in the still, five minutes were allowed to reach thermal steady state. The millivolt reading was recorded. The barometric pressure was also recorded. The boiling point temperatures of the calibration liquids were calculated at the measured barometric pressure.

Table X gives the results of the calibration tests. As indicated, the calibration checked within about 0.4 degree of the standard copper-constantan millivolt conversion tables.

TABLE X

THERMOCOUPLE CALIBRATION TESTS

Thermocouple Phase & Tray No.	Ethanol 740.6 mmHg 172.2 °F 3.260 mv	H_2O 739.5 mmHg 210.6 °F 4.243 mv	Octane 743.5 mmHg 256.8 °F 5.463 mv
L-1	3.251 mv	4.246 mv	5.459 mv
L-2	3.245	4.238	5.453
L-3	3.245	4.240	5.452
L-5	3.244	4.238	5.451
L-6	3.244	4.238	5.451
L-8	3.244	4.238	5.452
L-9	3.245	4.238	5.452
L-Reflux	3.244	4.239	5.450
V-Reboiler	3.245	4.239	5.451
V-1	3.243	4.238	5.451
V-2	3.251	4.246	5.459
V-3	3.244	4.239	5.451
V-4	3.243	4.241	5.452
V-5	3.243	4.239	5.451
V-6	3.251	4.246	5.459
V-7	3.245	4.241	5.450
V-8	3.244	4.239	5.451
V-9	3.251	4.246	5.459
V-OH	3.249	4.246	5.459
Average Deviation	0.3°F	0.1°F	0.4°F

APPENDIX C

ROTAMETER CALIBRATION

ROTAMETER CALIBRATION

A Fisher-Porter rotameter was employed to measure the reflux rate. This rotameter was calibrated prior to making the experimental runs. Calibration was performed by collecting and weighing samples at recorded rotameter readings and timed intervals. The temperature and composition of the calibration fluid was determined. From these data the actual flow rate for the calibration temperature and composition was calculated and a plot of this flow rate versus rotameter reading in per cent of maximum flow was constructed. The calibration is presented in Figure 20

Changes in fluid temperature and/or composition can change the actual flow rate from that indicated because either or both of these factors will change the fluid density. The ratio of flow rates for the actual to the calibrated rate at a constant rotameter reading is given in the following equation.

$$\frac{w_A}{w_C} = \sqrt{\frac{(\rho_f - \rho_A)\,\rho_A}{(\rho_f - \rho_C)\,\rho_C}} \qquad (25)$$

where ρ_f - rotameter float density (stainless steel - sp gr = 8.04)

ρ_A - fluid density at actual conditions

ρ_C - fluid density at calibration conditions

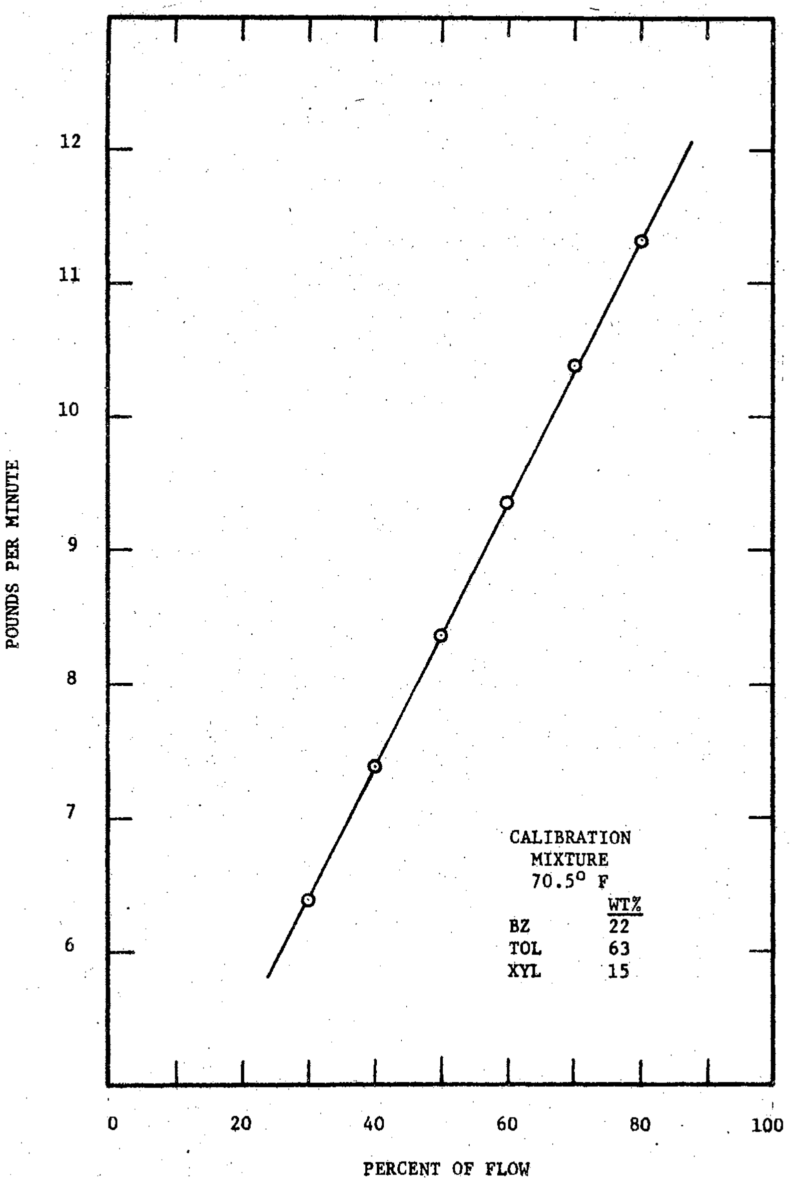

Figure 20. Rotameter Calibration Curve

w_A - flow rate of fluid at actual conditions

w_C - flow rate of fluid at calibration conditions

This can be simplified by cancelling the float minus fluid density ratio which will be essentially unity and rearranging to

$$w_A = w_C \sqrt{\frac{\rho_A}{\rho_C}} \tag{26}$$

In this manner the rotameter calibration can be applied to a reading of a fluid of known composition and temperature. And the calibration flow rate can be corrected to account for density changes.

APPENDIX D

CHROMATOGRAPH CALIBRATION

CHROMATOGRAPH CALIBRATION

The samples from the experimental runs were analyzed on an F & M model 500 Programmed High-Temperature Gas Chromatograph incorporating a Perkin-Elmer Model D2 Electronic Integrator. The integrator operates on the principle of voltage to frequency conversion. Output voltage from the chromatograph serves as input to the integrator. The output frequency from the integrator is proportional to the input voltage and these output pulses are fed into a seven-digit decade counter. These counts are stored in the counter until they are read out and printed by a Kienzle Digital Printer.

The peak area fraction or count fraction from a chromatograph is not a common indicator of composition for a particular sample. Composition is generally reported in terms of mole or weight fraction. The purpose of the chromatograph calibration was to develop a means for converting count fractions obtained from the chromatograph analysis to weight fraction.

The combination of sample size and column temperature which gave the best reproducibility was determined prior to calibrating the chromatograph. This was done by analyzing a large number of duplicate samples for different sample sizes and chromatograph column temperatures. A column temperature of 145°C and a liquid sample of two micro-liters gave the lowest standard deviation of any combination of column temperature and sample size used.

Eleven standard samples were used for calibration purposes. These were prepared from research grade materials with the aid of a Mettler balance. The samples covered the full range of compositions that were obtained in the experimental distillation runs. The standard samples were refrigerated at -10°C until analysis to prevent evaporative losses. Twelve analyses were made for each standard sample in order to make the results as accurate as possible and to provide a statistical evaluation of the chromatograph performance. The composition of the standards and the chromatograph results are shown in Table XI.

In order to facilitate the use of these results, the weight fraction of the components in the sample were correlated as a function of the count fractions from the chromatograph output. Figure 21 indicates how the weight fraction varied as a function of the count fraction for each component. Attempts to fit a simple linear or quadratic model to these results gives unsatisfactory results at 1) the extreme composition values and 2) for the toluene model where benzene and para-xylene switch as the second most prevalent component. Good fits were found by breaking the correlation up so either a linear or quadratic model could be

TABLE XI

CHROMATOGRAPH CALIBRATION STANDARDS AND RESULTS

Sample	Sample Composition Wt. fr.	Average Count Fraction of Chromatograph Output	Standard Deviation	Variance x 10^{-6}	Maximum Count Fraction Error (+)	(-)
Benzene						
A	.90308	.90733	.00038	.141	.00059	.00038
B	.70038	.70215	.00026	.066	.00033	.00042
C	.50543	.51144	.00027	.073	.00047	.00024
D	.30763	.31325	.00023	.054	.00062	.00028
E	.20185	.20569	.00044	.193	.00072	.00100
F	.10541	.10728	.00050	.253	.00057	.00135
G	.04936	.04916	.00029	.085	.00036	.00081
H	.02830	.02681	.00010	.009	.00014	.00019
I	.01104	.00919	.00008	.006	.00010	.00014
J	.00139	-	-	-	-	-
K	-	-	-	-	-	-
Toluene						
A	.09692	.09267	.00034	.116	.00038	.00059
B	.29926	.24073	.00024	.058	.00042	.00033
C	.49457	.48856	.00022	.049	.00024	.00047
D	.69237	.68675	.00024	.060	.00028	.00062
E	.79815	.79431	.00048	.228	.00100	.00072
F	.89296	.89272	.00052	.271	.00135	.00057
G	.94070	.94574	.00051	.260	.00097	.00064
H	.94626	.95266	.00031	.098	.00046	.00032
I	.93990	.94026	.00034	.119	.00054	.00043
J	.89990	.90556	.00034	.119	.00026	.00053
K	.83047	.83359	.00028	.076	.00037	.00057
Para-Xylene						
G	.00994	.00509	.00022	.049	.00052	.00026
H	.02544	.02054	.00025	.063	.00040	.00050
I	.04907	.04456	.00026	.066	.00033	.00048
J	.09871	.09443	.00028	.077	.00054	.00031
K	.16953	.16641	.00025	.062	.00057	.00037

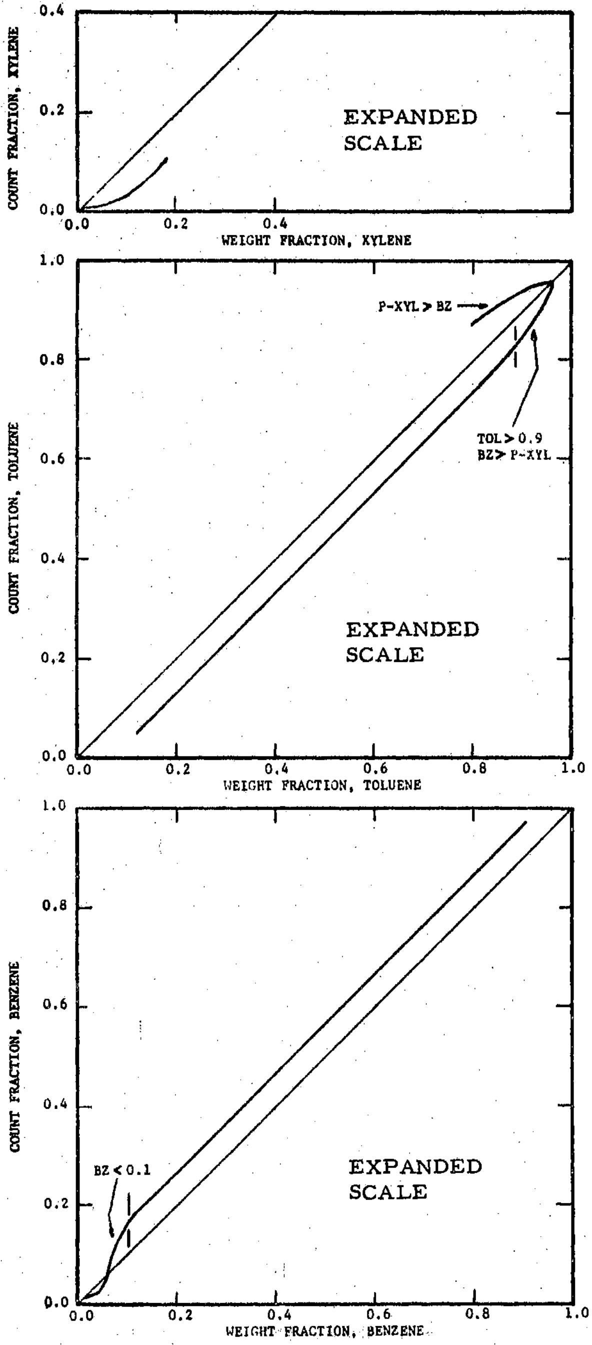

Figure 21. Chromatograph Calibration Data

applied over a portion of the total composition range. These ranges were determined primarily by inspection of the calibration data. The results were fitted by regression analysis and the model with least deviation was chosen. Table XII summarizes these analyses. These correlations were incorporated into a computer program used to reduce the chromatograph output data to weight fractions and mole fractions.

