

Copyright Warning & Restrictions

The copyright law of the United States (Title 17, United States Code) governs the making of photocopies or other reproductions of copyrighted material.

Under certain conditions specified in the law, libraries and archives are authorized to furnish a photocopy or other reproduction. One of these specified conditions is that the photocopy or reproduction is not to be “used for any purpose other than private study, scholarship, or research.” If a user makes a request for, or later uses, a photocopy or reproduction for purposes in excess of “fair use” that user may be liable for copyright infringement,

This institution reserves the right to refuse to accept a copying order if, in its judgment, fulfillment of the order would involve violation of copyright law.

Please Note: The author retains the copyright while the New Jersey Institute of Technology reserves the right to distribute this thesis or dissertation

Printing note: If you do not wish to print this page, then select “Pages from: first page # to: last page #” on the print dialog screen

The Van Houten library has removed some of the personal information and all signatures from the approval page and biographical sketches of theses and dissertations in order to protect the identity of NJIT graduates and faculty.

THE COUNTERCURRENT MULTISTAGE DESORPTION
OF THE ACETIC ACID/WATER SOLUTION WITH AN INERT VAPOR
BY
MICHAEL JOHN DANKO

A THESIS
PRESENTED IN PARTIAL FULFILLMENT OF
THE REQUIREMENTS FOR THE DEGREE
OF
MASTER OF SCIENCE IN CHEMICAL ENGINEERING
AT
NEW JERSEY INSTITUTE OF TECHNOLOGY
(FORMERLY NEWARK COLLEGE OF ENGINEERING)

This thesis is to be used only with due regard to the rights of the author. Bibliographical references may be noted, but passages must not be copied without permission of the Institute and without credit being given in subsequent written or published work.

Newark, New Jersey
1978

ABSTRACT

The desorption of a acetic acid and water solution with an inert gas was experimentally investigated using a perforated plate distillation column having 17 actual trays. An inert air stream flowing countercurrent was used to strip the water from the acetic acid and water solution.

The feed composition was 0.2509 mole fraction acetic acid. The experimental results showed the best obtainable separation to be 0.16118 and 0.3965 mole fraction acetic acid in the overhead condensate and bottoms respectively, at an average column operating temperature of 56.0 degrees centigrade. The concentration of acetic acid predicted by calculation is 0.003917 and 0.9860 mole fraction in the overhead condensate and bottoms respectively, at an average column operating temperature of 108.9 degrees centigrade.

It was found that inadequate temperature inhibited the activity of the system, limiting the amount of heat available as Heat of Vaporization, and therefore caused much larger amount of water to remain in solution than calculations indicate. This insufficient heat produced results less promising than those obtained by mathematical simulation.

APPROVAL OF THESIS
THE COUNTERCURRENT MULTISTAGE DESORPTION
OF THE ACETIC ACID/WATER SOLUTION WITH AN INERT VAPOR

BY

MICHAEL JOHN DANKO

FOR

DEPARTMENT OF CHEMICAL ENGINEERING
NEW JERSEY INSTITUTE OF TECHNOLOGY
(FORMERLY NEWARK COLLEGE OF ENGINEERING)

BY

FACULTY COMMITTEE

APPROVED:

NEWARK, NEW JERSEY

MAY, 1978

ACKNOWLEDGEMENT

The author wishes to express his deep appreciation for the patience and guidance given by Dr. John E. McCormick throughout the entire course of this work.

TABLE OF CONTENTS

	<u>Page Number</u>
Abstract	i
Acknowledgement	ii
List of Figures	v
List of Tables	vi
Introduction	1
Theoretical Development	
The development of the Analytical Equation and Calculation Technique	7
Applications in Absorption and Stripping	13
The method of Horton and Franklin	15
The Edmister Method for estimating an effective stripping factor	17
The development of the analytical method for calculating pH	19
Experimental Apparatus	21
Experimental operation of column and related equipment . .	25
Technique for titration and volumetric analysis	29
Experimental results	31
Data analysis: Calculation of apparent column efficiency .	33
Discussion of results	36
Conclusions	
Qualitative analysis	41
Recommendations	46

TABLE OF CONTENTS (Cont'd)

	<u>Page Number</u>
Appendix	
Derivation of the Horton-Franklin equation for predicting bottoms recovery	56
Derivation of the effective stripping factor - The Method of Edmister	66
Sample calculation: System parameters	68
Sample calculation: Volumetric analysis	77
Sample calculation: Feed sample pH	80
Sample calculation: Apparent column efficiency. .	81
Nomenclature	84
References	87

LIST OF FIGURES

		<u>Page Number</u>
Figure 1	Equilibrium stage extraction process - Two feeds and reflux at both ends	8
Figure 2	Equilibrium stage process - One feed, no refluxes	8
Figure 3	Process flow diagram	52
Figure 4	Molar ratio equilibrium diagram	53
Figure 5	Flooding Capacity diagram, sieve trays	54
Figure 6	Weeping diagram, sieve trays	55

LIST OF TABLES

		<u>Page Number</u>
Table 1	Tower specifications	48
Table 2	Summary of experimental results	32
Table 3	Summary of experimental data: Bottoms liquid	49
Table 4	Summary of experimental data: Condensed overhead vapor	50
Table 5	Vapor-Liquid equilibrium data, Water and Acetic acid	51

INTRODUCTION

Acetic acid (CH_3COOH) is presently produced in the United States at the rate of 2.5 billion pounds per year. It is rated within the top 20 industrial chemicals in annual rate of production increase, averaging approximately 8 percent per year for the past 10 years. In spite of the feedstock shortages experienced by many industrial users during the 1973 period, acetic acid production enjoyed a $12\frac{1}{2}$ percent production increase, more than three times the average for the top 50 industrial chemicals for the same time period.

The principle uses of acetic acid are for the production of vinyl acetate and acetic anhydride. Vinyl acetate has found a considerable market in the areas of latex paints, plastics, and adhesives; areas which have grown rapidly in recent years. The acetic anhydride market has experienced more moderate growth; its principle uses being in the areas of rayon and cellulose acetate.

The industrial synthesis of acetic acid is mainly by direct oxidation of paraffin hydrocarbons, primarily n-butane, by oxidation of acetaldehyde, by the reaction of methanol and carbon monoxide, and by processes involving recovery of by-product acid. These four routes present the largest share of the industrial market. The latest commercial process for acetic acid production is by methanol carbonylation; where the raw materials can be obtained directly or synthesized from fossil fuels.

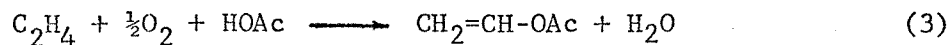
Approximately 93% of the products obtained from processes involving acetic acid production are acetate esters. Esterification of alcohols or olefins with acetic acid, for both gas and liquid phase reactions, are catalyzed by strong mineral acids, including acetic aluminas. Since acids may lead to undesirable by-products, bases could be used, although the rate of reaction is much less. Another problem with base catalyzation is that the conversion is limited by the approach to esterification equilibrium:



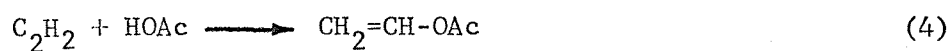
. . . where R denotes the hydrocarbon radical, such as CH_3- , C_6H_5- , etc. In some cases, the equilibrium limitation is relieved by removal of the water or ester by azeotropic distillation, or by first dehydrating the acetic acid to acetic anhydride.

The hydrolysis of esters, the reverse of reaction (1), is of significant commercial importance in producing large amounts of recycle diluted acetic acid. This is prominent in the rayon industry, where production from cellulose acetate produces recycle acid, which is concentrated for reuse.

Vinyl acetate is the largest end product obtained from acetic acid. The two major processes involve the principle of acetic acid-olefin addition. The latest commercial processes involve the use of ethylene and oxygen:

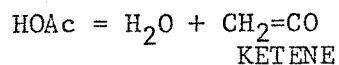


The more traditional esterification of acetylene to vinyl acetate is:

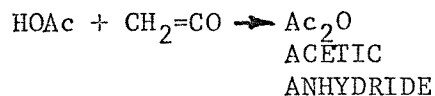


Both of these reactions apparently do not exhibit an equilibrium limitation. The resistance of acetic acid to destructive oxidation allows better than a 95% yield in ethylene based processes, where the by-products derive mainly from ethylene.

Dehydration is the second most popular industrial reaction involving acetic acid, being an important step in the synthesis of cellulose acetate and rayon. Esterification with acetic anhydride allows a much higher conversion of alcohols (or -OH groups) than esterification with acetic acid (reaction 1). The dehydration equilibrium restriction is predominant, requiring a reaction temperature and pressure of 700°C and 150 mm absolute. The commercial reaction is:



. . . which is catalyzed by small amounts of triethylphosphate, and followed (after quenching and cooling) by:



The acetic anhydride is present in the liquid phase.

As reactions (1) and (3) indicate, water is a co-product in reactions involving acetic acid. Both major users (acetate production via esterification and dehydration) produce water that must be removed in order to insure product purity.

All three of the predominant manufacturing processes involving acetic acid production rely on azeotropic distillation as a means of water removal. In the process involving direct oxidation of hydrocarbons, the acetic acid solution, with co-products alcohols, aldehydes, ketones, esters, acids, and water is purified using azeotropic distillation. Esters refluxed back to the separation column enhances the volatility of the water. The acetic acid is removed from the column bottoms; the azeotropic effluent is sent to an overhead separator.

In the process of acetaldehyde oxidation, water and other light ends are removed from acetic acid in a drying column. For this process, the azeotroping agent is commonly an ether, such as diisopropyl ether, which greatly increases the water volatility, along with entraining any light ends in solution. The process water is then stripped of any hydrocarbons and sent to a vent gas scrubber, or discarded.

The process involving acetic acid production by methanol carbonylation utilizes the conventional dehydration schemes with the exception that, in low pressure synthesis, water soluble methyl

acetate is used as the azeotroping agent. Following removal from the dehydration unit and separation from any organics, the water is recycled to a vent scrubber or discarded. Also, since reactions involving the production of esters from acetic acid are limited by hydrolysis equilibrium, acetic acid is frequently found in dilute quantities as an impure by-product. Here again, the water stream used in production is mixed with an azeotroping agent (esters, ketones, and ethers) and withdrawn.

This Thesis explores an alternate to azeotropic distillation, namely, the removal of the water by stripping with an inert vapor. Azeotropic distillation involves the addition of a third chemical used to entrain the water; an inert vapor such as air would probably be less expensive than the azeotroping agent, and more readily available. The process equipment required would basically remain the same; the overhead separation unit could be replaced with a condenser, or the stream sent directly to the vent scrubber and condensed with the wash water. This "once-through" type of operation, if successful, could prove more economical than the traditional azeotropic distillation process.

The analytical methods used to predict tower performance are the traditional Horton-Franklin equations combined with the Edmister equations for Stripping factors. This presents a complete method for calculating bottoms recovery.

THEORETICAL DEVELOPMENT

THE DEVELOPMENT OF THE ANALYTICAL EQUATIONS
AND CALCULATION TECHNIQUE

The calculation methods presented here were derived by Smith and Brinkley (1) which are applicable to a wide variety of equilibrium stage processes. Basically, the analytical method predicts the recovery of each component in the bottoms product. Originally developed for a countercurrent extraction process (see Figure 1) it has found use in many simpler equilibrium processes. Many of the stream variables and flowrates reduce to either zero or 1.0, thereby allowing for a relatively straightforward iterative type of calculation procedure.

The complete derivation of the equations used in predicting the bottoms recovery is presented in the Appendix. For clarity, the derivation will only be outlined here.

Referring again to Figure 1 and writing a material balance around stage (n+1), the following equation is obtained:

$$L_{n+2}x_{n+2} + V_n y_n = L_{n+1}x_{n+1} + V_{n+1}y_{n+1}$$

By definition, $y_n = K_n x_n$, and $y_{n+1} = K_{n+1} x_{n+1}$; substitution into the preceding equation yields:

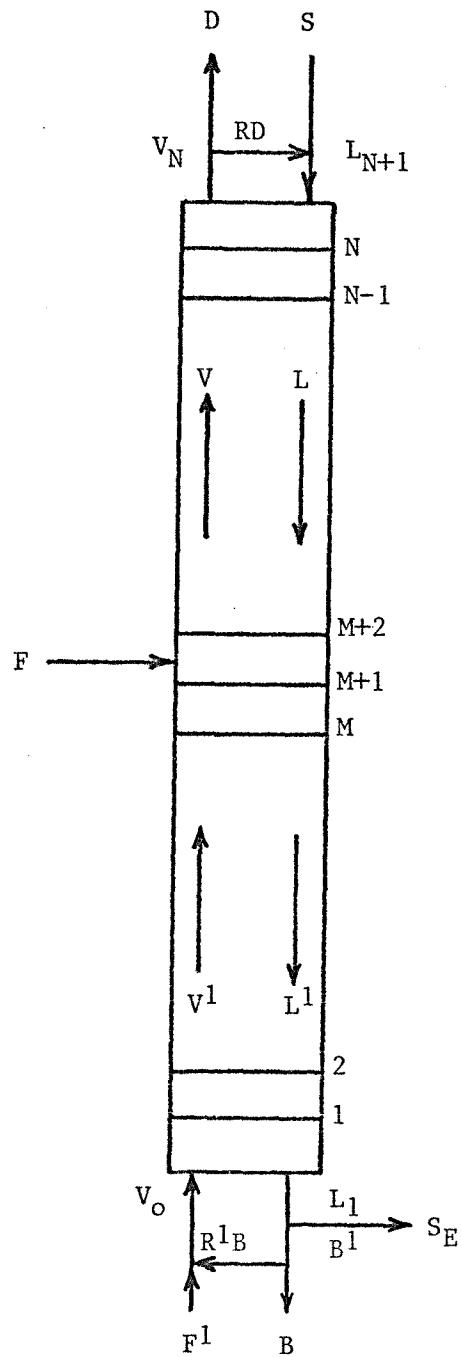
$$L_{n+2}x_{n+2} + V_n K_n x_n = L_{n+1}x_{n+1} + V_{n+1} K_{n+1} x_{n+1}$$

Rearranging in terms of the x variable gives:

$$x_{n+2} - \left[\frac{K_{n+1}V_{n+1} + L_{n+1}}{L_{n+2}} \right] x_{n+1} + \frac{K_n V_n}{L_{n+2}} x_n = 0$$

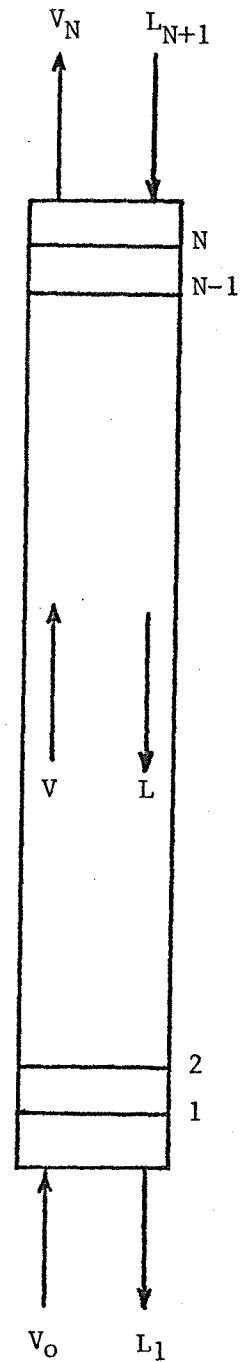
. . . which applies to any component under consideration.

FIGURE 1



Equilibrium Stage
Extraction Process
 Two Feeds and Reflux
 at both ends.

FIGURE 2



Equilibrium Stage
Process
 One Feed, no Refluxes

This equation can be solved by making the simplifying assumption of constant phase rates and distribution coefficients in the upper portion of the column. Incorporating this into the above equation and presenting in the differential "operator" form yields:

$$\left(E^2 - \frac{KV}{L} + 1\right) E + \frac{KV}{L} x_n = 0$$

. . . which has the roots KV/L and 1.0 . In terms of the Stripping factor, the solution can be written as:

$$x_n = C_1(S_n)^n + C_2 \quad (1)$$

. . . where C_1 and C_2 are constants and $S_n = KV/L =$ the average stripping factor for the component in the upper section of the column. The variable x_n relates to the concentration of the component in the heavy phase leaving stage n , and the exponent n on the stripping factor denotes the stage number.

In like manner, an equation can be derived for the lower section of the column.

$$x_m = C_3(S_m)^m + C_4 \quad (2)$$

. . . where $S_m = K^1V^1/L^1 =$ the average stripping factor in the lower section of the column. The "m" denotes the stage number in the lower section.

In order to obtain a working equation in x , the constants C_1 , C_2 , C_3 , and C_4 must be eliminated. For the upper section of the column (equation (1)), let A denote the total amount of a given component entering the column. By definition:

$$A = Fy_F + F^1y_{F1} + Sx_S$$

. . . where the y values denote the feed concentration (shown as y because in an extraction process, the feed is generally more similar to the raffinate phase). If f is designated as the fraction of the component which will be recovered in the bottoms, the equation:

$$Dy_N = (1-f)A$$

. . . can be written. Substituting $(1-f)A$ for Dy_N in the material balance equation:

$$V_N y_N = Dy_N + RDy_N = (1+R)Dy_N$$

. . . allows for a solution in terms of y_N , which upon algebraic manipulation yields:

$$y_N = \frac{(1+R)(1-f)A}{V_N}$$

. . . or, noting that $K = y_N/x_N$:

$$x_N = \frac{(1+R)(1-f)A}{KV_N}$$

Direct substitution into equation (1) yields:

$$\frac{(1+R)(1-f)A}{KV} = C_1 (S_n)^N + C_2$$

A similar equation with S_n raised to the $(N-1)$ power can be derived from a material balance around stage N . These two equations can then be combined to eliminate the constants C_1 and C_2 . An analogous procedure will eliminate the constants C_3 and C_4 from equation (2), and provide a working equation for the lower section of the column. The equations for the upper and lower sections of the column can be combined with a component balance around the feed stage to provide an equation depicting the entire process shown in Figure 1, which is:

$$f = \frac{(1-S_n^{N-M}) + q_s(S_n^{N-M}-S_n) + R(1-S_n) + hq_{F1}S_n^{N-M}(1-S_m^M)}{\left[(1-S_n^{N-M}) + hS_n^{N-M}(1-S_m^N) + R(1-S_n) + h \left(\frac{(1+R^1)}{(1+gR^1)} \right) S_m^M S_n^{N-M}(1-S_m) \right]} \quad (3)$$

The variables q_F and q_{F1} refer to the fraction of the component under consideration entering in either the upper or lower feed. R and R^1 denote the respective upper and lower reflux ratios, and the "g" variable, used strictly for an extraction process, denotes the assumed recovery factor in the solvent-recovery device. The variable h that is used depends upon the nature of the feed; for a light (vapor) feed (raffinate phase for extraction):

$$h = \frac{L}{L^1} \frac{1 - S_n}{1 - S_m} \quad (4)$$

For a more heavy, or liquid feed (extract phase for extraction):

$$h = \frac{K^1}{K} \frac{L}{L^1} \frac{1 - S_n}{1 - S_m} \quad (5)$$

Equations (4) and (5) used in equation (3) predicts the bottoms recovery for a distillation process.

Equation (3) is presented here in its most complicated form, being derived for the extraction process presented in Figure 1. For the inert gas desorption process under study, equation (3) degenerates to a much simpler form.

Referring to Figure (2), for a countercurrent desorption process, there are no refluxes ($R=R^1=0$), and no absorption section ($M=0$). Since the reflux ratio $R=0$, there is no lower feed other than V_0 , hence $F^1 = q_{F1} = 0$. Since for the purposes of this analysis, the water and

acetic acid enter only in stream L_{n+1} , $q_s = 1$; and by substitution of these quantities into equation (3), a much simpler form is developed:

$$f = \frac{1 - S}{1 - S^{N+1}} \quad (6)$$

The following section will illustrate how equation (6) is applied to the Stripping operation.

APPLICATIONS IN ABSORPTION AND STRIPPING

Both absorption and stripping (desorption) will be discussed here because they are essentially identical "reverse" operations. Mass transfer for both operations is essentially in one direction. The changes in phase rates are therefore proportional to the amount of material being transferred. In absorption, the relatively insoluble vapor phase (either fully or partially saturated) loses material as it proceeds up the column, but little if any nonvolatile absorber oil passes into the gas phase. The direction of mass transfer is reversed in stripping. The insoluble oil-free gas absorbs material as it proceeds up the column (the oil phase being "stripped"), and the oil phase decreases proportionately to the increase in vapor phase. It is evident that a constant L/V ratio does not apply to either absorption or stripping.

Aside from its effect on phase rates, the unidirectional transfer of material creates thermal effects which may be considered. In absorption, most of the heat released (as the vapor phase components condense) appears as sensible heat in the liquid phase. Since only a small amount of material is being vaporized, little of this heat is used as heat of vaporization. Hence, the liquid phase rises in temperature as it proceeds down the column. The opposite effect occurs in stripping. The heat of vaporization of the more volatile components is supplied by the sensible-heat contained in the liquid phase. Hence the temperature of the liquid phase decreases as it descends down the column.

Since the unidirectional flow of material in either absorption or stripping has such a pronounced non-linear effect on both phase rates and temperature profile, graphical solutions do not predict operation with sufficient accuracy. The variation in phase rates causes the operating-line equation:

$$y_n = \frac{L_{n+1}}{V_n} x_{n+1} + \frac{V_o y_o - L_1 x_1}{V_n}$$

to be non-linear, since the L/V ratio is no longer constant. The only exception would be the case of extremely dilute solutions, where both the operating and equilibrium curves drawn on an x-y diagram become linear as they approach the origin. For all other cases, equation (3) in its proper form must be used to predict the bottoms recovery for each individual component. For desorption, the method of Horton and Franklin can be used to predict changes in phase rates and temperature profile, using the method of Edmister to predict an overall Stripping factor for each individual component.

THE METHOD OF HORTON AND FRANKLIN

Horton and Franklin suggests that the percentage of material either absorbed or desorbed is constant throughout the column and the temperature change is proportional to the amount of material being transferred. Mathematically, these relationships can be expressed as:

$$\left[\frac{L_1}{L_{N+1}} \right]^{1/N} = \frac{L_n}{L_{n+1}} \quad (7)$$

. . . for the liquid phase rates, and:

$$\frac{L_{N+1} - L_n}{L_{N+1} - L_1} = \frac{t_{N+1} - t_n}{t_{N+1} - t_1} \quad (8)$$

. . . for the temperature profile. The Horton and Franklin method for predicting internal phase rates is as follows: an initial separation (f) is assumed for each component present in the feed. By performing a material balance around the entire column, the overhead vapor stream (V_N) and bottoms product stream (L_1) can be determined.

Having determined L_1 , and knowing L_{N+1} (feed), the ratio (L_1/L_{N+1}) can be determined. Using equation (7), setting $n = 1$, L_2 can be determined. By material balances, the difference in moles ($L_2 - L_1$) will be reflected as an increase in vapor rate ($V_0 + (L_2 - L_1)$), and thereby fix V_1 . The iteration is repeated setting $n=2$, and proceeding up the column until $n=N$. Equation (8) can be used simultaneously with equation (7), as the phase rates are determined, stage temperatures can be calculated. This would also require an estimation of t_{N+1} .

These equations are used in conjunction with the Edmister method for estimating the overall Stripping factor (described in the next section), for calculating the fraction of each component present in the bottoms product. This is accomplished by the application of equations (3) or (6). The material balance equations incorporating the f factor can be expressed as:

$$L_1 x_1 = f(V_0 y_0 + L_{N+1} x_{N+1}) \quad (9)$$

and

$$V_N y_N = (1-f)(V_0 y_0 + L_{N+1} x_{N+1}) \quad (10)$$

The subscripts on equations (9) and (10) refer to the stage number. The recovery calculated by equations (9) and (10) is compared to the initial assumption, and the iteration is repeated if not sufficiently close.

THE EDMISTER METHOD FOR ESTIMATING AN EFFECTIVE
STRIPPING FACTOR

The Edmister method for predicting an effective stripping factor initiates with the Horton and Franklin expression for an N-stage stripper (with stages numbered from bottom to top). This expression is:

$$\frac{X_{N+1} - X_1}{X_{N+1}} = \frac{S_1 S_2 \cdots S_N + S_2 S_3 \cdots S_N + \cdots + S_N}{S_1 S_2 \cdots S_N + S_2 S_3 \cdots S_N + \cdots + S_N + 1} - \frac{V_0 Y_0}{L_{N+1} X_{N+1}} \frac{S_2 S_3 \cdots S_N + S_3 S_4 \cdots S_N + \cdots + S_N + 1}{S_1 S_2 \cdots S_N + S_2 S_3 \cdots S_N + \cdots + S_N + 1} \quad (11)$$

By definition, an "effective" stripping factor is that value which if substituted for each S_n in equation (11) would leave the value of $(X_{N+1} - X_1)/(X_{N+1})$ unchanged.

It can be shown that the rigorous expression for S (equation 11), when applied to a two-stage stripper, will yield the quadratic equation:

$$S^2 + S - S_2(S_1 + 1) = 0$$

. . . where the unsubscripted values of S refer to the effective stripping factor, the subscripted values refer to their respective stage. (A complete derivation of the above equation appears in the Appendix). This equation has the positive solution:

$$S = (S_2(S_1 + 1) + 0.25)^{0.5} - 0.5$$

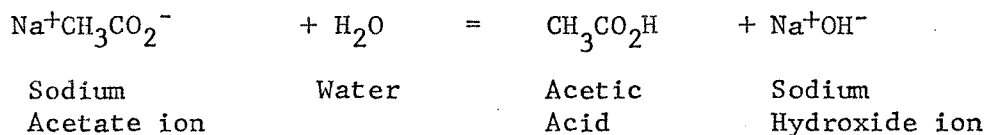
Edmister suggests that the effective stripping factor is to be considered independent of the number of stages; or expressed as the form:

$$S = (S_N(S_1 + 1) + 0.25)^{0.5} - 0.5$$

Calculation of effective stripping factors by the Edmister method will provide results that are generally comparable to those obtained by the method proposed by Horton and Franklin, which requires much more judgement and experience in selecting these factors. The Edmister method provides a relatively straightforward method of calculation, which when used in conjunction with equation (6), will allow for the calculation of the bottoms composition, considering only the top and bottom stages. The final calculation trial, which outlines the above method, appears in the Appendix.

THE DEVELOPMENT OF THE ANALYTICAL
METHOD FOR CALCULATING pH

The equilibrium expression pertinent to the titration of an acetic acid solution with sodium hydroxide is:



As with any weak base, the equilibrium-constant expression for sodium acetate can be written as:

$$K_B = (C_{\text{CH}_3\text{CO}_2\text{H}})(C_{\text{CH}_3\text{CO}_2^-})$$

. . . where (C_x) denotes the equilibrium concentration of component x.

The purpose here is to determine C_{OH^-} , hence enabling the pH to be calculated. Since the value of K_B was not readily available, an alternate approach to obtaining it was investigated.

The equilibrium-constants for water and acetic acid can be expressed as:

$$K_W = (C_{\text{H}^+})(C_{\text{OH}^-})$$

. . . for water, and:

$$K_A = (C_{\text{CH}_3\text{CO}_2^-})(C_{\text{H}^+}) / (C_{\text{CH}_3\text{CO}_2\text{H}})$$

for acetic acid. Division of (K_W/K_A) yields the expression:

$$\frac{K_W}{K_A} = \frac{(C_{\text{H}^+})(C_{\text{OH}^-})}{(C_{\text{CH}_3\text{CO}_2^-})(C_{\text{H}^+})} = \frac{(C_{\text{CH}_3\text{CO}_2\text{H}})(C_{\text{OH}^-})}{(C_{\text{CH}_3\text{CO}_2^-})}$$

. . . which is exactly identical to the expression for K_B . Hence, K_B can be calculated by dividing the known values of the equilibrium-constants for water and acetic acid, or:

$$K_B = \frac{1.0 \times 10^{-14}}{1.8 \times 10^{-5}} = 5.6 \times 10^{-10}$$

From the stoichiometry of the equilibrium equation, it can be seen that:

$$C_{\text{CH}_3\text{CO}_2\text{H}} = C_{\text{OH}^-}$$

By substituting back into the original expression for K_B :

$$K_B = \frac{(C_{\text{OH}^-})(C_{\text{OH}^-})}{(C_{\text{CH}_3\text{CO}_2^-})} = \frac{(C_{\text{OH}^-})^2}{(C_{\text{CH}_3\text{CO}_2^-})}$$

Since K_B and $C_{\text{CH}_3\text{CO}_2^-}$ are known, ($C_{\text{CH}_3\text{CO}_2^-}$, or the molarity of sodium acetate, is found by sample titration), the value of C_{OH^-} can be determined by re-arrangement of the above equation to:

$$C_{\text{OH}^-} = (K_B (C_{\text{CH}_3\text{CO}_2^-}))^{0.5}$$

This value is used in calculating the pH value. Further discussion and sample calculations are found in the Appendix.

EXPERIMENTAL APPARATUS

A process flow diagram for the desorption system is shown in Figure 3. The apparatus (hardware) required to construct this system is described in the following sections.

Liquid feed to Column. This system consists of a storage vessel, feed pump, and 0.25 inch diameter flexible copper tubing. The pump is also equipped with a recycle line to the storage vessel, with the necessary valving to regulate the flow to the tower as the liquid head in the storage vessel is decreased. The recycle stream also served to provide a degree of continuous mixing in the storage vessel.

The liquid feed system also consists of three feed preheaters. The first preheater consists of a 5 pass coiled heater, (2 feet of 0.25 inch copper tubing per pass) heated by two gas burners. The second and third feed preheaters each consist of 0.25 inch copper tubing spiral wound around a 5 inch outer diameter asbestos pipe, approximately 12 inches long. The heating medium is electrical heating tape, and each heater is independently controlled by a voltage transformer (Variac). The second and third preheaters, as well as the interconnecting tubing and the tubing from heater number three to the tower, was insulated with blanket and/or pipe insulation. A rotameter is inserted between the first feed preheater and the pump.

An inlet feed temperature indicator is inserted between the third preheater and the column by means of a tee connection. A thermometer is used as the indicator, exposed to the process fluid by means of the tee.

Liquid removal from the Column - Bottoms product. This circuit consists of 0.25 inch copper tubing connected to the laboratory drain. A bottoms sample tap is inserted in the line between the column and the drain by means of a tee. Within this circuit is also the necessary valving needed to facilitate startup, and to maintain sufficient liquid inventory in the column during operation and while taking samples.