TABLE XII

REGRESSION COEFFICIENTS FOR CHROMATOGRAPH CALIBRATION

Component & Range	Model	Regression Coefficients a	b	c	Estimates of Std. Deviation σ	Estimates of Variance $\times 10^{-6}$
Benzene						
wt fraction >0.1	linear	-0.0034654	0.99803	-	0.00151	9.086
	quadratic	-0.00039430	0.98015	0.017746	0.00079	.630
wt fraction <0.1	linear	0.0023013	0.96090	-	0.00022	.050
	quadratic	0.0023480	0.95829	0.021546	0.00031	.098
Toluene						
wt fraction <0.9 (Bz>P-XYL)	linear	0.0058227	0.99682	-	0.00203	4.130
	quadratic	0.0019855	1.0198	-0.023197	0.00119	1.408
wt fraction >0.9 (Bz>P-XYL)	linear	0.09533	0.89351	-	0.00044	.192
	quadratic	3.48800	-6.4776	4.0000	-	-
wt fraction (Bz<P-XYL)	linear	0.067742	0.92125	-	0.00172	2.954
	quadratic	0.73936	-0.59197	0.84946	0.00012	.016
Para-Xylene						
entire range of study	linear	0.0050476	0.98921	-	0.00019	.037
	quadratic	0.0048810	0.99712	-0.04569	0.00014	.021

APPENDIX E

DATA AND RESULTS

TABLE XIII

DATA AND RESULTS – RUN 303

	303-1					303-2				
Date: December 1, 1967	Condenser Duty 155730 Btu/hr					Condenser Duty 156650 Btu/hr				
	Vapor OH & Reflux, moles/hr 10.93					Vapor OH & Reflux, moles/hr 10.93				
OH Pressure	Vapor	Bz	.618	Reflux Bz	.618	Vapor	Bz	.608	Reflux Bz	.610
25.33 psia	Comp.,	Tol	.382	Comp., Tol	.382	Comp.,	Tol	.392	Comp., Tol	.390
		Xyl	.000	Xyl	.000		Xyl	.000	Xyl	.000
Sump Pressure	Temp. °F		211.4	Temp. °F	191.6	Temp., °F		211.8	Temp., °F	190.4
36.58 psia										
	Reboiler Duty (not measured)					Reboiler Duty (not measured)				
	V_{reb} & L_1, moles/hr 10.71					V_{reb} & L_1, moles/hr 10.78				
	Vapor	Bz	.000	Tray 1 Bz	.000	Vapor	Bz	.000	Tray 1 Bz	.000
	Comp.	Tol	.816	Liq., Tol	.788	Comp.,	Tol	.816	Liq., Tol	.788
		Xyl	.184	Xyl	.212		Xyl	.184	Xyl	.212
	Temp., °F		256.7	Temp., °F	249.8	Temp., °F		256.8	Temp., °F	250.0

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		10.85	10.98	10.98	10.91	11.04	11.05
t_n, °F		248.5	240.4	229.3	246.0	240.4	229.5
$x_{n,i}$,	Bz	.000	.020	.148	.000	.020	.151
	Tol	.888	.954	.847	.891	.956	.844
	Xyl	.112	.026	.005	.109	.024	.005
L_{n+1}, moles/hr		10.90	10.98	11.01	10.96	11.05	11.08
t_{n+1}, °F		244.4	238.0	221.7	244.1	238.0	221.9
$x_{n+1,i}$,	Bz	.004	.042	.253	.004	.044	.255
	Tol	.922	.943	.747	.923	.943	.745
	Xyl	.074	.015	.000	.072	.013	.000
V_n, moles/hr		10.90	10.98	11.01	10.96	11.05	11.08
T_n, °F		246.2	239.5	228.0	246.2	239.5	228.0
$y_{n,i}$,	Bz	.006	.047	.277	.006	.047	.277
	Tol	.934	.940	.723	.934	.940	.723
	Xyl	.060	.013	.000	.060	.013	.000
V_{n-1}, moles/hr		10.85	10.98	10.98	10.91	11.04	11.05
T_{n-1}, °F		248.5	247.4	233.7	248.5	241.4	233.7
$y_{n-1,i}$,	Bz	.002	.023	.162	.002	.023	.162
	Tol	.895	.955	.833	.895	.955	.833
	Xyl	.103	.022	.005	.103	.022	.005
Murphree Equilibrium State							
T_n^*, °F		247.8	242.2	230.7	247.9	242.1	230.5
$y_{n,i}^*$,	Bz	.000	.046	.290	.000	.046	.295
	Tol	.945	.943	.708	.947	.943	.703
	Xyl	.055	.012	.002	.053	.011	.002
t_n^*, °F		249.0	242.6	232.2	248.9	242.4	232.1
$x_{n,i}^*$,	Bz	.002	.019	.126	.002	.019	.128
	Tol	.850	.949	.874	.853	.952	.872
	Xyl	.148	.032	.000	.145	.029	.000
Generalized Equilibrium State							
V_n^*, moles/hr		10.80	10.84	10.85	10.85	10.92	10.94
T_n^*, °F		248.17	242.34	231.67	248.06	242.23	231.51
$y_{n,i}^*$,	Bz	.003	.044	.268	.003	.045	.271
	Tol	.936	.944	.731	.937	.944	.728
	Xyl	.061	.012	.001	.060	.012	.001
L_n^*, moles/hr		10.96	11.12	11.14	11.02	11.18	11.20
t_n^*, °F		248.17	242.34	231.67	248.06	242.23	231.51
$y_{n,i}^*$,	Bz	.001	.019	.134	.001	.020	.136
	Tol	.875	.953	.862	.878	.955	.860
	Xyl	.124	.028	.004	.121	.026	.004
Tray Efficiencies							
E_{MVi},	Bz	-	.879	.736	.000	.915	.725
	Tol	.588	.990	.716	.578	1.012	.706
	Xyl	.665	.790	1.596	.659	.824	1.592
E_{MLi},	Bz	1.680	.940	.828	1.681	.958	.819
	Tol	.470	1.795	.786	.460	1.470	.781
	Xyl	.512	.635	.000	.505	.684	.000
E_i,	Bz	1.415	.973	.904	1.416	.986	.895
	Tol	.883	.486	.782	.883	.513	.786
	Xyl	.736	.832	1.370	.731	.859	1.370
E_H		.286	.378	.611	.282	.374	.636

TABLE XIV

DATA AND RESULTS – RUN 304

Date: December 4, 1967

OH Pressure
35.44 psia

Sump Pressure
36.61 psia

304-1

Condenser Duty 95070 Btu/hr
Vapor OH & Reflux, moles/hr 6.58

Vapor Comp.,	Bz	.567	Reflux Comp.,	Bz	.565
	Tol	.433		Tol	.435
	Xyl	.000		Xyl	.000
Temp. °F		215.1	Temp., °F		191.7

Reboiler Duty (not measured)
V_{reb} & L_1, moles/hr 6.46

Vapor Comp.,	Bz	.000	Tray 1 Liq.,	Bz	.000
	Tol	.823		Tol	.897
	Xyl	.177		Xyl	.103
Temp., °F		252.4	Temp., °F		249.0

304-2

Condenser Duty 95330 Btu/hr
Vapor OH & Reflux, moles/hr 6.58

Vapor Comp.,	Bz	.574	Reflux Comp.,	Bz	.568
	Tol	.426		Tol	.432
	Xyl	.000		Xyl	.000
Temp., °F		214.1	Temp., °F		189.6

Reboiler Duty (not measured)
V_{reb} & L_1, moles/hr

Vapor Comp.,	Bz	.000	Tray 1 Liq.,	Bz	.000
	Tol	.831		Tol	.893
	Xyl	.169		Xyl	.107
Temp., °F		252.5	Temp., °F		248.7

		304-1			304-2		
Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		6.62	6.70	6.72	6.63	6.71	6.74
t_n, °F		247.0	241.4	233.8	246.3	241.0	233.3
$x_{n,i}$,	Bz	.000	.013	.091	.000	.014	.102
	Tol	.878	.959	.903	.882	.960	.893
	Xyl	.122	.028	.006	.118	.026	.005
L_{n+1}, moles/hr		6.65	6.72	6.73	6.66	6.72	6.73
t_{n+1}, °F		245.1	240.0	227.2	244.7	239.1	226.5
$x_{n+1,i}$,	Bz	.003	.025	.178	.003	.029	.187
	Tol	.911	.959	.822	.914	.956	.813
	Xyl	.086	.016	.000	.083	.015	.000
V_n, moles/hr		6.64	6.72	6.73	6.66	6.72	6.74
T_n, °F		247.0	240.4	231.2	240.0	240.4	231.2
$y_{n,i}$,	Bz	.004	.040	.243	.004	.040	.243
	Tol	.938	.949	.757	.938	.949	.757
	Xyl	.058	.011	.000	.058	.011	.000
v_{n-1}, moles/hr		6.62	6.70	6.72	6.63	6.71	6.74
T_{n-1}, °F		249.0	242.2	235.6	249.0	242.2	235.6
$y_{n-1,i}$,	Bz	.000	.018	.132	.000	.018	.132
	Tol	.897	.962	.866	.897	.962	.866
	Xyl	.103	.020	.002	.103	.020	.002
Murphree Equilibrium State							
T_n^*, °F		248.3	243.0	235.2	248.2	242.8	234.3
$y_{n,i}^*$,	Bz	.000	.030	.190	.000	.032	.210
	Tol	.940	.957	.808	.942	.956	.788
	Xyl	.060	.013	.002	.058	.012	.002
t_n^*, °F		250.0	243.5	235.5	249.8	243.2	235.2
$x_{n,i}^*$,	Bz	.001	.011	.085	.001	.013	.090
	Tol	.829	.954	.915	.834	.955	.910
	Xyl	.170	.036	.000	.164	.032	.000
Generalized Equilibrium State							
V_n^*, moles/hr		6.59	6.64	6.63	6.59	6.64	6.65
T_n^*, °F		248.86	243.21	235.4	248.68	243.00	234.90
$y_{n,i}^*$,	Bz	.002	.027	.183	.002	.030	.196
	Tol	.929	.960	.815	.931	.957	.803
	Xyl	.069	.014	.002	.067	.013	.001
L_n^*, moles/hr		6.68	6.78	6.82	6.70	6.80	6.83
t_n^*, °F		248.86	243.21	235.4	248.68	243.00	234.90
$y_{n,i}^*$,	Bz	.001	.012	.088	.001	.013	.094
	Tol	.860	.958	.908	.865	.959	.902
	Xyl	.139	.030	.004	.134	.028	.004
Tray Efficiencies							
E_{MVi},	Bz	.000	.687	.882	.000	.812	.779
	Tol	.536	.139	.854	.534	.797	.754
	Xyl	.585	.758	1.674	.588	.816	1.657
E_{MLi},	Bz	1.666	.831	.936	1.669	.905	.870
	Tol	.407	-.053	.876	.405	-3.303	.818
	Xyl	.434	.596	.000	.435	.675	.000
E_i,	Bz	1.412	.891	.975	1.413	.947	.926
	Tol	.790	-.236	.816	.819	.136	.794
	Xyl	.670	.808	1.393	.670	.853	1.300
E_H		.308	.223	.630	.224	.298	.652