Vapor feed to Column. This circuit consists of a rotameter, and an in-line gas drier consisting of 0.017 ft.³ of desiccant. Heating is accomplished by means of a feed gas preheater. This preheater, similar to the second and third liquid feed preheaters, consists of 0.375 inch copper tubing spiral wound around a 5 inch outer diameter asbestos pipe approximately 12 inches long.

The circuit also consists of a pressure and temperature indicator connected in series between the preheater and tower connection. A pressure guage serves as the PI, connected in-line by means of a tee, and the temperature indicator consists of a thermometer exposed to the process fluid by means of an additional tee connected in series with the PI tee. The feed preheater is wrapped with blanket insulation, and the line between the preheater and the vessel is insulated with asbestos pipe insulation.

Overhead vapor-condensate recovery system. A pressure indicator used to monitor the tower pressure above the top stage is connected by means of a glass tee. The overhead circuit consists of two vertical water-cooled glass condensers connected in series. The water-side of these condensers is also connected in series. Water flow is regulated to insure total condensation.

Directly beneath the condensers is a valved condensate trap, which served as the overhead condensate sample point. An in-line drain is installed after the trap to recover any condensate that by-passes the trap. The vapor outlet line is then connected from the vapor-side of the drain to the suction on the laboratory hood.

The overhead vapor line from the tower to the condensers, both condensers, and the valved condensate trap were all constructed of glass. The reason is that acetic acid present in the vapor phase will attack the copper tubing, causing contamination of the process fluid.

Desorption (Stripping) Column. The stripping section of the column consists of 19 radial-flow sieve trays; each tray having centrally located circular downcomers and overflow weirs. During the experiment, the lower two trays failed to start-up, hence for the purposes of this analysis, 17 trays only were used.

A glass spool piece containing a magnetic actuated reflux mechanism served to distribute the liquid to the top stage. Since no external reflux was associated with this analysis, the reflux mechanism was left in a position allowing for total feed to the top tray. The vapor feed

was accommodated by means of a glass spool-piece (with radial connection) situated below the bottom tray.

Removal of vapor and liquid from the tower was accommodated by means of glass caps with centrally located nozzles. The spool pieces, caps, and column were assembled by means of flanges.

The column has provisions for tray vapor sampling; two of these connections provided means for the thermometer taps used to monitor the temperatures of stages 16 and 5. These points were chosen because they were furthest apart from each other.

The entire column, upper spool piece and head, and a portion of the lower spool piece above the vapor feed was wrapped in blanket insulation, over which asbestos pipe insulation was applied. Insulation was not applied to the lower portion of the bottom spool piece and bottom head to allow for visual monitor of the liquid level.

EXPERIMENTAL OPERATION OF
COLUMN AND RELATED EQUIPMENT

The feed solution (0.25 mole fraction acetic acid) was prepared in the storage vessel, using the necessary volumes of acetic acid and water to obtain this concentration. The volumetric analysis (titration) indicates that, in effect, a 0.25 mole fraction solution was indeed obtained. The feed inventory would last approximately two hours.

To begin process flow, the pump had to be primed on total recycle. It was from this recycle stream that the feed sample was taken. At this time, the condenser cooling water was introduced.

Inlet vapor feed was then introduced to the column, slowly at first; the vapor preheater was activated. As the feed temperature began to rise, the vapor flowrate was increased, and the variac voltage increased in proportion until the design flowrate was registered on the rotameter.

As the vapor feed was heating, liquid feed was introduced to the tower in a small amount at first, and the second and third feed preheaters were activated. As with the vapor, the heat input was increased in proportion to the flowrate until design flowrate was read on the rotameter. At this point, the vapor feed had reached approximately 67.0°C, the liquid feed approximately 86.0°C; the timer was then started.

The column insulation was temporarily removed to insure that all stages were fractionating. Owing to a recent accident, there had been repair work done on the column; work which necessitated removal and re-installation of the bottom two trays. Upon re-insertion, the underdownflow clearance was erroneously set higher than the weir height. therefore it was impossible to maintain a liquid seal. All other stages were fractionating properly, so for the purposes of this analysis, 17 stages were considered.

To prevent vapor bypass during startup, the bottoms liquid outlet valve was left in the closed position. As the inventory was increased, the valve was opened enough to maintain a static liquid head.

As the tower began to fractionate, the liquid level in the overhead condensate trap began to increase. The trap valve was opened enough to reduce the level in the trap and still maintain the vapor seal.

Initially, some overhead product had bypassed the condenser and had condensed in the line after the trap. This was evident as a level in the drain trap was noticed. The cooling water circulation rate was increased to compensate for the increased condensing rate; and no further increase in drain level was noticed.

Heat Input

As the variac voltage on the second and third liquid feed preheaters was increased, the heating tapes began to "smoke". This smoke

represented the maximum allowable heat input from these preheaters. To continue safe operation, the voltage was reduced, and the insulation around the preheaters was loosened to promote heat removal from the outer surface of the tapes. At this point, the first (gas) feed preheater was activated; this would serve to increase the inlet temperature to the second and third preheaters. As the smoking decreased, the variac voltage was increased; the heat input required from the second and third feed preheaters was now reduced due to the first preheater.

The vapor feed preheater also began to smoke and the same procedure was used to rectify it. As there was no gas preheater for the vapor feed, the inlet gas temperature had to be regulated by the physical constraints of the preheater.

The temperatures selected for the vapor and liquid feed correspond to the equilibrium concentrations predicted by calculation. In spite of the problems associated with the preheaters, appreciable feed temperatures were obtained. The liquid feed leveled off between 95 and 105 degrees Centigrade; the vapor feed approximately 100 degrees Centigrade which closely correspond to the respective equilibrium temperatures for the top and bottom stages. An additional problem was caused by flashing of the liquid feed. A transparent piece of flexible tubing was installed on the feed nozzle to monitor the liquid flow, and when surging slugs of liquid were noticed, the heat input had to be reduced to re-establish single phase flow. Flashing was noticed during sampling, as the data for

samples 8 and 22 indicates.

Although the feed temperatures were sufficient, the actual column stage temperature was grossly insufficient. The stage temperatures monitored on trays 16 and 5 reached maximum temperatures of 69 and 50 degrees Centigrade respectively.

During the course of the experiment, the liquid level in the column bottoms and the overhead condensate trap was constantly monitored. Since the storage capacity in the bottoms was large compared to the flowrate, periodic observation was all that was necessary to insure that the level was maintained below the vapor inlet. The overhead condensate trap had a rather limited inventory, and being that the amount of liquid condensed was influenced to a great degree by the heat input to the tower, any variation in heat input was noticed in the level in the condensate trap. This therefore required almost constant monitoring.

The information recorded at the time of each sample was as follows:

- (1) Elapsed time.
- (2) Respective feed temperature at time of sample.
- (3) Column temperature (stages 16 and 5).
- (4) Column bottoms and overhead pressure (vapor samples only).

The data is presented in tabular form in Tables 3 and 4, and a critical analysis of the data is presented in the section "Discussion of Results". The process flow diagram (Figure 3) illustrates the equipment arrangement and flow scheme.

TECHNIQUE FOR TITRATION AND VOLUMETRIC ANALYSIS

Titration

In order to determine the efficiency with which the desorption column had performed, the concentration of the acetic acid in the condensed overhead and bottoms product had to be determined. The method chosen to determine the concentrations was titration. A Gas Chromatograph was available, but the type of packing that would be needed to resolve the acetic acid/water solution was not available, hence titration proved to be the most feasible method with the available equipment.

The samples were collected in acetone-rinsed sample bottles, approximately 30 milliliters in capacity. This would be sufficient to allow for spillage and retitration if necessary. The titration was performed at room temperature.

The indicator chosen was Ortho-Cresolsulfonephthalein, commonly known as Cresol Red; which has an indicator range of 7.0 to 8.8 pH. The upper value of pH approximately corresponds to a 0.071 molal solution of Sodium Acetate, which could be achieved by sufficient dilution of the sample.

The selections of the volumes used for titration was rather arbitrary, but the pH of the titrated solution was required to lie reasonably within the range of the selected indicator. The procedure used is as follows:

A one milliliter sample was withdrawn and diluted with 50 milliliters of distilled water. After thorough mixing, a one milliliter sample of the dilute solution was withdrawn and deposited in a titration bottle. 0.1 molal Sodium Hydroxide solution was introduced by a buret, and added until a color shift to neutral or neutral-red was observed. A white background was used to facilitate observation of the color shift, and the sample was agitated to insure complete reaction.

Volumetric Analysis

After determining the amount of 0.1 molal Sodium Hydroxide needed for the titration of each sample, the concentration of Sodium Acetate, which is the Acetic Acid concentration, was calculated. A sample calculation, which was done to determine the feed sample concentration, along with the logic used for the analysis, appears in the Appendix.

Once the concentrations had been determined, the pH was calculated. This step in the analysis served only to determine if the selected dilutions for the titration were sufficient for the indicator that was used. A sample calculation, used to determine the feed sample pH, along with explanatory derivation and logic, also appears in the Appendix. The pH values for the respective samples is shown in Table 2.

EXPERIMENTAL RESULTS

Table 2 lists the molarity, pH, and Acetic Acid and Water composition for each respective sample. A sample calculation (using the feed sample) appears in the Appendix. This set of sample calculations illustrate the various calculation methods and logic used to convert the volume of Sodium Hydroxide needed for titration into molarity, pH, and compositions (expressed in terms of mole fraction).

The following table summarizes the best experimental results and the results predicted by calculation. The experimental results correspond to samples and for the condensed overhead and bottoms compositions, respectively. The compositions are in units of mole fraction.

COMPONENT	EXPERIMENTAL COMPOSITIONS		EXPECTED COMPOSITIONS	
	<u>Overhead</u>	<u>Bottoms</u>	<u>Overhead</u>	<u>Bottoms</u>
Acetic Acid	0.16118	0.3965	0.003917	0.9860
Water	0.83882	0.6035	0.996083	0.0140

TABLE 2

SUMMARY OF EXPERIMENTAL RESULTS

Sample #	Molarity of Sodium Acetate (moles/liter)	pH	$x_{\text{acetic acid}}$	x_{water}
Feed	0.1765	8.998	0.2509	0.7491
1	0.1350	8.940	0.1700	0.8300
2	0.1906	9.024	0.2834	0.7166
3	0.1436	8.953	0.1853	0.8147
4	0.2014	9.030	0.3103	0.6897
5	0.1298	8.931	0.1612	0.8388
6	0.2314	9.056	0.3965	0.6035
7	0.1436	8.953	0.1853	0.8147
8	0.2057	9.031	0.3217	0.6783
9	0.1373	8.943	0.1740	0.8260
10	0.2286	9.054	0.3876	0.6124
11	0.1329	8.936	0.1664	0.8336
12	0.1899	9.013	0.2817	0.7183
13	0.1644	8.982	0.2253	0.7747
14	0.1891	9.012	0.2797	0.7203
15	0.1533	8.967	0.2043	0.7957
16	0.2068	9.032	0.3248	0.6752
17	0.1577	8.973	0.2117	0.7883
18	0.1878	9.011	0.2767	0.7233
19	0.1351	8.939	0.1702	0.8298
20	0.2034	9.028	0.3155	0.6845
21	0.1384	8.945	0.1760	0.8240
22	0.1965	9.021	0.2978	0.7022
23	0.1394	8.946	0.1777	0.8223
24	0.1950	9.019	0.2940	0.7060

DATA ANALYSIS- CALCULATION OF APPARENT
COLUMN EFFICIENCY

Using the data presented in Table 2, the column efficiency relating to mass transfer can be estimated. This efficiency will be a direct indication of the feasibility of separating the acetic acid/water solution by the methods proposed in this Thesis.

Essentially, the analysis relates to a reverse calculation of that used to determine the bottoms composition. In that case, the number of stages was known; the variable being the bottoms composition. For the purposes of this analysis, the bottoms composition has been experimentally determined, the variable will now be the number of stages (N).

In order to determine the efficiency, the bottoms and overhead molal flowrates have to be determined. This can be accomplished by examining component material balances around the column:

$$L_{N+1}x_{N+1} = L_1x_1 + V_Ny_N$$

. . . for acetic acid, and

$$L_{N+1}x_{N+1} = L_1x_1 + V_Ny_N$$

. . . for water.

The terms L_{N+1} , x_{N+1} (for both water and acetic acid) have been determined - they are the feed molal flowrate and compositions. The values of x_1 and y_N for both acetic acid and water can be determined from the experimental results. The material balances present 2 equations in terms of 2 unknowns; subtraction of these equations will yield values of L_1 and V_N . Note that the inlet vapor stream does not

enter the analysis due to the fact that it is essentially inert.

Having determined L_1 the values of f can be calculated for both components by substitution of the respective values into the equation:

$$f_{\text{HAC}} = L_1 x_1 / L_{N+1} x_{N+1}$$

. . . and

$$f_{\text{H}_2\text{O}} = L_1 x_1 / L_{N+1} x_{N+1}$$

Stripping Factor

In order to calculate the efficiency, the effective stripping factor must be estimated. The stripping factor (for stage 1) is defined as:

$$S_1 = \frac{V_0 y}{L_1 x}$$

This equation can be used for either the acetic acid or water. To be totally rigorous, V_1 , which is the vapor from stage 1, should be used in place of V_0 . This value, however, cannot be determined - the composition on stage two is not known. Also, the liquid compositions on stage 1 are known (experimentally determined), but the actual vapor phase compositions above stage 1 at the operating temperature are unknown - hence the equilibrium values will be used.

In like manner, the stripping factor for the uppermost stage is estimated as:

$$S_N = \frac{L_N y}{L_{N+1} x}$$

. . . for both acetic acid and water; using the respective compositions. As before, L_{N+1} is used (L_N cannot be determined) and the respective liquid compositions are determined from the equilibrium values.

Having estimated S_1 and S_N for both components, an estimation of the effective stripping factor is determined by the Edmister equation:

$$S = (S_N(S_1+1) + 0.25)^{0.5} - 0.5$$

. . . for both acetic acid and water. This value of S represents an "estimation" of the effective stripping factor for the actual column operation. The number of stages, N , can be determined by the equation:

$$f = \frac{1-S}{1-S^{N+1}}$$

. . . for both components which is recognized as equation (6). The value of N can be determined for both acetic acid and water; each of these values can be used to obtain an apparent column efficiency by the relationship:

$$\eta \% = (N/17) \times 100$$

. . . expressed in "per-cent". The calculation for the efficiency (based on water compositions) with explanatory logic appears in the Appendix.

DISCUSSION OF RESULTS

The data presented in Table 2 indicates that the acetic acid and water compositions in both the overhead and bottoms streams differ greatly from the expected calculated compositions. This is due to a number of factors which will be discussed in the following sections. These factors will be analyzed for their effect on tower operation and hydraulic and mass transfer efficiency.

Tower Operation

Feed contamination. To prevent any contaminants from entering the system in the liquid feed, distilled water was used as the dilutant in preparing the feed solution. The acetic acid, manufactured by Celanese Company, is certified to be glacial (and was so analyzed). In the vapor feed the inert gas is passed through a bed of desiccant prior to entering the column. This is done to insure that variations in humidity would not affect the vapor water composition to Tray 1; it would be essentially water-free.

Copper alloy contamination. A test run using copper tubing from the column to the first condenser resulted in a blue-green condensate. Further research indicated that the contamination was due to acetic acid present in vapor phase attacking the copper alloy. This problem was rectified by replacing the copper tubing with glass tubing. An additional test run then produced a clear, colorless condensate.