TABLE XV

DATA AND RESULTS — RUN 305

Date: April 23, 1968

OH Pressure
35.13 psia

Sump Pressure
36.08 psia

305-1

Condenser Duty 120,000 Btu/hr
Vapor OH & Reflux, moles/hr 8.39

Vapor	Bz	.835	Reflux	Bz	.830
Comp.,	Tol	.165	Comp.,	Tol	.170
	Xyl	.000		Xyl	.000
Temp. °F		197.8	Temp., °F		169.9

Reboiler Duty 120500 Btu/hr
V_{reb} & L_1, moles/hr 8.30

Vapor	Bz	.004	Tray 1	Bz	.003
Comp.,	Tol	.813	Liq.,	Tol	.780
	Xyl	.183		Xyl	.217
Temp., °F		254.7	Temp., °F		249.5

305-2

Condenser Duty 120300 Btu/hr
Vapor OH & Reflux, moles/hr 8.39

Vapor	Bz	.827	Reflux	Bz	.828
Comp.,	Tol	.173	Comp.,	Tol	.172
	Xyl	.000		Xyl	.000
Temp., °F		198.0	Temp., °F		169.7

Reboiler Duty 120500 Btu/hr
V_{reb} & L_1, moles/hr 8.32

Vapor	Bz	.004	Tray 1	Bz	.003
Comp.,	Tol	.814	Liq.,	Tol	.779
	Xyl	.182		Xyl	.218
Temp., °F		254.8	Temp., °F		249.5

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		8.36	8.45	8.54	8.38	8.47	8.56
t_n, °F		244.8	236.8	217.5	245.0	237.0	217.7
$x_{n,i}$,	Bz	.007	.062	.325	.007	.059	.316
	Tol	.885	.914	.670	.885	.917	.679
	Xyl	.108	.024	.005	.108	.024	.005
L_{n+1}, moles/hr		8.40	8.46	8.64	8.42	8.48	8.64
t_{n+1}, °F		242.7	232.0	207.4	242.9	232.2	207.6
$x_{n+1,i}$	Bz	.014	.117	.496	.014	.113	.479
	Tol	.915	.869	.504	.915	.873	.520
	Xyl	.070	.014	.000	.071	.014	.000
V_n, moles/hr		8.40	8.46	8.64	8.42	8.48	8.64
T_n, °F		244.9	235.2	214.0	244.9	235.2	214.0
$y_{n,i}$,	Bz	.018	.145	.558	.018	.145	.558
	Tol	.926	.845	.442	.926	.845	.442
	Xyl	.056	.010	.000	.056	.010	.000
V_{n-1}, moles/hr		8.36	8.45	8.54	8.38	8.47	8.56
T_{n-1}, °F		247.3	239.0	222.5	247.3	239.0	222.5
$y_{n-1,i}$,	Bz	.010	.073	.394	.010	.073	.394
	Tol	.892	.908	.605	.892	.908	.605
	Xyl	.098	.020	.001	.098	.020	.001
Murphree Equilibrium State							
T_n^*, °F		246.2	238.0	218.3	246.2	239.2	218.8
$y_{n,i}^*$,	Bz	.018	.134	.537	.017	.051	.527
	Tol	.930	.856	.462	.930	.917	.471
	Xyl	.052	.010	.001	.052	.032	.002
t_n^*, °F		247.6	239.0	220.2	247.7	238.2	221.1
$x_{n,i}^*$,	Bz	.006	.053	.292	.006	.129	.278
	Tol	.852	.915	.708	.850	.861	.722
	Xyl	.142	.032	.000	.144	.010	.000
Generalized Equilibrium State							
V_n^*, moles/hr		8.32	8.35	8.51	8.35	8.37	8.50
T_n^*, °F		246.71	238.57	219.34	246.77	238.73	220.04
$y_{n,i}^*$,	Bz	.016	.123	.516	.015	.119	.503
	Tol	.926	.866	.483	.927	.870	.496
	Xyl	.058	.011	.001	.058	.011	.001
L_n^*, moles/hr		8.43	8.56	8.67	8.46	8.58	8.71
t_n^*, °F		246.71	238.57	219.3	246.77	238.73	220.04
$y_{n,i}^*$,	Bz	.007	.056	.307	.006	.054	.296
	Tol	.874	.917	.689	.874	.919	.700
	Xyl	.119	.057	.004	.120	.027	.004
Tray Efficiencies							
E_{MV_i},	Bz	.668	.764	.809	.649	.769	.774
	Tol	.671	.768	.799	.663	.775	.763
	Xyl	.670	.751	1.431	.661	.743	1.437
E_{ML_i},	Bz	.818	.866	.839	.805	.870	.810
	Tol	.478	.982	.814	.471	.996	.786
	Xyl	.519	.578	.000	.509	.567	.000
E_i,	Bz	.879	.922	.934	.869	.925	.919
	Tol	.928	.746	.849	.911	.741	.821
	Xyl	.743	.803	1.301	.736	.796	1.303
E_H		.318	.544	.677	.322	.540	.638

TABLE XVI

DATA AND RESULTS – RUN 306

Date: April 25, 1968
OH Pressure 36.65 psia
Sump Pressure 37.75 psia

306-1

Condenser Duty 111670 Btu/hr
Vapor OH & Reflux, moles/hr 6.99

Vapor Comp.,	Bz	.810	Reflux Comp.,	Bz	.809
	Tol	.190		Tol	.191
	Xyl	.000		Xyl	.000
Temp. °F		201.7	Temp., °F		130.3

Reboiler Duty 116500 Btu/hr
V_{reb} & L_1, moles/hr 7.75

Vapor Comp.,	Bz	.003	Tray 1 Liq.,	Bz	.000
	Tol	.810		Tol	.779
	Xyl	.187		Xyl	.221
Temp., °F		257.2	Temp., °F		252.4

306-2

Condenser Duty 111830 Btu/hr
Vapor OH & Reflux, moles/hr 6.99

Vapor Comp.,	Bz	.805	Reflux Comp.,	Bz	.804
	Tol	.195		Tol	.196
	Xyl	.000		Xyl	.000
Temp., °F		220.2	Temp., °F		130.4

Reboiler Duty 116500 Btu/hr
V_{reb} & L_1, moles/hr 7.77

Vapor Comp.,	Bz	.003	Tray 1 Liq.,	Bz	.000
	Tol	.809		Tol	.779
	Xyl	.188		Xyl	.221
Temp., °F		257.4	Temp., °F		252.8

	306-1			306-2		
Measured Data	**Tray 2**	**Tray 5**	**Tray 8**	**Tray 2**	**Tray 5**	**Tray 8**
L_n, moles/hr	7.80	7.88	7.95	7.81	7.90	7.99
t_n, °F	247.7	240.1	222.6	247.8	240.3	224.2
$x_{n,i}$, Bz	.006	.050	.278	.006	.048	.265
Tol	.887	.926	.717	.882	.927	.730
Xyl	.107	.024	.005	.113	.025	.005
L_{n+1}, moles/hr	7.84	7.90	8.04	7.85	7.91	8.07
t_{n+1}, °F	245.6	236.0	212.9	245.8	236.3	214.40
$x_{n+1,i}$, Bz	.012	.094	.446	.011	.093	.428
Tol	.917	.892	.554	.916	.893	.572
Xyl	.071	.014	.000	.073	.014	.000
V_n, moles/hr	7.84	7.90	8.04	7.85	7.91	8.07
T_n, °F	247.7	238.6	218.8	247.7	238.6	218.8
$y_{n,i}$, Bz	.015	.120	.521	.015	.120	.521
Tol	.929	.868	.479	.929	.868	.479
Xyl	.056	.012	.000	.056	.012	.000
V_{n-1}, moles/hr	7.80	7.88	7.95	7.81	7.90	7.99
T_{n-1}, °F	250.2	242.1	227.5	250.2	242.1	227.5
$y_{n-1,i}$, Bz	.007	.060	.344	.007	.060	.344
Tol	.896	.921	.656	.896	.921	.656
Xyl	.097	.019	.000	.097	.019	.000
Murphree Equilibrium State						
T_n^*, °F	249.2	241.9	224.0	249.5	242.1	224.9
$y_{n,i}^*$, Bz	.015	.110	.478	.013	.105	.462
Tol	.933	.880	.520	.932	.884	.536
Xyl	.052	.010	.002	.055	.011	.002
t_n^*, °F	250.8	242.8	225.5	250.9	242.9	226.4
$x_{n,i}^*$, Bz	.005	.043	.253	.004	.042	.240
Tol	.852	.926	.747	.849	.926	.760
Xyl	.144	.032	.000	.146	.032	.000
Generalized Equilibrium State						
V_n^*, moles/hr	7.75	7.77	7.89	7.76	7.79	7.92
T_n^*, °F	249.80	242.34	224.85	250.01	242.48	225.71
$y_{n,i}^*$, Bz	.013	.100	.462	.012	.097	.445
Tol	.929	.889	.537	.927	.891	.554
Xyl	.058	.011	.001	.061	.012	.001
L_n^*, moles/hr	7.88	8.00	8.10	7.80	8.02	8.13
t_n^*, °F	249.80	242.34	224.85	250.01	242.48	225.71
$y_{n,i}^*$, Bz	.005	.045	.265	.005	.044	.252
Tol	.875	.928	.731	.871	.929	.744
Xyl	.119	.026	.004	.124	.027	.004
Tray Efficiencies						
$E_{MV,i}$, Bz	.637	.741	.836	.713	.777	.826
Tol	.646	.739	.825	.687	.782	.814
Xyl	.645	.746	1.471	.690	.760	1.481
$E_{ML,i}$, Bz	.797	.854	.872	.846	.877	.867
Tol	.459	.998	.847	.515	1.033	.840
Xyl	.492	.575	.000	.544	.592	.000
E_i, Bz	.864	.916	.956	.903	.932	.951
Tol	.935	.658	.850	.982	.666	.844
Xyl	.720	.796	1.316	.757	.807	1.321
E_H	.288	.445	.629	.278	.450	.649

TABLE XVII

DATA AND RESULTS - RUN 307

Date: April 28, 1968

OH Pressure
34.29 psia

Sump Pressure
35.59 psia

307-1

Condenser Duty 168640 Btu/hr
Vapor OH & Reflux, moles/hr 11.68

Vapor Comp.,	Bz	.904	Reflux Comp.,	Bz	.893
	Tol	.096		Tol	.107
	Xyl	.000		Xyl	.000
Temp. °F		190.9	Temp., °F		157.6

Reboiler Duty 171500 Btu/hr
V_{reb} & L_1, moles/hr 11.59

Vapor Comp.,	Bz	.007	Tray 1 Liq.,	Bz	.006
	Tol	.823		Tol	.803
	Xyl	.170		Xyl	.191
Temp., °F		253.7	Temp., °F		246.4