There was also the possibility of copper contamination entering the column via the liquid feed. Since the copper alloy is sensitive

to an aerated acid system only, any pockets in the feed line would increase the possibility of contamination. To minimize this possibility the physical arrangement of the feed line is such that an incline between the column and pump is maintained. This would insure that the line was operating full of liquid. During column operation, observation of the feed entering the column did not indicate any alloy contamination.

Hydraulic Efficiency.

The predominant factors affecting hydraulic efficiency are temperature, pressure, and flowrate; and the degree of accuracy to which each was monitored during operation.

Temperature and pressure. Inadequacy of temperature and pressure could affect the vapor and liquid densities, the vapor being more sensitive. The net effect would be to introduce uncertainty into the parameters used to calculate flowrates and hydraulic efficiency. The vapor and liquid densities are used to generate both axes of the Flooding Capacity diagram (Figure 5) and appear in practically all equations used to generate the liquid head parameters (the axes of the Weeping diagram, Figure 6).

Flowrate. Fluctuations in vapor and liquid flowrate could have a definite effect on hydraulic efficiency. Any variations would effect the vapor-liquid ratio, which is used in the abscissa of the Flooding Capacity diagram. Fluctuations in flowrate could also have a pronounced effect on accurately predicting the weep point. As with the

densities, flowrates appear in practically all equations that are used.

Mass Transfer Efficiency.

As with hydraulic efficiency, inadequacy of temperature, pressure, and variations in flowrate could have effect on the ability of the system to fractionate. The most pronounced effect of temperature and pressure is mainly with the equilibrium constant, K . The K values are a direct function of temperature and pressure. Also, variations in flowrate could affect calculation of the stripping factors. Combination of these two factors could cause gross uncertainty in calculating the acetic acid and water separation.

Other factors affected by variations in temperature and pressure are the equilibrium concentrations and relative volatility. Since operation was not at equilibrium temperature, the chart equilibrium values (Table 5) do not strictly apply to this system. Also, the relative volatilities shown in Table 5 indicate the water to be the more volatile component. Since the equilibrium temperatures were not obtained, these volatility values would be subject to uncertainty, being direct functions of both the water and acetic acid equilibrium concentrations. However, the relative volatilities would all be affected in the same proportion; and the overhead condensate would still be expected to be richer than the bottoms in water.

Volumetric Analysis

The uncertainty associated with the volumetric analysis and titration lies basically with measurement and end-point determination. As the procedure indicates, a 1.0 milliliter sample was withdrawn and diluted with 50.0 milliliters of distilled water. A 1.0 milliliter sample of the dilute mixture was then withdrawn and titrated. Each step imposes a degree of uncertainty in measurement.

Also, determination of the end-point is subject to interpretation. The color shift that would indicate neutralization is from colorless to light pink. The titration volumes of NaOH (0.01 milliliter per drop from the buret) in most samples caused a color shift from colorless to red. This indicated a shift from an acidic to basic solution, passing through the neutral region.

CONCLUSIONS

CONCLUSIONS
QUALITATIVE ANALYSIS

Contamination:

Contaminants present in sufficient quantity during column operation could have a definite detrimental effect on the expected acetic acid and water separation. The use of distilled water and glacial acetic acid insured a pure liquid feed. The calculation procedure assumed a water-free vapor feed, which was obtained by the use of desiccant. Copper alloy present in the product streams would not only exhibit uncertain titration results, but would greatly impair observation of the color shift during titration. The methods previously outlined apparently rectified the problem of alloy contamination, as both product streams were colorless. It seems unlikely therefore, that any contaminants, whether internal or external, could adequately justify the serious discrepancy between the expected and observed acetic acid/water separation.

Hydraulic effects. These were also considered but are disregarded from being a major cause of poor column operation. Although all parameters (temperature, pressure, and flowrate) contribute partially to the control of both flooding and weeping, only a very large deviation would have any kind of noticeable effect. In the Flooding analysis, the intersection of the curve and the x-coordinate on Figure 5 is in a relatively horizontal portion (the slope is approximately zero), so any changes in the x-coordinate resulting from deviations in densities or flowrate would have little effect

on C_{SB} . Also, the flooding capacity of both ends of the tower is approximately 50%, which is well below the limit of efficient operation.

The effects of dumping could also have a detrimental effect on efficient hydraulic operation. By comparison, h_{σ} is much greater than h_h , and is essentially controlling the y-coordinate on the dumping curve (Figure 6). Therefore any changes in density would not have a great effect. Surface tension is also a function of temperature, and any variations would cause a proportional change in h_{σ} . Therefore, any uncertainty in the value of the surface tension would be proportionally reflected in the y-axis of Figure 6. The effect on efficient operation is suppressed by the low values of $(h_w + h_{ow})$, the x-coordinate, which are 0.197 for Tray 17 and 0.1307 for Tray 1. Extrapolating the dumping curve to the origin indicates that both trays operate in the region of efficient hydraulic performance.

Mass transfer effects. Probably the greatest sources of uncertainty introduced into this analysis resulted from the inadequate temperature at which the system was operated. The inadequate temperature would lead to errors in equilibrium values, which then influence the stripping factors; and both the equilibrium and stripping factors are used in predicting the obtainable separation (see "Sample Calculation - System Parameters").

Control of feed temperatures did not impose a major problem; the liquid feed was heated to flash temperature in spite of the physical constraints inherent in the feed preheater system. Samples 8 and 22 indicate temperatures of approximately 105 degrees Centigrade. This heat, however, was quickly dissipated; being lost through the column insulation and used as Heat of Vaporization. The heat required for mass transfer was greater than that which entered with the feed streams, hence a larger amount of water remained in solution than the calculation indicates. This is reflected in the relatively high concentration of water in the condensed overhead samples.

Another point worthy of mention is a comparison between the values of f obtained by the calculation methods, and the effective f obtained by the methods suggested in the "Column Efficiency" section of this Thesis. The effective f values are 0.80 and 0.4075 for acetic acid and water, respectively. The assumed values obtained by the iterative calculation are 0.98834 and 0.00472 for acetic acid and water, respectively. A comparison of the respective acetic acid and water f values reinforces the postulation that insufficient heat input retarded the separation. The reasonable agreement in the acetic acid f values indicates that the reduced temperature had little effect on its performance in solution. The large difference between the f values for water indicates that much more water was present in the bottoms stream than was expected (recalling the

definition of f , which is the fraction of a given component in the bottoms). This can be explained by insufficient mass transfer of water from the liquid to vapor phase, possibly caused by insufficient heat input. The column operating temperature was deficient in heat because the available heat was used to vaporize as much water as possible.

Since the equilibrium data (Table 5) represents atmospheric conditions, the slight pressure needed to maintain effluent to the exhaust hood is not expected to introduce a great amount of uncertainty into the analysis. Fluctuation in molar flowrate would cause proportional uncertainty in the stripping factors; being a direct function of V/L . However, the molar flowrates were regulated by rotameters, and a slight variation in the V/L ratio could not be expected to justify the poor column performance.

When referring to molar flowrates, it should be noted that the only streams controlled externally are the liquid feed to Tray 17 and the vapor feed to Tray 1. The vapor and liquid flowrates on Trays 17 and 1 respectively are inherent in the system; being regulated by the amount of mass transfer being obtained. As much more water remained in solution than expected, the "observed" stripping factor is expected to be lower than the calculated value by virtue of the increased denominator in the V/L ratio. Comparison of the assumed stripping factor (1.2463) and the observed stripping factor (0.7354) indicates that this is indeed the case.

Volumetric analysis. Any uncertainty in titration could not in itself substantiate the poor performance of the column. As Table 2 indicates, the calculated pH values agree very closely to the upper pH indicator limit. This implies that a genuinely neutral solution was achieved when a color shift was observed. Also, each drop contained approximately 0.01 milliliters. The smallest amount of NaOH used for any sample was 1.298 milliliters (sample 5). This indicates that an additional drop would have increased the amount of NaOH required to 1.308 ml., or a 0.7704% increase in volume. It would therefore seem reasonable to assume that the methods used for titration and volumetric analysis were satisfactory.

RECOMMENDATIONS

There are many other factors that influence the rate of mass transfer in an equilibrium stage. Molecular diffusivities, concentration gradients imposed by poor vapor-liquid contact, molecular interactions, etc., are only a few. It is probable that a combination of all factors considered as part of this analysis, from both a hydraulic and mass transfer efficiency standpoint, contributed in some degree to the poor operation of the column. However, if any single parameter could be isolated as being predominant, it would be the temperature. No other single parameter has such a widespread effect as the temperature. Temperature influences equilibrium data, equilibrium constants, relative volatility, and to a degree, molal flowrate. The net effect of inadequate temperature is erroneous prediction by calculation methods, and poor column operation. It seems probable that the system as described in this Thesis, with an operating temperature more closely approximating equilibrium conditions, would produce results more consistent with calculation.

APPENDIX

TABLE 1

TOWER SPECIFICATIONS

Tower Outer Diameter	3.1875 inches
Tower Inner Diameter	2.9375 inches
<u>Tray Data</u>	
Tray Type	Radial Single Flow (Perforated)
Number of Trays	19
Numbering Sequence	Bottom to Top
Tray Spacing	2.1875 inches
Tray Area (Total)	0.047063 square feet
Tray Active Area, A_n	0.039297 square feet
Downcomer Area	0.003883 square feet
Downcomer Type	Circular
Sealpot Area	0.003883 square feet
Diameter of Perforations	0.037 inches
Number of Perforations per Tray	601
Perforated Area	0.004488 square feet
% Hole Area/Active Area	11.42%
Vapor Discharge Coefficient, C_{v_0}	1.0
Outlet Weir Length	0.703800 inches
Outlet Weir Height	0.078125 inches

TABLE 3SUMMARY OF EXPERIMENTAL DATA
- BOTTOMS LIQUID -

Sample #	Time Into Run (min.)	Liquid Feed Temp. (°C)	Tower Temperature (°C)	
			Tray 16	Tray 5
2	17.0	86.0	64.0	47.0
4	45.0	90.0	76.0	47.0
6	55.0	95.0	77.0	48.0
8	65.0 (Flash)	105.0	68.5	50.0
10	75.0	94.0	69.0	50.0
12	85.0	100.0	68.0	50.0
14	90.0	98.0	67.5	50.0
16	100.0	100.0	68.0	47.5
18	105.0	101.0	69.0	48.0
20	110.0	97.0	68.0	49.0
22	115.0 (Flash)	104.0	70.5	50.0
24	120.0	103.5	68.0	50.0

TABLE 4

SUMMARY OF EXPERIMENTAL DATA
 - CONDENSED OVERHEAD VAPOR -

Vapor Sample #	Time Into Run (min.)	Vapor Feed Inlet T°C	P _{Top} PSIG	P _{Btm} PSIG	Tower Temperature (°C)	
					Tray 16	Tray 5
1	10.0/15.0	66.5/78.0	1.80	2.10	64.5	46.0
3	40.0	86.5	2.25	2.50	65.0	48.0
5	55.0	90.0	2.25	2.60	68.0	47.0
7	60.0	92.0	2.25	2.60	69.5	49.5
9	75.0	93.5	2.25	2.60	68.5	50.0
11	85.0	94.0	2.25	2.60	68.0	50.0
13	90.0	96.0	2.50	2.75	67.5	50.0
15	100.0	101.5	2.40	2.75	68.0	47.5
17	105.0	103.0	2.40	2.75	69.0	48.0
19	110.0	104.5	2.40	2.80	68.0	49.0
21	115.0	104.0	2.50	2.90	70.5	50.0
23	120.0	103.5	2.60	2.95	68.0	50.0

TABLE 5

VAPOR-LIQUID EQUILIBRIUM DATA
WATER (1) AND ACETIC ACID (2)

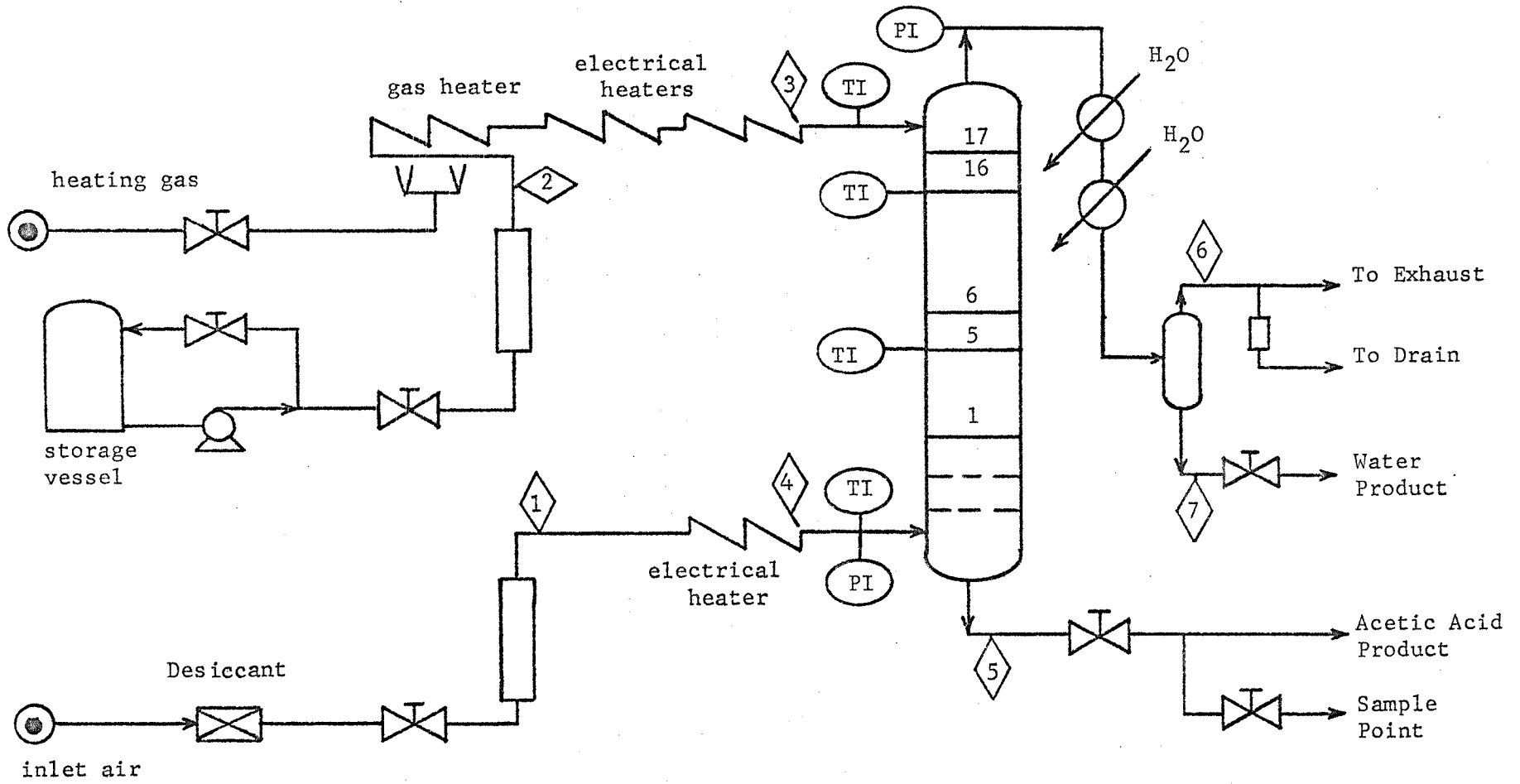
Source: Brown I., Ewald A. H.: Austr. J. Sci. Res. A3,306 (1950).