307-2

Condenser Duty 168810 Btu/hr
Vapor OH & Reflux, moles/hr 11.68

Vapor Comp.,	Bz	.899	Reflux Comp.,	Bz	.892
	Tol	.101		Bz	.108
	Xyl	.000		Xyl	.000
Temp., °F		190.6	Temp., °F		157.6

Reboiler Duty 171500 Btu/hr
V_{reb} & L_1, moles/hr 11.56

Vapor Comp.,	Bz	.007	Tray 1, Liq.,	Bz	.006
	Tol	.821		Tol	.802
	Xyl	.172		Xyl	.192
Temp., °F		253.7	Temp., °F		246.4

		307-1			307-2		
Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		11.72	11.82	12.13	11.76	11.89	12.20
t_n, °F		241.6	229.2	204.0	242.0	230.04	204.6
$x_{n,i}$,	Bz	.017	.129	.532	.020	.143	.557
	Tol	.889	.850	.468	.887	.837	.443
	Xyl	.094	.021	.000	.093	.020	.000
L_{n+1}, moles/hr		11.77	11.90	12.26	11.79	11.94	12.35
t_{n+1}, °F		239.2	222.5	197.2	239.2	222.5	197.2
$x_{n+1,i}$,	Bz	.032	.240	.644	.038	.257	.681
	Tol	.906	.749	.347	.902	.733	.319
	Xyl	.062	.011	.009	.060	.100	.000
V_n, moles/hr		11.77	11.90	12.26	11.79	11.94	12.35
T_n, °F		242.3	228.4	203.3	242.3	228.4	203.27
$y_{n,i}$,	Bz	.037	.240	.700	.037	.240	.700
	Tol	.908	.750	.300	.908	.750	.300
	Xyl	.055	.010	.000	.055	.010	.000
V_{n-1}, moles/hr		11.72	11.82	12.13	11.76	11.89	12.20
T_{n-1}, °F		242.7	234.0	211.8	244.7	234.05	211.8
$y_{n-1,i}$,	Bz	.018	.144	.550	.018	.144	.550
	Tol	.888	.838	.450	.888	.838	.450
	Xyl	.094	.018	.000	.094	.018	.000
Murphree Equilibrium State							
T_n^*, °F		243.8	231.4	205.1	243.5	230.3	203.8
$y_{n,i}^*$,	Bz	.040	.261	.736	.046	.285	.756
	Tol	.916	.731	.264	.910	.707	.244
	Xyl	.044	.008	.000	.044	.008	.000
t_n^*, °F		245.4	232.5	211.8	245.0	231.8	208.5
$x_{n,i}^*$,	Bz	.013	.117	.420	.016	.127	.466
	Tol	.858	.855	.549	.859	.847	.534
	Xyl	.129	.027	.031	.125	.026	.000
Generalized Equilibrium State							
V_n^*, moles/hr		11.61	11.72	12.10	11.68	11.85	12.31
T_n°, °F		244.44	231.96	207.84	244.11	231.11	205.91
$y_{n,i}^\circ$,	Bz	.034	.249	.696	.041	.268	.723
	Tol	.915	.742	.302	.910	.723	.277
	Xyl	.050	.009	.002	.049	.008	.000
L_n°, moles/hr		11.88	12.01	12.30	11.87	11.98	12.25
t_n^*, °F		244.44	231.96	207.84	244.11	231.11	205.91
$y_{n,i}^*$,	Bz	.014	.123	.482	.017	.133	.516
	Tol	.880	.855	.511	.879	.845	.484
	Xyl	.106	.022	.007	.104	.022	.000
Tray Efficiencies							
E_{MV_i},	Bz	.643	.841	.551	.694	.804	.623
	Tol	.646	.848	.593	.642	.806	.623
	Xyl	.644	.778	.000	.670	.782	.000
E_{ML},	Bz	.798	.900	.502	.830	.873	.578
	Tol	.363	.948	.600	.344	.911	.578
	Xyl	.485	.594	-.376	.511	.599	.000
E_i,	Bz	.868	.957	.735	.890	.928	.768
	Tol	1.151	.838	.702	1.043	.872	.734
	Xyl	.717	.818	5.010	.742	.830	.000
E_H		.295	.558	.446	.411	.744	.687

TABLE XVIII

DATA AND RESULTS - RUN 308

Date: May 4, 1968

OH Pressure 34.87 psia

Sump Pressure 36.07 psia

308-1

Condenser Duty 143470 Btu/hr

Vapor OH & Reflux, moles/hr 10.02

Vapor	Bz	.869	Reflux	Bz	.879
Comp.,	Tol	.131	Comp.,	Tol	.121
	Xyl	.000		Xyl	.000
Temp., °F		193.3	Temp., °F		165.3

Reboiler Duty 143000 Btu/hr

V_{reb} & L_1, moles/hr 9.89

Vapor	Bz	.005	Tray 1	Bz	.004
Comp.,	Tol	.819	Comp.,	Tol	.793
	Xyl	.176		Xyl	.203
Temp., °F		254.3	Temp., °F		248.2

308-2

Condenser Duty 143720 Btu/hr

Vapor OH & Reflux, moles/hr 10.02

Vapor	Bz	.867	Reflux	Bz	.876
Comp.,	Tol	.133	Comp.,	Tol	.124
	Xyl	.000		Xyl	.000
Temp., °F		193.2	Temp., °F		164.7

Reboiler Duty 143000 Btu/hr

V_{reb} & L_1, moles/hr 9.97

Vapor	Bz	.005	Tray 1	Bz	.004
Comp.,	Tol	.819	Comp.,	Tol	.750
	Xyl	.176		Xyl	.245
Temp., °F		254.2	Temp., °F		248.3

		308-1			308-2		
Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		10.00	10.09	10.28	10.01	10.10	10.27
t_n, °F		243.8	233.3	209.7	243.8	233.4	209.8
$x_{n,i}$,	Bz	.013	.099	.452	.012	.090	.437
	Tol	.887	.879	.544	.885	.887	.559
	Xyl	.100	.022	.004	.103	.023	.004
L_{n+1}, moles/hr		10.03	10.13	10.42	10.05	10.12	10.42
t_{n+1}, °F		241.2	226.9	200.8	241.4	226.85	200.9
$x_{n+1,i}$	Bz	.025	.185	.616	.023	.171	.598
	Tol	.910	.803	.384	.910	.817	.402
	Xyl	.065	.012	.000	.067	.012	.000
V_n, moles/hr		10.03	10.13	10.42	10.05	10.12	10.42
T_n, °F		243.8	231.5	207.6	243.8	231.5	207.6
$y_{n,i}$,	Bz	.027	.206	.662	.027	.206	.662
	Tol	.917	.783	.338	.917	.783	.338
	Xyl	.056	.010	.000	.056	.010	.000
V_{n-1}, moles/hr		10.00	10.09	10.28	10.01	10.09	10.28
T_{n-1}, °F		246.5	236.8	216.5	246.5	236.81	216.5
$y_{n-1,i}$,	Bz	.013	.110	.500	.013	.110	.500
	Tol	.893	.871	.500	.893	.871	.500
	Xyl	.094	.019	.000	.094	.019	.000
Murphree Equilibrium State							
T_n^*, °F		245.3	234.7	210.4	245.5	235.4	211.3
$y_{n,i}^*$,	Bz	.031	.206	.669	.028	.191	.655
	Tol	.921	.785	.330	.923	.802	.344
	Xyl	.048	.009	.001	.049	.009	.001
t_n^*, °F		246.8	235.9	213.4	247.0	236.5	214.4
$x_{n,i}^*$,	Bz	.010	.087	.398	.009	.080	.381
	Tol	.856	.884	.602	.854	.890	.619
	Xyl	.134	.029	.000	.137	.030	.000
Generalized Equilibrium State							
V_n^*, moles/hr		9.93	9.98	10.30	9.94	9.94	10.25
T_n^*, °F		245.87	235.36	211.87	246.08	236.00	212.77
$y_{n,i}^*$,	Bz	.027	.193	.644	.025	.179	.628
	Tol	.920	.797	.355	.920	.811	.371
	Xyl	.054	.010	.001	.055	.010	.001
L_n^*, moles/hr		10.10	10.23	10.40	10.12	10.27	10.45
t_n^*, °F		245.87	235.36	211.87	246.08	236.00	212.77
$y_{n,i}^*$,	Bz	.011	.092	.476	.010	.084	.410
	Tol	.877	.884	.570	.875	.891	.587
	Xyl	.112	.024	.004	.115	.025	.003
Tray Efficiencies							
E_{MV_i},	Bz	.664	.799	.757	.710	.804	.742
	Tol	.666	.805	.748	.658	.810	.733
	Xyl	.665	.755	1.355	.673	.770	1.362
E_{ML_i},	Bz	.813	.879	.754	.842	.885	.744
	Tol	.427	.945	.733	.442	.954	.724
	Xyl	.510	.572	.000	.520	.594	.000
E_i,	Bz	.876	.938	.893	.901	.945	.894
	Tol	.976	.804	.820	1.006	.772	.802
	Xyl	.738	.803	1.266	.743	.812	1.268
E_H		.384	.575	.641	.317	.536	.575

TABLE XIX

DATA AND RESULTS - RUN 309

309-1

Date: May 14, 1968

OH Pressure
35.06 psia

Sump Pressure
37.26 psia

Condenser Duty 146540 Btu/hr
Vapor OH & Reflux, moles/hr 9.[illegible]

Vapor	Bz	.518	Reflux	Bz	.541
Comp.,	Tol	.482	Comp.,	Tol	.459
	Xyl	.000		Xyl	.000
Temp., °F		215.0	Temp., °F		185.0

Reboiler Duty 144000 Btu/hr
V_{reb} & L_1, moles/hr 10.08

Vapor	Bz	.000	Tray 1	Bz	.000
Comp.,	Tol	.814	Comp.,	Tol	.787
	Xyl	.186		Xyl	.213
Temp., °F		255.4	Temp., °F		249.1

Condenser Duty 146720 Btu/hr
Vapor OH & Reflux, moles/hr 9.85

Vapor	Bz	.510	Reflux	Bz	.530
Comp.,	Tol	.490	Comp.,	Tol	.470
	Xyl	.000		Xyl	.000
Temp., °F		215.5	Temp., °F		185.2

Reboiler Duty 144000 Btu/hr
V_{reb} & L_1, moles/hr 10.09

Vapor	Bz	.000	Tray 1	Bz	.000
Comp.,	Tol	.812	Comp.,	Tol	.786
	Xyl	.188		Xyl	.214
Temp., °F		255.6	Temp., °F		249.2

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		10.19	10.30	10.31	10.20	10.32	10.32
t_n, °F		245.2	240.0	231.2	245.2	240.1	231.2
$x_{n,i}$,	Bz	.000	.015	.109	.000	.014	.103
	Tol	.888	.959	.885	.887	.960	.891
	Xyl	.112	.026	.006	.113	.026	.006
L_{n+1}, moles/hr		10.24	10.33	10.32	10.26	10.33	10.32
t_{n+1}, °F		243.3	238.4	224.7	243.4	238.2	224.9
$x_{n+1,i}$,	Bz	.004	.031	.195	.004	.029	.188
	Tol	.923	.954	.805	.922	.956	.812
	Xyl	.073	.015	.000	.074	.015	.000
V_n, moles/hr		10.24	10.33	10.32	10.26	10.33	10.32
T_n, °F		245.5	239.5	230.8	245.5	230.8	245.5
$y_{n,i}$,	Bz	.004	.034	.228	.004	.034	.228
	Tol	.934	.953	.772	.934	.953	.772
	Xyl	.062	.013	.000	.062	.013	.000
V_{n-1}, moles/hr		10.19	10.30	10.31	10.20	10.32	10.32
T_{n-1}, °F		247.9	241.4	234.9	247.9	241.4	247.9
$y_{n-1,i}$,	Bz	.000	.016	.123	.000	.016	.123
	Tol	.896	.961	.872	.896	.961	.872
	Xyl	.104	.022	.005	.104	.022	.005
Murphree Equilibrium State							
T_n^*, °F		248.7	243.0	233.5	248.8	243.1	234.0
$y_{n,i}^*$,	Bz	.000	.034	.223	.000	.032	.213
	Tol	.945	.954	.775	.945	.956	.785
	Xyl	.055	.012	.002	.055	.012	.002
t_n^*, °F		249.9	243.4	234.5	250.0	243.5	234.8
$x_{n,i}^*$,	Bz	.002	.013	.094	.002	.013	.090
	Tol	.852	.954	.906	.850	.954	.910
	Xyl	.146	.032	.000	.148	.034	.000
Generalized Equilibrium State							
V_n^*, moles/hr		10.03	10.13	10.15	10.04	10.14	10.13
T_n^*, °F		249.04	243.15	234.16	249.10	243.26	234.50
$y_{n,i}^*$,	Bz	.003	.032	.206	.003	.030	.198
	Tol	.937	.955	.792	.936	.957	.800
	Xyl	.060	.013	.002	.061	.013	.002
L_n^*, moles/hr		10.40	10.50	10.48	10.41	10.52	10.51
t_n^*, °F		249.04	243.15	234.16	249.10	243.26	234.50
$y_{n,i}^*$,	Bz	.001	.014	.100	.001	.013	.095
	Tol	.876	.958	.896	.874	.959	.901
	Xyl	.123	.028	.004	.125	.028	.004
Tray Efficiencies							
E_{MV_i},	Bz	.000	.821	.760	.000	.843	.778
	Tol	.610	.960	.732	.611	1.167	.749
	Xyl	.676	.767	1.647	.674	.758	1.655
E_{ML_i},	Bz	1.682	.909	.855	1.680	.921	.869
	Tol	.489	-28.076	.800	.489	-2.484	.811
	Xyl	.525	.606	.000	.523	.594	.000
E_i,	Bz	1.425	.958	.921	1.425	.967	.934
	Tol	1.158	.132	.761	1.160	.152	.751
	Xyl	.733	.807	1.385	.731	.800	1.385
E_H		.172	.170	.556	.143	.222	.520

TABLE XX

DATA AND RESULTS – RUN 310

Date: May 16, 1968

OH Pressure
34.66 psia

Sump Pressure
35.86 psia

310-1

Condenser Duty 121530 Btu/hr
Vapor OH & Reflux, moles/hr 8.17

Vapor	Bz	.522	Reflux	Bz	.543
Comp.,	Tol	.478	Comp.,	Tol	.457
	Xyl	.000		Xyl	.000
Temp., °F		214.4	Temp., °F		184.3

Reboiler Duty 125000 Btu/hr
V_{reb} & L_1, moles/hr 8.38

Vapor	Bz	.000	Tray 1	Bz	.000
Comp.,	Tol	.811	Liq.,	Tol	.776
	Xyl	.189		Xyl	.224
Temp., °F		254.6	Temp., °F		249.2

310-2

Condenser Duty 121560 Btu/hr
Vapor OH & Reflux, moles/hr 8.17

Vapor	Bz	.522	Reflux	Bz	.540
Comp.,	Tol	.478	Comp.,	Tol	.460
	Xyl	.000		Xyl	.000
Temp., °F		214.4	Temp., °F		184.2

Reboiler Duty 125000 Btu/hr
V_{reb} & L_1, moles/hr 8.38

Vapor	Bz	.000	Tray 1	Bz	.000
Comp.,	Tol	.809	Liq.,	Tol	.773
	Xyl	.191		Xyl	.227
Temp., °F		254.6	Temp., °F		249.1

		310-1			310-2		
Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		8.45	8.54	8.57	8.45	8.55	8.57
t_n, °F		244.7	239.6	231.8	244.7	239.6	231.7
$x_{n,i}$,	Bz	.000	.014	.098	.000	.014	.094
	Tol	.888	.959	.897	.885	.959	.900
	Xyl	.112	.027	.006	.115	.027	.006
L_{n+1}, moles/hr		8.49	8.56	8.58	8.49	8.56	8.57
t_{n+1}, °F		242.8	237.8	225.3	243.0	237.8	225.2
$x_{n+1,i}$,	Bz	.004	.029	.187	.004	.027	.180
	Tol	.920	.956	.813	.920	.957	.820
	Xyl	.076	.015	.000	.077	.016	.000
V_n, moles/hr		8.49	8.56	8.58	8.49	8.56	8.57
T_n, °F		244.9	238.8	229.7	244.9	238.8	229.7
$y_{n,i}$,	Bz	.004	.036	.223	.004	.036	.223
	Tol	.935	.952	.777	.935	.952	.777
	Xyl	.061	.012	.000	.061	.012	.000
V_{n-1}, moles/hr		8.45	8.55	8.57	8.45	8.55	8.57
T_{n-1}, °F		247.2	240.6	234.0	247.2	240.6	234.0
$y_{n-1,i}$,	Bz	.000	.016	.128	.000	.016	.128
	Tol	.898	.962	.867	.898	.962	.867
	Xyl	.102	.022	.005	.102	.022	.005
Murphree Equilibrium State							
T_n^*, °F		246.5	241.5	233.3	246.6	241.5	233.5
$y_{n,i}^*$,	Bz	.000	.032	.202	.000	.032	.196
	Tol	.945	.956	.796	.944	.956	.801
	Xyl	.055	.012	.002	.056	.012	.002
t_n^*, °F		248.0	241.8	233.7	248.0	241.9	234.0
$x_{n,i}^*$,	Bz	.001	.013	.090	.001	.012	.086
	Tol	.846	.955	.910	.845	.954	.914
	Xyl	.153	.032	.000	.154	.034	.000
Generalized Equilibrium State							
V_n^ϕ, moles/hr		8.40	8.45	8.46	8.40	8.45	8.43
T_n^ϕ, °F		246.95	241.61	233.6	247.0	241.67	233.9
$y_{n,i}^\phi$,	Bz	.002	.030	.193	.002	.029	.186
	Tol	.936	.957	.805	.934	.958	.812
	Xyl	.062	.013	.002	.063	.013	.002
L_n^ϕ, moles/hr		8.54	8.66	8.70	8.54	8.66	8.71
t_n^ϕ, °F		246.95	241.6	233.6	247.0	241.7	233.9
$y_{n,i}^\phi$,	Bz	.001	.013	.093	.001	.013	.089
	Tol	.873	.958	.903	.871	.958	.907
	Xyl	.126	.029	.004	.128	.029	.004
Tray Efficiencies							
E_{MV_i},	Bz	.000	.873	.855	.000	.774	.837
	Tol	.557	1.094	.828	.590	.771	.807
	Xyl	.618	.826	1.661	.650	.774	1.666
E_{ML_i},	Bz	1.673	.938	.919	1.673	.884	.909
	Tol	.429	-3.281	.862	.465	-.836	.846
	Xyl	.463	.689	.000	.498	.616	.000
E_i,	Bz	1.415	.971	.964	1.415	.932	.958
	Tol	.865	.118	.810	.905	.098	.784
	Xyl	.696	.860	1.389	.723	.819	1.390
E_H		.272	.276	.616	.230	.292	.582

TABLE XXI

DATA AND RESULTS - RUN 311

	311-1	311-2
Date: June 8, 1968 OH Pressure 33.41 psia Sump Pressure 34.65 psia	Condenser Duty 152570 Btu/hr Vapor OH & Reflux, moles/hr 11.14 Vapor Bz .966 Reflux Bz .967 Comp., Tol .034 Comp., Tol .033 Xyl .000 Xyl .000 Temp. °F 183.2 Temp. °F 169.3	Condenser Duty 152610 Btu/hr Vapor OH & Reflux, moles/hr 11.14 Vapor Bz .962 Reflux Bz .965 Comp., Tol .038 Comp., Tol .035 Xyl .000 Xyl .000 Temp. °F 183.1 Temp. °F 169.4
	Reboiler Duty 183000 Btu/hr V_{reb} & L_1, moles/hr 10.48 Vapor Bz .029 Tray 1 Bz .024 Comp., Tol .814 Liq., Tol .798 Xyl .157 Xyl .178 Temp. °F 249.8 Temp. °F 242.7	Reboiler Duty 183000 Btu/hr V_{reb} & L_1, moles/hr 10.47 Vapor Bz .027 Tray 1 Bz .022 Comp., Tol .813 Liq., Tol .800 Xyl .161 Xyl .178 Temp. °F 249.9 Temp. °F 242.7

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		10.59	10.82	11.34	10.60	10.80	11.31
t_n, °F		235.4	212.2	189.6	236.0	212.8	190.2
$x_{n,i}$,	Bz	.071	.387	.821	.062	.352	.788
	Tol	.847	.599	.179	.854	.634	.212
	Xyl	.082	.014	.000	.084	.014	.000
L_{n+1}, moles/hr		10.63	10.99	11.42	10.62	10.95	11.41
t_{n+1}, °F		229.9	202.8	186.1	230.3	203.5	186.2
$x_{n+1,i}$	Bz	.128	.549	.872	.112	.506	.867
	Tol	.821	.444	.128	.836	.487	.133
	Xyl	.051	.007	.000	.052	.007	.000
V_n, moles/hr		10.63	10.99	11.42	10.62	10.95	11.41
T_n, °F		235.8	209.9	188.8	235.8	209.9	188.8
$y_{n,i}$,	Bz	.131	.568	.899	.131	.568	.899
	Tol	.824	.426	.101	.824	.426	.101
	Xyl	.045	.006	.000	.045	.006	.000
V_{n-1}, moles/hr		10.59	10.82	11.34	10.60	10.80	11.31
T_{n-1}, °F		240.4	219.9	193.8	240.4	219.9	193.8
$y_{n-1,i}$,	Bz	.069	.382	.819	.069	.382	.819
	Tol	.806	.605	.181	.806	.605	.181
	Xyl	.080	.013	.000	.080	.013	.000
Murphree Equilibrium State							
T_n^*, °F		237.0	212.4	190.1	237.8	214.5	191.5
$y_{n,i}$,	Bz	.158	.608	.920	.140	.578	.904
	Tol	.807	.388	.080	.824	.425	.096
	Xyl	.035	.004	.000	.036	.004	.000
t_n^*, °F		239.4	215.9	193.9	240.0	218.3	194.4
$x_{n,i}^*$,	Bz	.056	.331	.731	.048	.295	.721
	Tol	.829	.646	.269	.837	.682	.279
	Xyl	.115	.023	.000	.115	.023	.000
Generalized Equilibrium State							
V_n^*, moles/hr		10.50	10.88	11.35	10.47	10.73	11.29
T_n^*, °F		238.16	214.08	191.42	238.84	216.29	192.42
$y_{n,i}^*$,	Bz	.138	.579	.904	.122	.538	.891
	Tol	.821	.417	.096	.837	.457	.109
	Xyl	.041	.004	.000	.042	.005	.000
L_n^*, moles/hr		10.71	10.94	11.41	10.74	11.01	11.43
t_n^*, °F		238.16	214.08	191.42	238.84	216.29	192.47
$y_{n,i}^*$,	Bz	.061	.359	.789	.054	.323	.765
	Tol	.846	.625	.211	.853	.661	.235
	Xyl	.093	.016	.000	.093	.016	.000
Tray Efficiencies							
E_{MV_i},	Bz	.653	.733	.519	.641	.703	.677
	Tol	.650	.734	.519	.600	.704	.677
	Xyl	.655	.703	.000	.666	.687	.000
E_{ML_i},	Bz	.785	.744	.367	.780	.729	.538
	Tol	3.047	.768	.367	25.486	.754	.538
	Xyl	.485	.437	.000	.500	.431	.000
E_i,	Bz	.863	.876	.682	.860	.878	.847
	Tol	.704	.821	.610	.600	.778	.747
	Xyl	.728	.750	.000	.736	.732	.000
E_H		.525	.665	.453	.515	.532	.443