x_1	y_1	x_2	y_2	Temperature °Centigrade	$K_1=y_1/x_1$	$K_2=y_2/x_2$	$\alpha_{12}=K_1/K_2$
0.0034	0.0069	0.9966	0.9931	117.64	2.0294	0.9965	2.0366
0.0055	0.0112	0.9945	0.9888	117.51	2.0364	0.9943	2.0481
0.0474	0.0979	0.9526	0.9021	115.03	2.0654	0.9470	2.1810
0.0812	0.1446	0.9188	0.8554	113.81	1.7808	0.9637	1.9127
0.1497	0.2382	0.8503	0.7618	111.51	1.5912	0.8959	1.7760
0.2198	0.3273	0.7802	0.6727	109.84	1.4891	0.8622	1.7270
0.2917	0.4071	0.7083	0.5929	108.16	1.3956	0.8371	1.6671
0.3378	0.4573	0.6622	0.5427	107.36	1.3538	0.8195	1.6520
0.4198	0.5496	0.5802	0.4504	105.85	1.3092	0.7763	1.6865
0.5359	0.6591	0.4641	0.3409	104.17	1.2300	0.7345	1.6745
0.6463	0.7524	0.3537	0.2476	102.86	1.1642	0.7000	1.6630
0.7388	0.8217	0.2612	0.1783	101.92	1.1120	0.6826	1.6291
0.8251	0.8783	0.1749	0.1217	101.24	1.0645	0.6958	1.5300
0.9210	0.9429	0.0790	0.0571	100.54	1.0238	0.7228	1.4165
0.9676	0.9761	0.0324	0.0239	100.24	1.0088	0.7377	1.3676
0.9891	0.9921	0.0109	0.0079	100.07	1.0030	0.7248	1.3840

FIGURE 3

PROCESS FLOW DIAGRAM

Note: molar flowrates and temperatures based on theoretical calculation



◇ 1	0.1249 moles/hr	◇ 4	T=117.0°C.	◇ 7	0.5642 moles/hr
◇ 2	0.7538 moles/hr	◇ 5	0.1895 moles/hr		
◇ 3	T=109.0°C.	◇ 6	0.1294 moles/hr		

FIGURE 4
MOLAR RATIO DIAGRAM

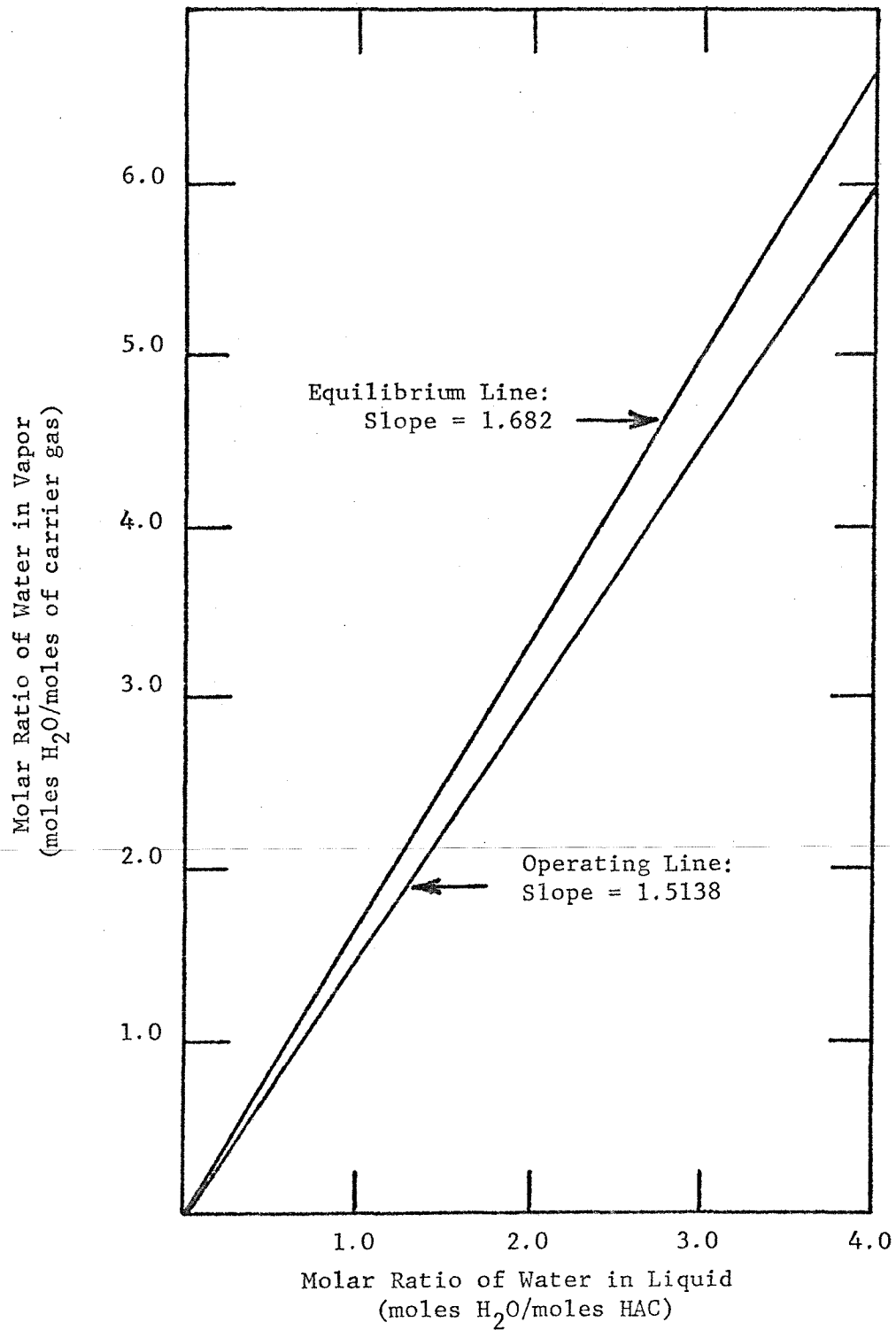


FIGURE 5

FLOODING CAPACITY DIAGRAM, SIEVE TRAYS

Source: Reference (6)

Note: Curve shown is interpolated to 3 inch trayspacing; surface tension of 20 dynes/cm., and a (hole area/active area) ratio greater than 10 percent.

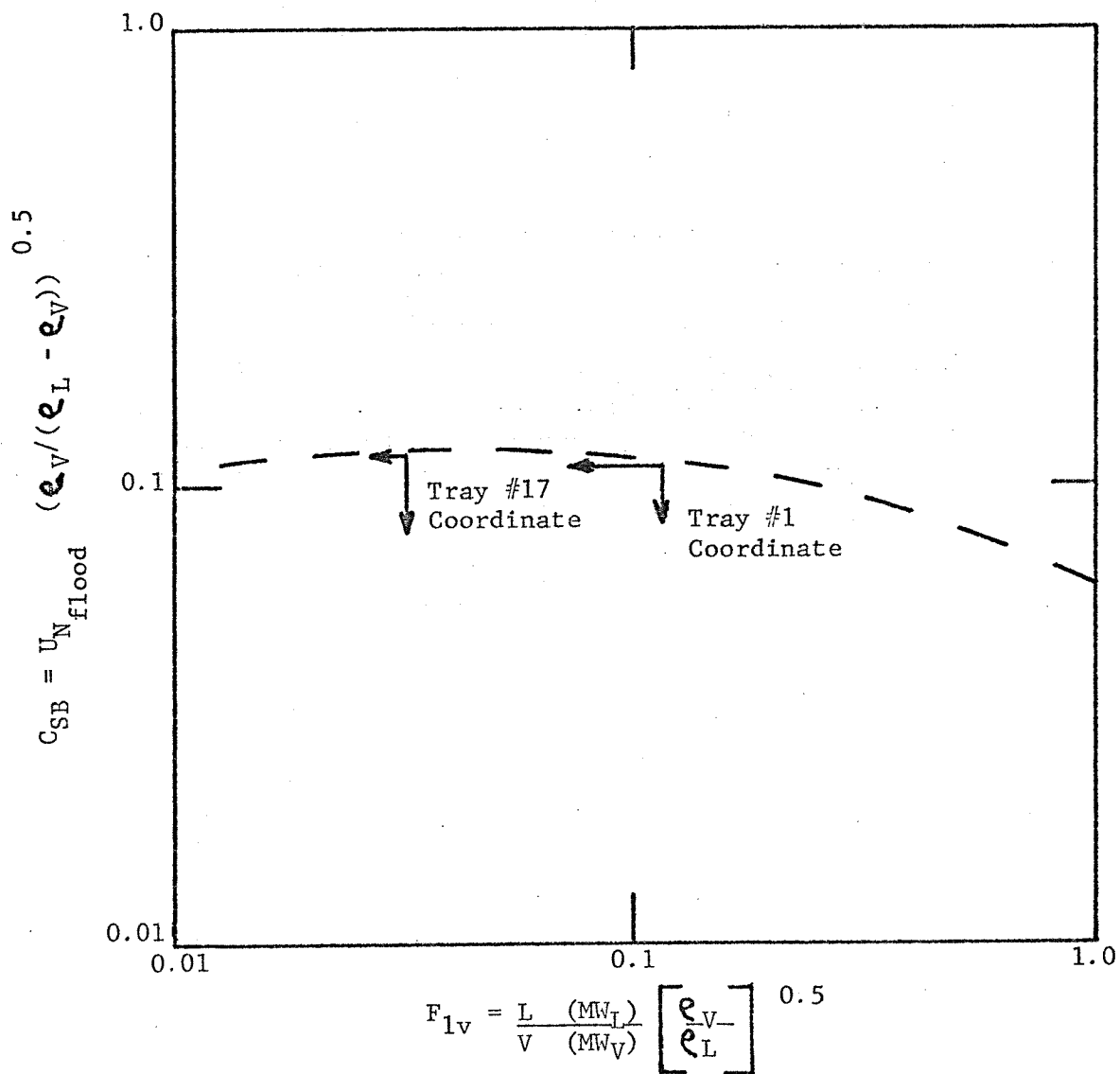
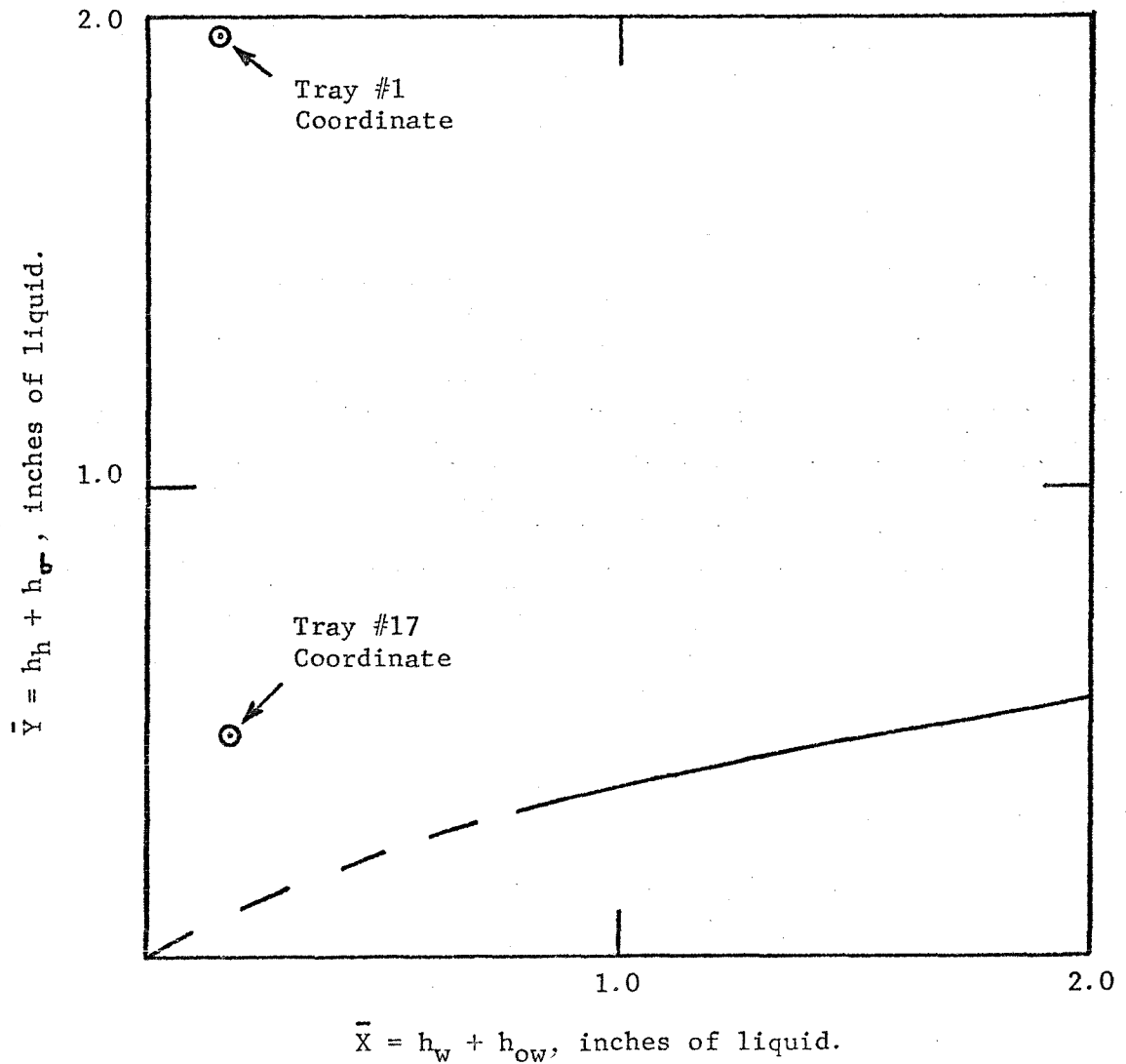


FIGURE 6

WEEPING DIAGRAM, SIEVE TRAYS

Source: Reference (6)

Note: Curve shown applies to a (hole area/active area) ratio range of 6 to 14 percent. Curve is extrapolated to the origin.



DERIVATION OF THE HORTON-FRANKLIN
EQUATION FOR PREDICTING BOTTOMS RECOVERY

Referring to Figure 1, and writing a material balance around stage n+1, the equation:

$$L_{n+2}x_{n+2} + V_n y_n = L_{n+1}x_{n+1} + V_{n+1}y_{n+1}$$

... is obtained. Noting that $y_n = K_n x_n$, and $y_{n+1} = K_{n+1} x_{n+1}$:

$$L_{n+2}x_{n+2} + V_n K_n x_n = L_{n+1}x_{n+1} + V_{n+1}K_{n+1}x_{n+1}$$

Rearranging this equation gives:

$$x_{n+2} - \left[\frac{K_{n+1}V_{n+1} + L_{n+1}}{L_{n+2}} \right] x_{n+1} + \frac{K_n V_n}{L_{n+2}} x_n = 0$$

In order to facilitate a mathematical solution, assume the K values and phase rates are constant within a column section. Making these assumptions, the only variables are the values of x. Rewriting this equation in "operator" form:

$$\left(E^2 - \left(\frac{KV}{L} + 1 \right) E + \frac{KV}{L} \right) x_n = 0$$

or rearranging:

$$\left(E - \frac{KV}{L} \right) (E - 1) x_n = 0$$

The subscripts have been eliminated because these values are now constant.

The two roots of the above equation are

$$S_1 = \frac{KV}{L} \quad \text{and} \quad S_2 = 1.0$$

The solution to the differential equation is therefore:

$$x_n = C_1 (S_1)^n + C_2 \quad (1)$$

Since this equation applies to any stage above the feed stage, S_1 will now be designated S_n (to denote the upper section stripping factor for whatever component the equation is written). For the upper section therefore:

$$x_n = C_1(S_n)^n + C_2 \quad (1a)$$

By analogy, an equation for the lower section can be written:

$$x_m = C_3(S_m)^m + C_4$$

These equations express the liquid concentration of a given component as a function of position within the column (exponent on stripping factor).

In order to solve equations (1) and (2), the constants must be eliminated. Recognizing that:

$$V_N Y_N = Dy_N + RDy_N = (1+R)Dy_N$$

... by material balance about the section above stage N. By definition, the component concentration in D can be expressed as:

$$Dy_N = (1-f)A$$

... where A is the total amount of a given component entering the column. By rearranging the material balance equation, and making the substitution for Dy_N :

$$y_N = (1+R)(1-f)A/V_N$$

and since $y_N = K_N x_N$:

$$x_N = (1+R)(1-f)A/K_N V_N$$

Substituting this expression of x_N into equation (1a) yields:

$$\frac{(1+R)(1-f)A}{KV} = C_1(S_n)^N + C_2 \quad (1b)$$

A material balance around stage N will yield the equation:

$$L_{N+1}x_{N+1} + V_{N-1}y_{N-1} = L_Nx_N + V_Ny_N$$

Rearranging in terms of y_{N-1}

$$y_{N-1} = \frac{L_Nx_N}{V_{N-1}} + \frac{V_Ny_N}{V_{N-1}} - \frac{L_{N+1}x_{N+1}}{V_{N-1}}$$

Since by material balance of the liquid to stage N:

$$L_{N+1}x_{N+1} = S_{x_s} + RDy_N$$

. . . and by definition:

$$q_sA = S_{x_s} \text{ and } D = V_N/(1+R)$$

The material balance around the section above tray N can be written:

$$L_{N+1}x_{N+1} = q_sA + \frac{R}{(1+R)} V_Ny_N$$

Substituting for x_N , y_N , and $L_{N+1}x_{N+1}$ into the material balance around stage N (again deleting the subscripts):

$$y_{N-1} = \frac{(1-f)A}{V^2} (V(1+R) + \frac{L}{K} (1+R) - RV - \frac{Vq_s}{(1-f)})$$

Noting that $y_{N-1} = Kx_{N-1}$:

$$x_{N-1} = \frac{(1-f)A}{KV^2} (V(1+R) + \frac{L}{K} (1+R) - RV - \frac{Vq_s}{(1-f)})$$

Noting that equation (1a) applies to any stage in the upper section:

$$x_{N-1} = C_1(S_n)^{N-1} + C_2 \quad (1c)$$

Subtraction of equation (1b) from (1c) (using the expanded expression

for x_{N-1} given above):

$$\begin{aligned} & \frac{(1-f)A}{KV^2} (V(1+R) + \frac{L}{K} (1+R) - RV - \frac{Vq_s}{(1-f)}) \\ & - \frac{(1+R)(1-f)A}{KV} = C_1(S_n^{N-1} - S_n^N) \end{aligned}$$

. . . which gives an equation for C_1 as a function of stream properties.

Rearranging for C_1 :

$$C_1 = \frac{(1-f)A \left(\frac{(L/K)(1+R)}{KV^2(S_n^{N-1} - S_n^N)} - RV - V_{qs}/(1-f) \right)}{KV^2(S_n^{N-1} - S_n^N)}$$

In terms of the constant C_1 , C_2 can be expressed by rearranging equation (1b) to:

$$C_2 = \frac{(1+R)(1-f)A}{KV} - C_1 S_n^N$$

Direct substitution of the expression for C_1 into the above equation will give a lengthy expression for C_2 as a function of stream properties. These equations for C_1 and C_2 , when substituted into equation (1a), will yield an expression for stream concentration strictly as a function of stream properties; or:

$$x_N = \frac{(1-f)A \left(\frac{(L/K)(1+R)}{KV^2(S_n^N - S_n^{N-1})} - RV - V_{qs}/(1-f) \right)}{KV^2(S_n^N - S_n^{N-1})} + \frac{(1+R)(1-f)A}{KV} \quad (1d)$$

A similar procedure can be used to eliminate C_3 and C_4 from equation (2). For a given component, a material balance around the section of the column below tray 1 (in Figure 1) is:

$$L_1 x_1 = R^1 B x_B + S_E x_{S_E} + B x_B$$

By definition:

$$fA = S_E x_{S_E} + B x_B$$

. . . for a given component. Therefore, substituting into the above equation:

$$L_1 x_1 = R^1 B x_B + fA$$

By definition, the fraction of a given component in L_1 that is removed in S_E (designated g) is:

$$g = S_E x_{S_E} / L_1 x_1$$

The material balance equation can be written (multiplying the equation by $(L_1 x_1 / L_1 x_1)$):

$$\begin{aligned} (1+R^1) Bx_B &= (L_1 x_1 - S_E x_{S_E}) \frac{L_1 x_1}{L_1 x_1} \\ &= \left(\frac{1 - S_E x_{S_E}}{L_1 x_1} \right) L_1 x_1 = (1-g)(L_1 x_1) \end{aligned}$$

Therefore:

$$Bx_B = \frac{(1-g)}{(1+R^1)} L_1 x_1$$

Recognizing from previous derivation that:

$$Bx_B = (L_1 x_1 - fA) / R^1$$

. . . and equating these two expressions gives:

$$\frac{L_1 x_1 - fA}{R^1} = \frac{(1-g)L_1 x_1}{(1+R^1)}$$

Cross-multiplying and expanding:

$$L_1 x_1 - fA - fAR^1 + L_1 x_1 R^1 = L_1 x_1 R^1 - L_1 x_1 R^1 g$$

Collecting like terms and solving for x_1 :

$$x_1 = \frac{(1+R^1) fA}{(1+gR^1) L_1}$$

Substituting the above expression into equation (1a), and

designating the extract rate L^1 (which is the assumed constant liquid rate in the lower section of the column):

$$\frac{(1+R^1) fA}{(1+gR^1) L^1} = C_3 (S_m)^1 + C_4 \quad (1e)$$

A material balance around stage 1 will yield the expression:

$$L_2x_2 + V_0y_0 = V_1y_1 + L_1x_1$$

Rearranging for x_2 :

$$x_2 = \frac{V_1y_1 + L_1x_1 - V_0y_0}{L_2}$$

By material balance around the section below stage 1:

$$V_0y_0 = F^1y_F^1 + R^1Bx_B$$

Noting that by definition:

$$F^1y_F^1 = q_F^1A$$

Substituting into the material balance equation for $F^1y_F^1$, and the previously derived expression for Bx_B , will yield:

$$V_0y_0 = q_F^1A + R^1 \left[\frac{(1-g)}{1+R^1} L_1x_1 \right]$$

Also, since $y_1 = K_1x_1$, y_1 can be expressed as a function of stream rates and properties by substituting x_1 for the previously derived value of y_1 or:

$$y_1 = K_1x_1 = K_1 \left(\frac{(1+R^1)fA}{(1+gR^1)L_1} \right)$$

Substituting the individual values derived for x_1 , y_1 , and V_0y_0 into the material balance around stage 1 yields the following expression:

$$L_2x_2 + \left(q_F^1A + \frac{R^1(1-g)L_1x_1}{(1+R^1)} \right) = V_1 \left(\frac{K_1(1+R^1)fA}{(1+gR^1)L_1} \right) + L_1 \left(\frac{(1+R^1)fA}{(1+gR^1)L_1} \right)$$

Factoring out the term $(fA/L(1+gR^1))$, and multiplying the (q_F^1A) term by $(L_1(1+gR^1)/f)(f/L_1(1+gR^1))$, the following equation is obtained (in terms of x_2):

$$x_2 = \frac{fA}{(L^1)^2(1+gR^1)} \left(V^1K^1(1+R^1) + L^1(1+R^1) - \frac{L^1(1+gR^1)}{f} - L^1R^1(1-g) \right)$$

The subscripts have been deleted on the L and K values because they are assumed constant.

Substituting this expression for x_2 into equation 2 gives (for stage 2):

$$\frac{fA}{(L^1)^2(1+gR^1)} K^1 V^1 (1+R^1) + L^1 (1+R^1) - L^1 R^1 (1-g) - \frac{L^1 (1+gR^1) q_{F1}}{f}$$

$$= C_3 (S_m)^2 + C_4 \quad (1f)$$

Subtraction of equation (1e) from equation (1f) and solving in terms of C_3 gives

$$C_3 = \frac{fA(K^1 V^1 (1+R^1) - L^1 R^1 (1-g) - L^1 (1+gR^1) q_{F1}/f)}{(L^1)^2 (1+gR^1) (S_m^2 - S_m)}$$

. . . which gives an equation for C_3 as a function of stream properties. Rewriting equation (1e) in terms of C_4 gives the equation:

$$C_4 = \frac{(1+R^1) fA}{(1+gR^1) L^1} - C_3 (S_m)^1$$

Direct substitution of this expression into equation (2) gives:

$$x_m = C_3 (S_m^m - S_m^1) + \frac{(1+R^1) fA}{(1+gR^1) L^1}$$

Direct substitution of the above equation for C_3 into the equation for x_m , after factoring an (S_m) out of the first term (and canceling), gives:

$$x_m = \frac{fA K^1 V^1 (1+R) - L^1 R^1 (1-gR^1) q_{F1}/f}{(L^1)^2 (1+gR^1) (S_m-1) (S_m)} (S_m^{m-1} - 1) (S_m)$$

$$+ \frac{(1+R^1) fA}{(1+gR^1) L^1} \quad (1g)$$

Equations (lg) and (ld) express stream composition as a function of system properties (for both sections of the column). An equation for the entire column can be obtained by combining equations (lg) and (ld) with a material balance around the feed stage, as follows. The material balance equation is:

$$V_M y_M + L_{M+2} x_{M+2} + F y_F = V_{M+1} y_{M+1} + L_{M+1} x_{M+1}$$

Again assuming constant phase rates, and since $x_{M+1} = y_{M+1}/K_{M+1}$, the equation:

$$V^1 y_M + L x_{M+2} + F y_F = V y_{M+1} + \frac{L^1 y_{M+1}}{K_{M+1}}$$

. . . is formed. The subscript (M+1) on the K value will be held for now; it will later be deleted and the K value designated either K or K^1 , depending on which section of the tower is under consideration.

A material balance around the upper section of the column (cutting V_N , L_{M+2} , V_{M+1} , and L_{N+1}) is expressed as:

$$V_N y_N + L_{M+2} x_{M+2} = V_{M+1} y_{M+1} + L_{N+1} x_{N+1}$$

Recognizing from previous derivation that:

$$y_N = (1+R)(1-f)A/V_N \quad ; \text{ and}$$

$$L_{N+1} x_{N+1} = q_s A + (R/1+R)V_N y_N$$

These expanded forms of y_N and $L_{N+1} x_{N+1}$ can be substituted into the above material balance equation as:

$$V_N \frac{(1+R)(1-f)A}{V_N} + L_{M+2} x_{M+2} = V_{M+1} y_{M+1} + q_s A + \frac{R}{1+R} \frac{(V_N)(1+R)(1-f)A}{(V_N)}$$

Expanding this equation and collecting like terms gives an equation (in terms of y_{M+1}) as:

$$y_{M+1} = \frac{L_{M+2}x_{M+2}}{V_{M+1}} + \frac{(1-f)A(1+R-R)}{V_{M+1}} - \frac{q_s A}{V_{M+1}}$$

The canceled terms in both previous equations have been shown for clarity.

Deleting the subscripts (assuming constant phase rates) gives the equation:

$$y_{M+1} = \frac{Lx_{M+2}}{V} + \frac{(1-f)A}{V} - \frac{q_s A}{V}$$

Substituting this expression into the feed stage material balance (and noting that $Fy_F = q_F A$) yields:

$$V^1 y_M + L_{M+2} x_{M+2} + q_F A = V_M \left(\frac{(1-f)A}{V} + \frac{Lx_{M+2}}{V} - \frac{q_s A}{V} \right) + \frac{L^1}{K_{M+1}} \left(\frac{(1-f)A}{V} + \frac{Lx_{M+2}}{V} - \frac{q_s A}{V} \right)$$

Expanding this term and dropping the subscript on V_M :

$$V^1 y_M + L_{M+2} x_{M+2} + q_F A = (1-f)A + Lx_{M+2} - q_s A + \frac{L^1(1-f)A}{K_{M+1}V} + \frac{LL^1 x_{M+2}}{K_{M+1}V} - \frac{L^1 q_s A}{K_{M+1}V}$$

The (Lx) terms cancel because the phase rate L is assumed constant.

Noting that $y_M = K^1 x_M$ equation (1g) can be used to substitute for y_M and equation (1d) for x_{M+2} in the material balance. The equation for x_n holds for x_{M+2} since both stages are in the upper section of the column. These expressions substituted into the above expanded feed stage material balance equation (making the proper adjustments on the exponents for the respective stripping factors) will yield the equation:

$$\frac{K^1 V^1 f A (K^1 V^1 (1+R^1) - L^1 R^1 (1-g) - q_F^1 L^1 (1+gR^1)/f)}{(L^1)^2 (1+gR^1) (S_m - 1)} (S_m^{M-1} - 1)$$

$$+ \frac{K^1 V^1 (1+R^1) f A}{L^1 (1+gR^1)} + q_F^1 A = (1-f)A - q_S A + \frac{L^1 (1-f)A}{K_{M+1} V} - \frac{L^1 q_S A}{K_{M+1} V}$$

$$+ \frac{L L^1 (1-f) A ((L/K) (1+R) - R V - V q_S / (1-f))}{K_{M+1} K V^2 (S_n^N - S_n^{N-1})} (S_n^N - S_n^{M+2})$$

$$+ \frac{L L^1 (1+R) (1-f) A}{K_{M+1} K V^2}$$

After considerable algebraic manipulation, the following simplified equation for f (which is equation 3) is formed:

$$f = \frac{(1-S_n^{N-M}) + q_S (S_n^{N-M} - S_n) + R(1-S_n) + h q_F^1 S_n^{N-M} (1-S_m^M)}{(1-S_n^{N-M}) + h S_n^{N-M} (1-S_m^M) + R(1-S_n)}$$

$$+ h((1+R^1)/(1+gR^1)) S_m^M S_n^{N-M} (1-S_m) \quad (3)$$

The expression for h depends upon the condition of the feed stream, F .