TABLE XXII

DATA AND RESULTS - RUN 312

Date: June 9, 1968

OH Pressure
35.99 psia

Sump Pressure
37.09 psia

312-1

Condenser Duty 104730 Btu/hr
Vapor OH & Reflux, moles/hr 7.60

Vapor	Bz	.946	Reflux	Bz	.951
Comp.,	Tol	.054	Comp.,	Tol	.049
	Xyl	.000		Xyl	.000
Temp. °F		189.5	Temp. °F		172.4

Reboiler Duty 140000 Btu/hr
V_{reb} & L_1, moles/hr 7.25

Vapor	Bz	.012	Tray 1	Bz	.010
Comp.,	Tol	.809	Liq.,	Tol	.792
	Xyl	.179		Xyl	.198
Temp. °F		253.9	Temp. °F		249.2

312-2

Condenser Duty 104820 Btu/hr
Vapor OH & Reflux, moles/hr 7.60

Vapor	Bz	.945	Reflux	Bz	.950
Comp.,	Tol	.055	Comp.,	Tol	.050
	Xyl	.000		Xyl	.000
Temp. °F		189.5	Temp. °F		172.2

Reboiler Duty 140000 Btu/hr
V_{reb} & L_1, moles/hr 7.24

Vapor	Bz	.010	Tray 1	Bz	.010
Comp.,	Tol	.803	Liq.,	Tol	.790
	Xyl	.186		Xyl	.200
Temp. °F		254.0	Temp. °F		249.2

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		7.31	7.40	7.65	7.32	7.41	7.68
t_n, °F		243.8	227.1	201.0	243.8	227.0	201.2
$x_{n,i}$,	Bz	.031	.207	.633	.030	.209	.641
	Tol	.876	.775	.367	.876	.773	.359
	Xyl	.093	.018	.000	.094	.017	.000
L_{n+1}, moles/hr		7.34	7.45	7.76	7.35	7.46	7.78
t_{n+1}, °F		240.2	218.6	193.9	240.2	218.3	194.0
$x_{n+1,i}$,	Bz	.075	.398	.834	.075	.398	.834
	Tol	.880	.595	.166	.880	.595	.166
	Xyl	.045	.007	.000	.045	.077	.000
V_n, moles/hr		7.34	7.45	7.76	7.35	7.46	7.78
T_n, °F		243.2	224.6	198.8	243.2	224.6	198.8
$y_{n,i}$,	Bz	.075	.398	.834	.075	.398	.834
	Tol	.880	.595	.166	.880	.595	.166
	Xyl	.045	.007	.000	.045	.007	.000
V_{n-1}, moles/hr		7.31	7.40	7.65	7.32	7.41	7.68
T_{n-1}, °F		246.8	232.4	206.2	246.8	232.3	206.2
$y_{n-1,i}$,	Bz	.037	.248	.727	.037	.248	.727
	Tol	.883	.739	.273	.883	.739	.273
	Xyl	.080	.013	.000	.080	.013	.000
Murphree Equilibrium State							
T_n^*, °F		245.4	228.6	203.0	245.4	228.4	202.6
$y_{n,i}^*$,	Bz	.072	.384	.810	.070	.387	.815
	Tol	.885	.609	.190	.887	.606	.185
	Xyl	.043	.006	.000	.043	.006	.000
t_n^*, °F		247.4	231.2	205.2	247.0	231.2	204.9
$x_{n,i}^*$,	Bz	.023	.173	.587	.025	.174	.594
	Tol	.897	.801	.413	.851	.801	.406
	Xyl	.130	.026	.000	.124	.025	.000
Generalized Equilibrium State							
V_n^*, moles/hr		7.25	7.32	7.64	7.26	7.32	7.67
T_n^*, °F		246.23	230.00	203.82	246.10	229.90	203.46
$y_{n,i}^*$,	Bz	.061	.356	.798	.063	.358	.803
	Tol	.890	.637	.202	.889	.635	.197
	Xyl	.050	.007	.000	.049	.007	.000
L_n^*, moles/hr		7.41	7.53	7.78	7.41	7.54	7.78
t_n^*, °F		246.23	230.00	203.82	246.10	229.90	203.46
$y_{n,i}^*$,	Bz	.026	.188	.616	.027	.189	.623
	Tol	.869	.792	.384	.869	.791	.377
	Xyl	.105	.020	.000	.104	.020	.000
Tray Efficiencies							
E_{MV_i},	Bz	.598	.713	.819	.726	.702	.881
	Tol	.726	.713	.819	.539	.701	.881
	Xyl	.621	.717	.000	.687	.723	.000
E_{ML_i},	Bz	.760	.788	.760	.847	.779	.749
	Tol	.189	.820	.760	.182	.808	.749
	Xyl	.460	.506	.000	.530	.514	.000
E_i,	Bz	.840	.891	.955	.908	.883	.939
	Tol	1.799	.781	.857	1.831	.777	.855
	Xyl	.698	.767	.000	.753	.773	.000
E_H		.392	.561	.537	.418	.574	.588

TABLE XXIII

DATA AND RESULTS - RUN 313

	313-1	313-2
Date: June 11, 1968	Condenser Duty 149760 Btu/hr	Condenser Duty 139430 Btu/hr
	Vapor OH & Reflux, moles/hr 10.01	Vapor OH & Reflux, moles/hr 10.01
OH Pressure	Vapor Bz .965 Reflux Bz .869	Vapor Bz .965 Reflux Bz .967
35.87 psia	Comp. Tol .035 Comp. Tol .031	Comp. Tol .035 Comp Tol .033
	Xyl .000 Xyl .000	Xyl .000 Xyl .000
Sump Pressure	Temp. °F 187.4 Temp. °F 166.0	Temp. °F 187.5 Temp. °F 166.1
36.97 psia		
	Reboiler Duty 138000 Btu/hr	Reboiler Duty 138000 Btu/hr
	V_{reb} & L_1, moles/hr 10.27	V_{reb} & L_1, moles/hr 9.56
	Vapor Bz .021 Tray 1 Bz .021	Vapor Bz .021 Tray 1 Bz .020
	Comp. Tol .792 Liq., Tol .794	Comp. Tol .792 Liq., Tol .795
	Xyl .187 Xyl .185	Xyl .187 Xyl .185
	Temp. °F 253.5 Temp. °F 247.3	Temp. °F 253.6 Temp. °F 247.3

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		10.44	10.60	11.12	9.71	9.87	10.35
t_n, °F		240.1	216.6	193.9	240.2	217.5	194.5
$x_{n,i}$,	Bz	.065	.354	.790	.058	.331	.776
	Tol	.851	.631	.210	.855	.654	.224
	Xyl	.084	.015	.000	.087	.016	.000
L_{n+1}, moles/hr		10.47	10.79	11.25	9.73	10.01	10.47
t_{n+1}, °F		234.7	207.5	190.2	235.0	207.7	190.4
$x_{n+1,i}$,	Bz	.116	.521	.875	.103	.496	.868
	Tol	.830	.471	.125	.841	.496	.132
	Xyl	.054	.008	.000	.056	.008	.000
V_n, moles/hr		10.47	10.79	11.25	9.73	10.01	10.47
T_n, °F		240.6	215.1	193.5	240.6	215.1	193.5
$y_{n,i}$,	Bz	.136	.564	.899	.136	.564	.899
	Tol	.821	.43-	.101	.821	.430	.101
	Xyl	.054	.008	.000	.056	.008	.000
V_{n-1}, moles/hr		10.44	10.60	11.12	9.71	9.87	10.35
T_{n-1}, °F		244.8	225.2	198.7	244.8	225.2	198.7
$y_{n-1,i}$,	Bz	.068	.388	.825	.068	.388	.825
	Tol	.853	.600	.175	.853	.600	.175
	Xyl	.079	.012	.000	.079	.012	.000
Murphree Equilibrium State							
T_n^* °F		241.9	218.7	195.7	242.6	220.2	196.3
$y_{n,i}^*$,	Bz	.145	.571	.904	.131	.545	.896
	Tol	.819	.425	.096	.831	.450	.104
	Xyl	.037	.004	.000	.038	.005	.000
t_n^* °F		244.3	221.7	197.9	244.9	223.0	198.5
$x_{n,i}^*$,	Bz	.051	.309	.738	.045	.289	.725
	Tol	.831	.667	.262	.835	.688	.275
	Xyl	.118	.024	.000	.121	.024	.000
Generalized Equilibrium State							
V_n^*, moles/hr		10.33	10.59	11.12	9.58	9.78	10.34
T_n^*, °F		243.05	220.13	196.46	243.67	221.50	197.06
$y_{n,i}^*$,	Bz	.126	.547	.894	.113	.521	.887
	Tol	.832	.448	.106	.843	.473	.113
	Xyl	.042	.005	.000	.044	.005	.000
L_n^*, moles/hr		10.57	10.80	11.24	9.87	10.10	10.48
t_n^*, °F		243.05	220.13	196.46	243.67	221.50	197.06
$y_{n,i}^*$,	Bz	.056	.332	.772	.050	.310	.758
	Tol	.849	.651	.228	.853	.672	.242
	Xyl	.095	.017	.000	.097	.017	.000
Tray Efficiencies							
E_{MVi},	Bz	.639	.772	.750	.623	.772	.761
	Tol	.642	.775	.750	.584	.774	.761
	Xyl	.637	.705	.000	.643	.739	.000
E_{MLi},	Bz	.776	.790	.624	.768	.798	.642
	Tol	17.722	.819	.624	-2.212	.823	.642
	Xyl	.468	.447	.000	.477	.497	.000
E_i,	Bz	.857	.919	.912	.853	.929	.924
	Tol	.661	.827	.806	.505	.814	.813
	Xyl	.711	.747	.000	.714	.775	.000
E_H		.501	.539	.410	.451	.523	.425

TABLE XXIV

DATA AND RESULTS - RUN 314

Date: June 13, 1968

OH Pressure
34.80 psia

Sump Pressure
36.45 psia

	314-1				314-2			
Condenser Duty	118940 Btu/hr				119170 Btu/hr			
Vapor OH & Reflux, moles/hr	8.75				8.75			
	Vapor Comp.,		Reflux Comp.,		Vapor Comp.,		Reflux Comp.,	
Bz	.970		.971		.968		.970	
Tol	.030		.029		.032		.030	
Xyl	.000		.000		.000		.000	
Temp. °F	185.9		173.5		186.1		173.1	
Reboiler Duty	120000 Btu/hr				120000 Btu/hr			
V_{reb} & L_1, moles/hr	8.19				8.18			
	Vapor Comp.		Tray 1 Liq.,		Vapor Comp.,		Tray 1 Liq.,	
Bz	.026		.023		.022		.022	
Tol	.794		.787		.787		.788	
Xyl	.180		.189		.190		.190	
Temp. °F	251.8		246.3		251.8		246.3	