If the feed is vapor, it is more similar to the light (raffinate) phase, and therefore $K_{M+1} = K$. The equation for h therefore is:

$$h = \frac{L - KV}{L^1 - K^1 V^1} = \frac{L(1 - S_n)}{L^1(1 - S_m)}$$

If the feed is liquid, it is more similar to the heavy (extract) phase, and therefore $K_{M+1} = K^1$. This would correspond to a bubble-point feed; and h would be expressed as:

$$h = \frac{L/K - V}{L^1/K^1 - V^1} = \frac{K}{K^1} \frac{L(1 - S_n)}{L^1(1 - S_m)}$$

DERIVATION OF THE EFFECTIVE STRIPPING FACTOR
- THE METHOD OF EDMISTER -

The Horton and Franklin expression for stripper efficiency in terms of the stripping factor (for a N-stage stripper) is:

$$\frac{X_{N+1} - X_1}{X_{N+1}} = \frac{S_1 S_2 \dots S_N + S_2 S_3 \dots S_N + \dots + S_N}{S_1 S_2 \dots S_N + S_2 S_3 \dots S_N + \dots + S_N + 1}$$

$$- \frac{V_o Y_o}{L_{N+1} X_{N+1}} \frac{S_2 S_3 \dots S_N + S_3 S_4 \dots S_N + \dots + S_N + 1}{S_1 S_2 \dots S_N + S_2 S_3 \dots S_N + \dots + S_N + 1} \quad (11)$$

In terms of an effective stripping factor (S), equation (11) would appear as:

$$\frac{X_{N+1} - X_1}{X_{N+1}} = \frac{S^N + S^{N-1} + \dots + S}{S^N + S^{N-1} + \dots + S + 1}$$

$$- \frac{V_o Y_o}{L_{N+1} X_{N+1}} \frac{S^{N-1} + S^{N-2} + \dots + S + 1}{S^N + S^{N-1} + \dots + S + 1} \quad (11a)$$

Since by definition:

$$S^N + S^{N-1} + \dots + S = \frac{S^{N+1} - 1}{S - 1} \quad (11b)$$

and:

$$S^N + S^{N-1} + \dots + S + 1 = \frac{S^{N+1} - 1}{S - 1} \quad (11c)$$

... these expressions can replace the series expressions used in equation (11a). Upon substitution:

$$\frac{X_{N+1} - X_1}{X_{N+1}} = \frac{S^{N+1} - S}{S - 1} - \frac{V_o Y_o}{L_{N+1} X_{N+1}} \left(\frac{S^{N-1} + S^{N-2} + \dots + S + 1}{\frac{S^{N+1} - 1}{S - 1}} \right)$$

Multiplying the second term by S/S, and canceling the (S-1) terms:

$$\frac{X_{N+1} - X_1}{X_{N+1}} = \frac{S^{N+1} - S}{S^{N+1} - 1} - \frac{V_o Y_o}{S L_{N+1} X_{N+1}} \left(\frac{S^{N+1} - S}{S^{N+1} - 1} \right)$$

or:

$$\frac{X_{N+1} - X_1}{X_{N+1}} = 1 - \frac{V_0 Y_0}{S^N L_{N+1} X_{N+1}} \left(\frac{S^{N+1} - S}{S^{N+1} - 1} \right) \quad (11d)$$

. . . where the $S^1 = S$ when the equation is written in terms of the effective stripping factor.

An expression can be derived for a two-stage stripper by examining the relationship between equations (11d) and (11). For two stages:

$$\frac{S^3 - S}{S^3 - 1} = \frac{S_1 S_2 + S_2}{S_1 S_2 + S_2 + 1} \quad (11e)$$

and:

$$\frac{S^3 - S}{S^1 (S^3 - 1)} = \frac{S_2 + 1}{S_1 S_2 + S_2 + 1} \quad (11f)$$

Multiplying the right-hand portion of equation (11f) by $(S_2)(S_1+1)/(S_2+1)$ will yield equation (11e). From this, S^1 can be considered to be:

$$S = S^1 = S_2(S_1+1)/(S_2+1)$$

. . . for two stages. Cross-multiplying, and expressing the equation in terms of the effective stripping factor:

$$S(S+1) = S_2(S_1+1) \quad , \text{ or:}$$

$$S^2 + S - S_2(S_1+1) = 0$$

. . . a quadratic equation which has the positive solution:

$$S = (S_2(S_1+1) + 0.25)^{0.5} - 0.5$$

Edmister suggests that the effective factor is independent of the number of stages, or;

$$S = (S_N(S_1+1) + 0.25)^{0.5} - 0.5$$

This equation is then used to calculate an effective stripping factor for an N stage column.

SAMPLE CALCULATION
SYSTEM PARAMETERS

This set of calculations establishes the design flowrates, predicts the Acetic Acid/Water compositions in the bottoms and overhead product streams, and compares the chosen flowrates and conditions to the existing tray design (in reference to hydraulic efficiency).

Design Flowrates

This calculation establishes the liquid and vapor flowrates used during column sampling. For the purposes of brevity, the term "HAC" will be used to designate acetic acid, and "H₂O" to designate water.

The molar ratio diagram (Figure 4) shows an equilibrium line slope of 1.682 moles HAC/mole carrier vapor. Since the bottoms is expected to be dilute in H₂O, the operating line and equilibrium line will essentially converge at the origin. Hence, 90% of the equilibrium line slope is considered to be the operating line slope, or:

$$0.9 \times 1.682 = 1.5138 \text{ moles HAC(in)/mole vapor(in)}$$

A vapor rotameter setting of 7.0 corresponds to a volumetric flowrate of 0.0116 cubic feet/second. The vapor molar flowrate to the tower (assuming an ideal gas) is therefore:

$$\frac{0.0116 \text{ ft}^3}{\text{sec.}} \times \frac{3600 \text{ sec.}}{\text{hr.}} \times \frac{\text{mole} \cdot \text{OR.}}{10.73 \text{ PSIA} \cdot \text{ft}^3} \times \frac{17.0 \text{ PSIA}}{530.0 \text{ OR.}} =$$

$$0.12494 \frac{\text{moles vapor}}{\text{hour}}$$

The corresponding acetic acid molar flowrate is:

$$1.5138 \frac{\text{moles HAC}}{\text{mole vapor}} \times 0.1249 \frac{\text{moles vapor}}{\text{hour}} = 0.1891 \frac{\text{moles HAC}}{\text{hour}}$$

Since the feed composition has been selected as 0.25 mole fraction HAC, the total molar flowrate is:

$$0.1891 \frac{\text{moles HAC}}{\text{hour}} \times \frac{\text{total moles}}{0.25 \text{ moles HAC}} = 0.7538 \frac{\text{total moles}}{\text{hour}}$$

The volumetric flowrate through the rotameter is therefore:

$$\left[\begin{aligned} & (0.1891 \frac{\text{moles HAC}}{\text{hour}} \times 60.052 \frac{\# \text{ HAC}}{\# \text{mole}}) + \\ & (0.56468 \frac{\text{moles H}_2\text{O}}{\text{hour}} \times 18.016 \frac{\# \text{ H}_2\text{O}}{\# \text{mole}}) \end{aligned} \right]$$

$$\times 28.24 \frac{\text{liters}}{\text{cu.foot}} \times 1000 \frac{\text{milliliters}}{\text{liter}} \times \frac{1 \text{ hour}}{3600 \text{ sec.}} \times \frac{1 \text{ cu.foot}}{66.2 \#} =$$

$$2.55 \frac{\text{milliliters}}{\text{second}} \text{ of feed}$$

. . . which corresponds to a rotameter setting of 2.5. Hence a vapor rotameter setting of 7.0 and a liquid rotameter setting of 2.5 will be used for the duration of the experiment.

Calculation of HAC/H₂O Separation

Since this is an iterative type of calculation, only the converged trial will be illustrated.

The following table lists the chosen flowrates and assumed overhead vapor and bottoms compositions:

	$V_0 y_0$	$L_{n+1} x_{n+1}$	f	$L_1 x_1$	x_1	$V_{17} y_{17}$	y_{17}
HAC	0.0000	0.1891	0.9883	0.1869	0.9860	0.0022	0.0032
H ₂ O	0.0000	0.5647	0.0047	0.0026	0.1406	0.5620	0.8155
AIR	0.1294	0.0000	0.0000	0.0000	0.0000	0.1249	0.1813
TOT.		0.7538		0.1895		0.6891	

Using the expected overhead vapor flowrates for each component, the expected concentrations in the condensate can be shown as:

$$\begin{aligned} x_{\text{HAC}} &= 0.0022 / (0.0022 + 0.5620) \\ &= 0.003917 \end{aligned}$$

$$\begin{aligned} x_{\text{H}_2\text{O}} &= 0.5620 / (0.0022 + 0.5620) \\ &= 0.996083 \end{aligned}$$

. . . assuming total condensation.

Using equation 7:

$$\left[\frac{L_1}{L_{N+1}} \right]^{1/N} = \frac{L_n}{L_{n+1}} = \left[\frac{0.1895}{0.7538} \right]^{1/17} = 0.922014$$

The iteration is to proceed as follows: set $n=1$. Using a value of $L_1 = 0.1895$, L_2 calculated by equation 7 is:

$$L_2 = (0.1895/0.922014) = 0.205115$$

The moles transferred to the vapor phase is therefore:

$$L_2 - L_1 = 0.205115 - 0.1895 = 0.015615$$

Using $V_0 = 0.1249$, the vapor flowrate leaving Tray 1 is therefore:

$$V_1 = V_0 + (L_2 - L_1) = 0.1249 + 0.015615 = 0.140515$$

By repetitive calculation ($n=2, 3, 4 \dots 17$) the vapor/liquid flowrates for each tray can be determined.

Since the Stripping Factors will be evaluated for the top and bottom trays only, the flowrates of interest are (in moles per hour):

	n=1	n=17
V_n	: 0.14048	0.68910
L_n	: 0.18950	0.69502
$(V/L)_n$: 0.74362	0.9915

Evaluation of Stripping Factors

For the water on Tray 17:

$$y_{H_2O} = 0.815507$$

$$x_{H_2O} = 0.730534; \text{ (interpolated from Table 5)}$$

The K value is defined as (y_{H_2O}/x_{H_2O}) . Therefore,

$$K_{H_2O} = 0.815507/0.730534 = 1.116316$$

The stripping factor, S_N , is defined as KV/L .

$$S_{17-H_2O} = 1.116316 \times 0.99156 = 1.1069$$

In like manner, stripping factors were calculated for both components on Trays 1 and 17 (shown in the following table).

COMPONENT	S_1	S_{17}
HAC	0.63983	0.011775
H ₂ O	1.52914	1.106900

The Effective Stripping Factor, S, is defined as

$$S = (S_N(S_1 + 1.0) + 0.25)^{0.5} - 0.5$$

By direct substitution, the Effective Stripping Factors were determined as:

$$S_{H_2O} = 1.246283$$

$$S_{HAC} = 0.0123753$$

The "f" factor, which is the fraction of each component recovered in the bottoms, is defined as:

$$f = \frac{[(1 - S^N) + q_s(S^N - S)]}{(1 - S^{N+1})}$$

. . . where q_s is the fraction of each component entering in the feed stream.

However, the HAC and H₂O enter only in the feed stream, therefore

$q_s = 1.0$, and the equation reduces to:

$$f = (1 - S)/(1 - S^{N+1})$$

By direct substitution of the stripping factors into the above equation, the calculated values of f are determined and compared to the assumed values, as shown below:

COMPONENT	$f_{\text{calculated}}$	f_{assumed}	% DIFFERENCE
HAC	0.9876	0.9883	0.071
H ₂ O	0.0047	0.0047	-

Since the "% DIFFERENCE" values are essentially zero, the calculation is assumed to be converged.

Tray Hydraulic Calculations

This analysis is used to determine if the selected flowrates will exhibit efficient hydraulic operation when compared to the given tray design. The equations are referenced in Smith, B. D., Design of Equilibrium Stage Processes.

Analysis of Tray 1: Dumping. The column bottoms temperature is set at 117°C. Therefore, the vapor volumetric flowrate to Tray 1 is:

$$0.1249 \frac{\text{moles}}{\text{hour}} \times \frac{1 \text{ hour}}{3600 \text{ sec.}} \times 10.73 \frac{\text{PSIA ft.}^3}{\text{mole } ^\circ\text{R}} \times \frac{703.0^\circ\text{R}}{17.0 \text{ PSIA}} = 0.015442 \frac{\text{ft}^3}{\text{sec.}}$$

The corresponding vapor density is:

$$\text{vapor} = \frac{P(\text{MW})}{(R''T)} = \frac{16.95 \text{ PSIA} \times 29.0 \text{ \#/ \# mole}}{10.73 \frac{\text{PSIA ft.}^3}{\text{mole } ^\circ\text{R}} \times 703^\circ\text{R.}} = 0.06516 \text{ \#/ft.}^3$$

The liquid density at this temperature is determined as 55.1 #/ft.³.

The vapor hole velocity, U_H , is defined as

$$U_H = CFS_{\text{vapor}}/A_h$$

. . . where A_h is the net perforated area. Substituting into the above equation, the vapor hole velocity is:

$$0.15442/0.004488 = 3.4407 \text{ ft./sec.}$$

From Smith (6), the vapor discharge coefficient, C_{v0} , is seen to be essentially 1.0 for the tray design. The head loss due to vapor flow through the perforations is defined as:

$$h_h = 0.186 \frac{\rho_V}{\rho_L} \left[\frac{U_H}{C_{v0}} \right]^2$$

By direct substitution, the head loss for Tray 1 is calculated as:

$$0.186 \times \frac{0.06516}{55.1000} \times \left[\frac{3.4407}{1.0000} \right]^2 = 0.002604 \text{ inches of liquid}$$

To account for the liquid level on the tray deck, the head loss due to bubble formation, h_σ , must be calculated. h_σ is defined as:

$$h_\sigma = \frac{0.040 \times \sigma}{\rho_L \times d_h}$$

The surface tension for this system is 10 dynes/cm. Using the hole diameter (d_h) of 0.037 inches, a direct substitution yields:

$$h_\sigma = (0.040 \times 10)/(55.1 \times 0.037) = 0.1962 \text{ inches of liquid}$$

The combination of h_h and h_σ fixes a coordinate on the y-axis of the "dumping" curve (Figure 6). This parameter \bar{Y} , is:

$$\bar{Y} = h_h + h_\sigma$$

By direct substitution

$$\bar{Y} = 0.002604 + 0.1962 = 0.19881 \text{ inches of liquid}$$

The liquid flowrate from Tray 1, in gallons per minute, is

$$0.1895 \frac{\text{moles}}{\text{hour}} \times 59.465 \frac{\#}{\text{mole}} \times \frac{1 \text{ ft.}^3}{55.1\#} \times 7.48 \frac{\text{gallons}}{\text{ft.}^3} \times \frac{1 \text{ hour}}{60 \text{ minutes}}$$

$$= 0.02551 \text{ gallons/minute}$$

The height of liquid crest over the weir, h_{ow} , is defined as

$$h_{ow} = 0.48 \left[\frac{\text{GPM}}{l_W} \right]^{0.667}$$

By substitution into the above equation, using a weir length (l_W) of 0.7038 inches, h_{ow} is determined as:

$$h_{ow} = 0.48 \left[\frac{(0.02551)}{0.70380} \right]^{0.667} = 0.05257 \text{ inches of liquid}$$

This value of h_{ow} , combined with the weir height, h_w , fixes a coordinate on the x-axis of Figure 6. This parameter, \bar{X} , is

$$\bar{X} = h_w + h_{ow}$$

By direct substitution

$$\bar{X} = 0.07813 + 0.05257 = 0.1307$$

Entering Figure 6 with the values of \bar{X} and \bar{Y} determined above, for the respective area ratio, it can be seen that this tray will operate above the dump point, or point of efficiency loss.