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		8.27	8.44	8.85	8.31	8.48	8.88
t_n, °F		238.4	214.4	192.3	239.1	216.3	193.6
$x_{n,i}$,	Bz	.068	.377	.806	.065	.349	.787
	Tol	.845	.614	.194	.850	.636	.213
	Xyl	.087	.014	.000	.085	.015	.000
L_{n+1}, moles/hr		8.30	8.57	8.95	8.34	8.62	8.96
t_{n+1}, °F		232.5	204.4	188.6	233.4	206.9	189.1
$x_{n+1,i}$,	Bz	.122	.549	.889	.116	.520	.873
	Tol	.823	.444	.113	.830	.473	.127
	Xyl	.055	.007	.000	.054	.007	.000
V_n, moles/hr		8.30	8.57	8.95	8.34	8.62	8.96
T_n, °F		238.0	212.2	191.2	238.0	212.2	191.2
$y_{n,i}$,	Bz	.143	.585	.911	.143	.585	.911
	Tol	.815	.410	.089	.815	.410	.089
	Xyl	.042	.005	.000	.042	.005	.000
V_{n-1}, moles/hr		8.27	8.44	8.85	8.31	8.48	8.88
T_{n-1}, °F		243.1	222.0	196.1	243.1	222.0	196.1
$y_{n-1,i}$,	Bz	.074	.401	.839	.074	.401	.839
	Tol	.850	.588	.161	.850	.588	.161
	Xyl	.076	.011	.000	.076	.011	.000
Murphree Equilibrium State							
T_n^*, °F		240.6	216.2	193.4	240.8	217.6	194.1
$y_{n,i}^*$,	Bz	.152	.591	.913	.145	.566	.903
	Tol	.810	.404	.087	.818	.429	.097
	Xyl	.088	.004	.000	.037	.004	.000
t_n^*, °F		243.1	218.8	195.3	243.2	220.3	196.5
$x_{n,i}^*$,	Bz	.053	.332	.761	.050	.308	.733
	Tol	.825	.645	.239	.832	.670	.267
	Xyl	.122	.022	.000	.118	.022	.000
Generalized Equilibrium State							
V_n^*, moles/hr		8.16	8.42	8.87	8.21	8.45	8.88
T_n^*, °F		241.76	217.36	193.99	241.92	218.86	194.95
$y_{n,i}^*$,	Bz	.133	.571	.905	.126	.544	.893
	Tol	.824	.424	.095	.831	.451	.107
	Xyl	.043	.005	.000	.042	.005	.000
L_n^*, moles/hr		8.42	8.59	8.93	8.44	8.64	8.96
t_n^*, °F		241.76	217.36	193.99	241.92	218.86	194.95
$y_{n,i}^*$,	Bz	.059	.353	.791	.056	.329	.768
	Tol	.843	.630	.209	.849	.655	.232
	Xyl	.098	.016	.000	.095	.016	.000
Tray Efficiencies							
E_{MV_i},	Bz	.636	.807	.777	.634	.786	.742
	Tol	.628	.810	.777	.607	.787	.742
	Xyl	.641	.738	.000	.652	.758	.000
E_{ML_i},	Bz	.773	.816	.650	.774	.804	.614
	Tol	.563	.843	.650	16.225	.827	.614
	Xyl	.473	.479	.000	.485	.516	.000
E_i,	Bz	.860	.936	.915	.858	.928	.885
	Tol	.584	.853	.829	.566	.833	.803
	Xyl	.710	.776	.000	.722	.793	.000
E_H		.461	.599	.495	.476	.579	.569

TABLE XXV

DATA AND RESULTS - RUN 315

Date: June 17, 1968

OH Pressure 34.52 psia

Sump Pressure 35.67 psia

315-1

Condenser Duty 146190 Btu/hr
Vapor OH & Reflux, moles/hr 9.95

Vapor Comp.,	Bz	.379	Reflux Comp.,	Bz	.442
	Tol	.621		Tol	.558
	Xyl	.000		Xyl	.000
Temp., °F		220.1	Temp., °F		201.4

Reboiler Duty 143500 Btu/hr
V_{reb} & L_1, moles/hr 9.80

Vapor Comp.,	Bz	.000	Tray 1 Comp.,	Bz	.000
	Tol	.605		Tol	.609
	Xyl	.395		Xyl	.391
Temp., °F		266.9	Temp., °F		259.2

315-2

Condenser Duty 145840 Btu/hr
Vapor OH & Reflux, moles/hr 9.95

Vapor Comp.,	Bz	.394	Reflux Comp.,	Bz	.442
	Tol	.606		Tol	.558
	Xyl	.000		Xyl	.000
Temp., °F		220.2	Temp., °F		200.9

Reboiler Duty 143500 Btu/hr
V_{reb} & L_1, moles/hr 9.78

Vapor Comp.,	Bz	.000	Tray 1 Comp.,	Bz	.000
	Tol	.606		Tol	.606
	Xyl	.394		Xyl	.394
Temp., °F		266.8	Temp., °F		259.1

		315-1			315-2		
Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		9.95	10.21	10.33	9.92	10.19	10.29
t_n, °F		250.5	241.0	233.8	250.9	241.3	233.3
$x_{n,i}$,	Bz	.000	.014	.089	.000	.012	.087
	Tol	.736	.918	.895	.721	.916	.898
	Xyl	.264	.068	.016	.279	.072	.015
L_{n+1}, moles/hr		10.06	10.28	10.30	10.01	10.23	10.23
t_{n+1}, °F		247.3	239.4	228.6	247.2	239.0	227.4
$x_{n+1,i}$	Bz	.003	.027	.148	.000	.026	.146
	Tol	.809	.931	.843	.802	.932	.845
	Xyl	.188	.041	.009	.198	.042	.009
V_n, moles/hr		10.06	10.28	10.30	10.01	10.23	10.23
T_n, °F		251.2	240.5	232.6	251.2	240.5	232.6
$y_{n,i}$,	Bz	.003	.028	.178	.003	.028	.178
	Tol	.840	.933	.814	.840	.933	.814
	Xyl	.157	.039	.008	.157	.039	.008
V_{n-1}, moles/hr		9.95	10.21	10.33	9.92	10.19	10.29
T_{n-1}, °F		255.7	243.0	235.0	255.7	243.0	235.0
$y_{n-1,i}$,	Bz	.000	.013	.092	.000	.013	.092
	Tol	.760	.922	.894	.760	.922	.894
	Xyl	.240	.065	.014	.240	.065	.014
Murphree Equilibrium State							
T_n^*, °F		252.3	242.7	234.0	252.9	243.0	234.1
$y_{n,i}^*$,	Bz	.000	.033	.187	.000	.027	.184
	Tol	.857	.936	.807	.848	.939	.810
	Xyl	.143	.032	.006	.152	.034	.006
t_n^*, °F		255.1	243.6	235.8	255.8	243.7	235.9
$x_{n,i}^*$,	Bz	.001	.011	.068	.000	.011	.067
	Tol	.666	.901	.910	.654	.899	.910
	Xyl	.333	.088	.022	.346	.090	.022
Generalized Equilibrium State							
V_n^*, moles/hr		9.95	10.16	10.22	9.87	10.11	10.13
T_n^*, °F		253.32	243.03	235.12	254.02	243.25	235.18
$y_{n,i}^*$,	Bz	.002	.029	.161	.000	.026	.159
	Tol	.838	.936	.831	.830	.937	.833
	Xyl	.160	.035	.007	.170	.036	.007
L_n^*, moles/hr		10.06	10.34	10.42	10.06	10.32	10.39
t_n^*, °F		253.32	243.03	235.12	254.02	243.25	235.18
$y_{n,i}^*$,	Bz	.001	.012	.076	.000	.011	.074
	Tol	.708	.913	.907	.694	.911	.908
	Xyl	.291	.074	.018	.306	.078	.017
Tray Efficiencies							
E_{MV_i},	Bz	.000	.715	.597	.000	.925	.608
	Tol	.599	.752	.587	.640	.664	.602
	Xyl	.627	.733	.699	.640	.770	.661
E_{ML_i},	Bz	1.574	.847	.741	.000	.960	.749
	Tol	.511	.438	.785	.547	.468	.805
	Xyl	.527	.577	.512	.547	.624	.465
E_i,	Bz	1.377	.904	.821	.000	.988	.830
	Tol	.804	1.482	.745	.842	1.454	.741
	Xyl	.711	.789	.768	.720	.818	.734
E_H		.297	.194	.668	.320	.339	.641

TABLE XXVI

DATA AND RESULTS - RUN 316

	316-1		316-2	
Date: June 18, 1968	Condenser Duty 128030 Btu/hr		Condenser Duty 127920 Btu/hr	
	Vapor OH & Reflux, moles/hr 8.78		Vapor OH & Reflux, moles/hr 8.70	
OH Pressure	Vapor Bz .412	Reflux Bz .434	Vapor Bz .417	Reflux Bz .432
35.40 psia	Comp., Tol .588	Comp., Tol .566	Comp., Tol .583	Comp., Tol .568
	Xyl .000	Xyl .000	Xyl .000	Xyl .000
Sump Pressure	Temp. °F 221.0	Temp. °F 201.1	Temp. °F 221.3	Temp. °F 201.0
36.50 psia				
	Reboiler Duty 128000 Btu/hr		Reboiler Duty 128000 Btu/hr	
	V_{reb} & L_1, moles/hr 8.61		V_{reb} & L_1, moles/hr 8.60	
	Vapor Bz .000	Tray 1 Bz .000	Vapor Bz .000	Tray 1 Bz .000
	Comp., Tol .597	Liq., Tol .602	Comp., Tol .595	Liq., Tol .602
	Xyl .403	Xyl .398	Xyl .405	Xyl .398
	Temp. °F 268.3	Temp. °F 261.5	Temp. °F 268.2	Temp. °F 261.5

Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		8.73	8.95	9.02	8.73	8.95	9.02
t_n, °F		252.4	242.8	235.4	252.7	242.9	235.5
$x_{n,i}$,	Bz	.000	.004	.067	.000	.009	.066
	Tol	.733	.916	.417	.735	.717	.918
	Xyl	.267	.075	.016	.265	.074	.016
L_{n+1}, moles/hr		8.82	8.99	8.18	8.80	8.98	9.00
t_{n+1}, °F		249.1	240.7	230.2	249.0	240.8	230.2
$x_{n+1,i}$,	Bz	.000	.018	.130	.000	.018	.127
	Tol	.808	.937	.961	.806	.938	.864
	Xyl	.192	.045	.009	.194	.044	.009
V_n, moles/hr		8.82	8.99	8.18	8.80	8.98	9.00
T_n, °F		253.0	242.3	234.2	253.0	242.3	234.2
$y_{n,i}$,	Bz	.003	.022	.159	.003	.022	.159
	Tol	.837	.940	.834	.837	.940	.834
	Xyl	.160	.038	.007	.160	.038	.007
V_{n-1}, moles/hr		8.73	8.95	9.02	8.73	8.95	9.02
T_{n-1}, °F		257.6	244.7	237.4	257.6	244.7	237.4
$y_{n-1,i}$,	Bz	.000	.011	.083	.000	.011	.083
	Tol	.755	.927	.904	.755	.927	.904
	Xyl	.245	.062	.013	.245	.062	.013
Murphree Equilibrium State							
T_n^*, °F		253.9	244.9	237.4	253.9	244.9	237.4
$y_{n,i}^*$,	Bz	.000	.021	.144	.000	.021	.143
	Tol	.856	.944	.849	.856	.944	.850
	Xyl	.144	.035	.007	.144	.035	.007
t_n^*, °F		257.0	245.7	244.8	257.1	245.7	238.2
$x_{n,i}^*$,	Bz	.000	.008	.054	.000	.008	.058
	Tol	.662	.898	.926	.659	.900	.921
	Xyl	.338	.094	.019	.341	.093	.021
Generalized Equilibrium State							
V_n^*, moles/hr		8.721	8.86	9.72	8.72	8.86	8.86
T_n^*, °F		255.13	245.23	238.38	255.16	245.22	237.86
$y_{n,i}^*$,	Bz	.000	.019	.121	.000	.019	.133
	Tol	.837	.942	.872	.837	.943	.860
	Xyl	.163	.038	.008	.163	.038	.007
L_n^*, moles/hr		8.82	9.08	7.48	8.82	9.07	9.15
t_n^*, °F		255.13	245.23	238.38	255.16	245.22	237.86
$y_{n,i}^*$,	Bz	.000	.008	.055	.000	.008	.061
	Tol	.705	.911	.927	.705	.912	.922
	Xyl	.295	.081	.018	.295	.080	.017
Tray Efficiencies							
E_{MV_i},	Bz	.000	.794	.787	.000	.772	.811
	Tol	.610	.745	.791	.582	.766	.659
	Xyl	.610	.760	.758	.582	.768	.757
E_{ML_i},	Bz	.000	.894	.879	.000	.882	.833
	Tol	.513	.535	.942	.484	.554	1.277
	Xyl	.513	.614	.581	.484	.625	.711
E_i,	Bz	.000	.942	.935	.000	.932	.780
	Tol	.801	1.488	.757	.769	1.465	-1.990
	Xyl	.699	.808	.806	.677	.815	1.010
E_H		.314	.262	.547	.370	.255	-.200

TABLE XXVII

DATA AND RESULTS - RUN 318

Date: June 20, 1968

OH Pressure
36.73 psia

Sump Pressure
37.83 psia

318-1

Condenser Duty 110000 Btu/hr
Vapor OH & Reflux, moles/hr 7.57

Vapor Comp.,	Bz	.476	Reflux Comp.,	Bz	.487
	Tol	.524		Tol	.513
	Xyl	.000		Xyl	.000
Temp., °F		220.9	Temp., °F		198.7

Reboiler Duty 110200 Btu/hr
V_{reb} & L_1, moles/hr 7.43

Vapor Comp.,	Bz	.000	Tray 1 Comp.,	Bz	.000
	Tol	.597		Tol	.604
	Xyl	.403		Xyl	.396
Temp., °F		269.7	Temp., °F		263.9

318-2

Condenser Duty 109830 Btu/hr
Vapor OH & Reflux, moles/hr 7.57

Vapor Comp.,	Bz	.487	Reflux Comp.,	Bz	.486
	Tol	.513		Tol	.514
	Xyl	.000		Xyl	.000
Temp., °F		221.2	Temp., °F		198.3

Reboiler Duty 110200 Btu/hr
V_{reb}, L_1, moles/hr 7.42

Vapor Comp.,	Bz	.000	Tray 1 Comp.,	Bz	.000
	Tol	.599		Tol	.604
	Xyl	.401		Xyl	.396
Temp., °F		269.9	Temp., °F		263.9

		318-1			318-2		
Measured Data		Tray 2	Tray 5	Tray 8	Tray 2	Tray 5	Tray 8
L_n, moles/hr		7.54	7.71	7.78	7.52	7.71	7.76
t_n, °F		255.4	245.2	237.4	255.5	245.4	237.3
$x_{n,i}$,	Bz	.000	.010	.074	.000	.010	.075
	Tol	.733	.914	.910	.728	.916	.909
	Xyl	.267	.076	.016	.272	.074	.016
L_{n+1}, moles/hr		7.58	7.75	7.78	7.58	7.74	7.77
t_{n+1}, °F		251.1	243.1	231.8	251.5	243.2	231.6
$x_{n+1,i}$,	Bz	.000	.020	.147	.000	.021	.148
	Tol	.799	.934	.844	.799	.934	.843
	Xyl	.201	.045	.009	.201	.045	.009
V_n, moles/hr		7.58	7.75	7.78	7.58	7.74	7.77
T_n, °F		255.0	244.1	235.4	255.0	244.1	235.4
$y_{n,i}$,	Bz	.003	.028	.187	.003	.028	.187
	Tol	.842	.937	.807	.842	.937	.807
	Xyl	.155	.035	.006	.155	.035	.006
V_{n-1}, moles/hr		7.54	7.71	7.78	7.52	7.71	7.76
T_{n-1}, °F		259.9	247.0	239.3	259.9	247.0	239.3
$y_{n-1,i}$,	Bz	.000	.013	.104	.000	.013	.104
	Tol	.763	.926	.884	.763	.926	.884
	Xyl	.237	.061	.011	.237	.061	.011
Murphree Equilibrium State							
T_n^*, °F		256.4	247.3	239.2	256.6	247.2	239.1
$y_{n,i}^*$,	Bz	.000	.023	.157	.000	.024	.160
	Tol	.855	.941	.836	.852	.941	.834
	Xyl	.145	.036	.007	.148	.035	.007
t_n^*, °F		260.0	248.1	239.8	260.0	248.1	239.8
$x_{n,i}^*$,	Bz	.000	.009	.069	.000	.009	.069
	Tol	.650	.897	.910	.650	.897	.911
	Xyl	.350	.094	.021	.350	.095	.020
Generalized Equilibrium State							
V_n^*, moles/hr		7.50	7.63	7.66	7.49	7.63	7.65
T_n^*, °F		257.80	247.59	239.52	257.92	247.53	239.44
$y_{n,i}^*$,	Bz	.000	.021	.151	.000	.022	.153
	Tol	.833	.940	.841	.831	.940	.840
	Xyl	.167	.039	.007	.169	.038	.007
L_n^*, moles/hr		7.62	7.83	7.90	7.61	7.82	7.89
t_n^*, °F		257.80	247.59	239.52	257.92	247.53	239.44
$y_{n,i}^*$,	Bz	.000	.009	.071	.000	.009	.072
	Tol	.700	.909	.911	.697	.910	.911
	Xyl	.300	.082	.018	.303	.081	.017
Tray Efficiencies							
E_{MV_i},	Bz	.000	.818	.882	.000	.747	.862
	Tol	.538	.741	.897	.570	.731	.873
	Xyl	.538	.766	.761	.570	.737	.771
E_{ML_i},	Bz	.000	.907	.934	.000	.866	.923
	Tol	.442	.533	1.001	.475	.495	.981
	Xyl	.442	.623	.580	.475	.585	.595
E_i,	Bz	.000	.952	.976	.000	.921	.968
	Tol	.731	1.563	.804	.767	1.498	.799
	Xyl	.639	.812	.806	.665	.790	.815
E_H		.420	.241	.550	.374	.261	.560

APPENDIX F

ERROR ANALYSIS

ERROR ANALYSIS

Davies (10) presents a method for estimating the standard error of a general function of variables given the standard error of the variables. For a general function $X = (x_1, x_2, \cdots x_n)$ the estimate of the variance of X is given by

$$\mathrm{var}(X) = \left(\frac{\partial X}{\partial x_1}\right)^2 \mathrm{var}(x_1) + \left(\frac{\partial X}{\partial x_2}\right)^2 \mathrm{var}(x_2) + \cdots$$
$$+ \left(\frac{\partial X}{\partial x_1}\right)\left(\frac{\partial X}{\partial x_2}\right) \text{covariance}(x_1\, x_2) + \cdots$$

The covariance terms are zero if the variables $(x_1, x_2 \cdots)$ are independent measurements.

Applying this analysis to a Murphree vapor efficiency calculation, the estimate of the standard error of the efficiency is

$$\mathrm{var}(E_{MV_n}) = \left(\frac{\partial E_{MV_n}}{\partial y_n}\right)^2 \mathrm{var}(y_n) + \left(\frac{\partial E_{MV_n}}{\partial y_{n-1}}\right)^2 \mathrm{var}(y_{n-1})$$
$$+ \left(\frac{\partial E_{MV_n}}{\partial y_n^*}\right)^2 \mathrm{var}(y_n^*)$$

For evaluation, the variance of the measured compositions was assumed to be 1.0×10^{-6} which corresponds to a standard deviation of 0.001. The variance of the equilibrium vapor composition, y_n^*, was assumed to be the same because it is calculated from the measured liquid composition. Table XXVIII summarizes the results of four evaluations at various conditions.

Table XXVIII

Estimates of Variance of Murphree Vapor Efficiencies

Component Run and Tray No.	y_n	y_{n-1}	y_n*	E_{MV_n}	Variance for E_{MV_n} $\times 10^{-4}$	Standard Deviation
Benzene 305-1, 8	.496	.324	.527	.809	1.0	.010
Benzene 303-1, 5	.042	.020	.046	.879	74.3	.085
Benzene 306-1, 2	.012	.006	.015	.637	419.0	.175
Toluene 307-1, 2	.906	.839	.916	.646	55.4	.074

The major factor in estimating the error for the calculated efficiencies is the difference in composition or change in composition across the tray. The absolute value of the composition has little effect on the estimated efficiency error.

APPENDIX G

VAPOR AND LIQUID TEMPERATURE MEASUREMENTS

VAPOR AND LIQUID PHASE TEMPERATURE MEASUREMENTS

The experimental data for this study indicates uncertainty concerning the ability to measure vapor phase temperatures in the distillation column. Similar results have been experienced by other investigators with experimental distillation equipment as mentioned in Chapter 3.

Because of the suspected effects of condensation on the thermocouple tip, the following experiment was performed. The total refluxed still employed in the thermocouple calibrations (Appendix B) was used. This equipment was operated with pure benzene and then with a mixture of benzene and toluene (approximately 50 per cent benzene). For both the pure component and the mixture, the thermocouple EMF was measured with the thermocouple tip in the liquid phase and then in the vapor phase. The results are given in the table following. The time between measurements was about 15 minutes.

TABLE XXIX

VAPOR AND LIQUID PHASE TEMPERATURE MEASUREMENTS

	Temperature, °F		
	Time 1	Time 2	Time 3
Benzene			
Vapor Phase	174.4	174.4	174.4
Liquid Phase	174.4	174.4	174.4
Benzene-Toluene			
Vapor Phase	183.1	183.1	183.1
Liquid Phase	187.8	187.8	187.8

The measured temperature of the "equilibrium" vapor phase for the benzene-toluene mixture was 4.7°F. less than that for the liquid phase.

During these measurements, the heat applied to the still was varied to assure that superheating was not affecting the results.

APPENDIX H

LIQUID SAMPLING TEST

LIQUID SAMPLING TEST

A brief study was made (5) to determine the accuracy of the liquid sampling procedure using an evacuated sample bomb. A standard solution of benzene and toluene was prepared by weight. This standard was analyzed in triplicate. The evacuated sample bomb was used to take a sample from the standard. The bomb was then cooled in ice, vented, and a sample withdrawn in the same manner used for the samples from the column. This sample was analyzed in triplicate. The results of these analyses are shown in the following table.

TABLE XXX

LIQUID SAMPLING TEST RESULTS

	Compositions, mole fraction Bz	
Analysis	Standard	Sample
1	0.5508	0.5499
2	0.5504	0.5508
3	0.5500	0.5494
average	0.5504	0.5500
Maximum Error		
(+)	0.0004	0.0008
(-)	0.0004	0.0006

These data show that the difference between the average of the three analyses of the standard and the average of the three analyses of the sample was 0.0004 mole fraction benzene.

The chromatograph calibration gave a standard deviation of 0.0005 mole fraction. Therefore, this study shows that the sampling procedure does not limit the accuracy of the experimental results.

VITA 3

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Doctor of Philosophy

Thesis: DISTILLATION TRAY EFFICIENCIES - AN EXPERIMENTAL STUDY WITH A TERNARY SYSTEM

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