Analysis of Tray 1: Flooding. The flow parameter for sieve tray design, F_{1V} , is defined as:

$$F_{1V} = \frac{L(MW_L)}{V(MW_V)} \left[\frac{\rho_{-V-}}{\rho_L} \right]^{0.5}$$

By direct substitution of the liquid and vapor molar flowrates, and their respective molecular weights and densities:

$$F_{1V} = \frac{0.1895}{0.1249} \times \frac{59.465}{29.000} \times \left[\frac{0.06516}{55.1000} \right]^{0.5} = 0.107$$

The trayspacing for the tower is 2.1875 inches. Entering Figure 5 with the calculated value of F_{1V} , and interpolating for the value of the trayspacing, the corresponding value of the vapor capacity parameter, C_{SB} , is (in feet/sec.):

$$C_{SB \text{ chart}} = 0.105$$

Since the chart values of C_{SB} are based on a surface tension of 20 dynes/cm., a correction factor relating the chart value of C_{SB} to the actual surface tension is required; and expressed as:

$$C_{SB \text{ actual}} = C_{SB \text{ chart}} \times \left[\frac{\sigma}{20} \right]^{0.2}$$

By substitution of the respective values, the corrected flow parameter is:

$$C_{SB \text{ actual}} = 0.105 \times \left[\frac{10}{20} \right]^{0.2} = 0.0915$$

The flow parameter is related to the liner tower vapor velocity, liquid density, and vapor density by the expression:

$$C_{SB \text{ actual}} = U_{N \text{ flood}} \left[\frac{\rho_V}{(\rho_L - \rho_V)} \right]^{0.5}$$

By direct substitution and algebraic rearrangement, the velocity of incipient flooding, U_N , is found to be (in feet/sec.):

$$U_{N \text{ flooding}} = 0.0915 / (0.06516 / (55.1 - 0.06516))^{0.5}$$

$$= 2.65$$

The actual tower velocity (based on the net tray area) is expressed (in feet/sec.) as:

$$U_{N \text{ actual}} = \frac{\text{ft}^3/\text{sec vapor}}{A_n}$$

By direct substitution:

$$\begin{aligned} U_{N \text{ actual}} &= 0.015442/0.039297 \\ &= 0.393 \end{aligned}$$

The approach to flooding, expressed as a ratio of the actual and flooding vapor velocities, is found to be:

$$\% \text{ flood} = (0.393/2.65) \times 100 = 14.83\%$$

(which indicates that efficient operation is expected).

Analysis of Tray 17: Dumping. In like manner, the \bar{X} and \bar{Y} values for Tray 17 are found to be:

$$\begin{aligned} \bar{X} &= 0.1975 \\ \bar{Y} &= 0.4750 \end{aligned}$$

This point, plotted on Figure 6 lies in the region above the "dump" curve.

Analysis of Tray 17: Flooding. In like manner, the approach to flooding for Tray 17 is found to be:

$$\% \text{ flood} = (2.11/4.363) \times 100 = 48.36\%$$

(which indicates that efficient operation is expected).

SAMPLE CALCULATION
VOLUMETRIC ANALYSIS

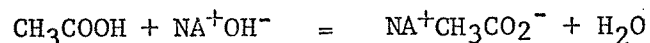
The following calculation illustrates the method by which the titration data is converted into the individual component compositions.

Sample: 1 ml. of fresh feed (dilute)

Amount of 0.1 molal NaOH required to titrate feed sample:

1.765 ml.

The titration equilibrium is expressed as:



. . . hence one mole of Acetic Acid in the sample will react to form one mole of Sodium Acetate.

The moles of Sodium Acetate formed during titration can be expressed as:

$$\text{moles} = VM$$

. . . where V is the volume in milliliters, and M is the molarity (moles/liter).

Since this relationship also applies to the Sodium Hydroxide solution, the molar equality can be expressed as:

$$(\text{VM})_{\text{Sodium Acetate}} = (\text{VM})_{\text{Sodium Hydroxide}}$$

Since the volume of sample, volume required for titration, and the molarity of the NaOH solution are known, the remaining variable, the molarity of Sodium Acetate, can be determined.

By direct substitution of these known values:

$$(1.0) (M)_{\text{Sodium Acetate}} = (1.765)(0.1)$$

$$M_{\text{Sodium Acetate}} = 0.1765 \frac{\text{moles of Sodium Acetate}}{\text{liter solution}}$$

The number of moles of Sodium Acetate in the 1.0 ml. sample is found to be:

$$(0.1765 \frac{\text{moles}}{\text{liter}}) \times \frac{1 \text{ liter}}{1000 \text{ ml.}} \times 1 \text{ ml. sample} = 0.0001765 \text{ moles}$$

Since the dilute feed sample is 51.0 ml., the moles of Sodium Acetate in the concentrated solution is determined as:

$$\frac{1.765 \times 10^{-4} \text{ moles Sodium Acetate}}{1.0 \text{ ml. dilute solution}} \times \frac{51.0 \text{ ml. dilute solution}}{1.0 \text{ ml. concentrated solution}} =$$

0.009 moles

. . . which is equivalent to 0.009 moles of Acetic Acid in the concentrated solution.

The volume of 0.009 moles of Acetic Acid in the 1.0 ml. concentrated solution is determined as:

$$0.009 \text{ moles HAC} \times \frac{60.052 \text{ gms. HAC}}{\text{gm. -mole HAC}} \times \frac{1 \text{ ml.}}{1.047708 \text{ gms. HAC}} =$$

0.51594 ml. HAC

Since the solution is two phase, the volume of Water is determined as:

$$1.0 \text{ ml total solution} - 0.51594 \text{ ml. HAC} = 0.48406 \text{ ml. H}_2\text{O}$$

The moles of Water in solution is thereby determined as:

$$0.48406 \text{ ml. H}_2\text{O} \times \frac{1.00013 \text{ gms. H}_2\text{O}}{\text{ml.}} \times \frac{1 \text{ gm. -mole}}{18.016 \text{ gms. H}_2\text{O}}$$
$$= 0.026872 \text{ moles of H}_2\text{O in solution}$$

Since the number of moles of each component have been determined, the mole fraction of Acetic Acid in solution can be expressed as:

$$x_{\text{HAC}} = \text{moles HAC} / (\text{moles HAC} + \text{moles H}_2\text{O})$$

By direct substitution:

$$x_{\text{HAC}} = 0.009 / (0.009 + 0.026872)$$
$$= 0.2509$$

The Water composition is determined by subtraction to be:

$$x_{\text{H}_2\text{O}} = 1 - 0.2509 = 0.7491$$

SAMPLE CALCULATION
DETERMINATION OF FEED SAMPLE pH

The equilibrium constant K_B has been shown to be:

$$K_B = (C_{OH^-})^2 / (C_{CH_3CO_2^-})$$

(for complete derivation, refer to the Appendix).

For the Sodium Acetate/Water solution, K_B has been determined to have a value of 5.6×10^{-10} . Since the molar Sodium Acetate concentration is 0.1765 moles/liter (Table 2), direct substitution into the above expression indicates the Hydroxide ion concentration to be:

$$\begin{aligned} C_{OH^-} &= (5.6 \times 10^{-10} \times 0.1765)^{\frac{1}{2}} \\ &= 9.94183 \times 10^{-6} \frac{\text{moles}}{\text{liter}} \end{aligned}$$

By definition:

$$pOH = -\text{LOG}_{10} C_{OH^-}$$

By direct substitution, the pOH is found to be:

$$\begin{aligned} pOH &= -\text{LOG}_{10}(9.94183 \times 10^{-6}) \\ &= 5.00253 \end{aligned}$$

The pH is defined as:

$$pH = 14.00 - pOH$$

By substitution of the calculated value of pOH, the pH of the solution is determined as:

$$\begin{aligned} pH &= 14.00 - 5.00253 \\ &= 8.9975 \end{aligned}$$

The pH indicator, Cresol Red, has an upper limit of 8.8, which is reasonably close to the above calculated pH value.

SAMPLE CALCULATION
CALCULATION OF APPARENT COLUMN EFFICIENCY

Before the column efficiency can be estimated, the overhead vapor compositions (y_N) must be determined. From experimental data, it can be seen that samples 5 and 6 exhibit the greatest range of concentration, hence these will be used for this analysis.

Making the assumption that the overhead vapor and feed are in equilibrium (this assumes a small change in concentration on the top tray), the vapor phase Water composition determined from the equilibrium curve is $y_{N-H_2O} = 0.82846$. From the data, the ratio of the components in the condensed overhead is seen to be:

$$\frac{y_{H_2O}}{y_{HAC}} = \frac{0.83882}{0.16118} = 5.20424$$

Assuming that this ratio also applies to the overhead vapor, the vapor phase Acetic Acid concentration is determined as:

$$y_{N-HAC} = \frac{0.82846}{5.20424} = 0.15919$$

The component material balance has been shown to be:

$$L_{N+1}x_{N+1} = L_1x_1 + V_Ny_N$$

. . . for each component. Direct substitution of the feed flowrate and compositions, bottoms composition, and vapor phase composition will yield the two equations:

$$(0.7538)(0.2509) = L_1(0.3965) + V_N(0.15919)$$

$$(0.7538)(0.7491) = L_1(0.6035) + V_N(0.82846)$$

. . . for Acetic Acid and Water respectively. Solving the equations for L_1 and V_N will give the values:

$$L_1 = 0.38126 \text{ moles/hr}$$

$$V_N = 0.62648 \text{ moles/hr}$$

The value of f can now be determined for both the Acetic Acid and Water. The f factor is, by definition:

$$f = L_1 x_1 / L_{N+1} x_{N+1}$$

Direct substitution will yield the following:

$$f = \frac{(0.38126)(0.3965)}{(0.7538)(0.2509)} = 0.80$$

. . . for Acetic Acid, and

$$f = \frac{(0.38126)(0.6035)}{(0.7538)(0.7491)} = 0.4075$$

. . . for Water.

Calculation of the Effective Stripping Factor

Stage N. Assuming once more a negligible change in concentration, tray N can be assumed to have essentially the same composition as the feed, hence $x_{N-H_2O} = 0.7491$. From the equilibrium data, $y_{N-H_2O} = 0.82846$. Direct substitution into the equation developed in the text for the stripping factor:

$$S_N = \frac{(0.62648)(0.82846)}{(0.7538)(0.7491)} = 0.92$$

Stage 1. The Water composition has been determined to be $x_{1-H_2O} = 0.6035$. From the equilibrium data, $y_{1-H_2O} = 0.71327$. The stripping factor is therefore:

$$S_1 = \frac{(0.12494)(0.71327)}{(0.38126)(0.6035)} = 0.3873$$

The effective stripping factor is therefore:

$$\begin{aligned} S_{\text{EFF}} &= (0.92(1+0.3873) + 0.25)^{0.5} - 0.5 \\ &= 0.7354 \end{aligned}$$

Substitution into the equation developed in the text ($f = (1-S)/(1-S^{N+1})$), using the effective stripping factor and the value of f determined from the experimental data, will upon rearrangement give the equation for $(N+1)$ to be

$$\begin{aligned} (N+1) &= \ln \left(1 - \frac{1-S}{f} \right) / \ln S \\ &= \ln \left(1 - \frac{1-0.7354}{0.4075} \right) / \ln 0.7354 \end{aligned}$$

$$N+1 = 3.41$$

$$N = 2.41 \text{ Theoretical Stages}$$

The apparent column efficiency is therefore:

$$\eta = \frac{2.41}{17.00} \times 100 = 14.18\%$$

NOMENCLATURE

- B = heavy or extract product rate; moles, weight, or volume per unit time.
- C_1, C_2, C_3, C_4 = constant coefficients in solutions of difference equations.
- D = light or raffinate product rate; moles, weight, or volume per unit time.
- E = operator in difference equation. For example,
 $E(x) = x_{n+1}$, $E^2(x) = x_{n+2}$, etc.
- f = fraction of a given component which is recovered in B (or B + S_E in extraction).
- F = upper feed rate.
- F^1 = lower feed rate. If only one feed is used, $F^1=0$.
- $g = S_E x_{S_E} / L_1 x_1$ = fraction of a given component in L_1 which is removed with S_E .
- $h = (L/L^1) [(1 - S_n)/(1 - S_m)]$ if intermediate feed is similar to light (vapor) or raffinate phase ($K_{M+1} = K$). Use $h = (K^1/K)(L/L^1) [(1 - S_n)/(1 - S_m)]$ if intermediate feed is similar to heavy (liquid) of extract phase ($K_{M+1} = K^1$). If the column contains only one section, $h = 1.0$.
- $K = y/x$ = distribution coefficient. Exponent 1 denotes lower or extract section of column.
- L = heavy or extract phase rate; moles, weight, or volume per unit time.
- m = any stage in lower or extract end of column (stages 1 to M).
- M = number of theoretical stages below the feed stage. Feed stage is M + 1.
- MW_i = molecular weight of component i.
- n = any stage in upper or raffinate end of column (stages M + 2 to N). If M = 0, n refers to any stage in column.

N = total number of theoretical stages including partial reboiler if any but excluding partial condenser if any.

P = absolute pressure, PSIA

q_s = fraction of a given component which enters in the fresh solvent stream S or in the stream L_{N+1} if raffinate reflux is not used.

q_F^1 = fraction of a given component which enters in the lower feed F^1 .

q_F = $1 - q_s - q_F^1$ = fraction of a given component which enters in the upper feed F .

R = external reflux ratio at top or raffinate end of column; amount refluxed/amount of product D .

R^1 = external reflux ratio at bottom or extract end of column; amount refluxed/amount of product B .

R'' = ideal gas law constant; $10.73 \text{ PSIA} \cdot \text{ft.}^3/\text{\#-mole} \cdot ^\circ\text{R}$.

S = fresh solvent rate; moles, weight, or volume per unit time. Also, S is used for effective stripping factor when $S_n = S_m$.

S_E = solvent-rich material recovered in the extract solvent-recovery equipment; moles, weight, or volume per unit time.

S_n = KV/L = effective component stripping factor in upper or raffinate end of column. The K , V , and L are representative averages for the section.

S_m = K^1V^1/L^1 = effective component stripping factor in lower or extract end of column. The K^1 , V^1 , and L^1 are representative averages for the section.

t = temperature, $^\circ\text{F}$.

T = temperature, $^\circ\text{R}$.

V = light or raffinate phase rate; moles, weight, or volume per unit time.

x = concentration in heavy or extract phase. Units consistent with units on rates.

X = moles of any component in liquid per mole of liquid entering absorber or stripper.

y = concentration in light or raffinate phase. Units consistent with units on rates.

Y = moles of any component in vapor per mole of vapor entering absorber or stripper.

ρ_V = vapor density, #/ft.³.

ρ_L = liquid density, #/ft.³.

σ = liquid surface tension, dynes/cm

η = apparent column efficiency, %

REFERENCES

1. A.I.Ch.E.J., Smith, B. D. and Brinkley, W. K., 6, 446 (1960).
2. Aust. J. Sci. Res., Brown, I. and Ewald, A. H., A3, 306 (1950)
3. Chemistry: A Conceptual Approach, Mortimer, C. E., 2nd ed., pg. 220-226. Reinhold Publishing Corp., 1967.
4. Chemical Engineer's Handbook, Perry, R. H. and Chilton C. H., 5th ed. McGraw-Hill Book Company, 1973.
5. Corrosion Data Survey, Hamner, N. E., 1st ed. National Association of Corrosion Engineers, 1974.
6. Design Of Equilibrium Stage Processes, Smith, B. D. 1st ed., pg. 252-275, pg. 544-555. McGraw-Hill Book Company, 1963.
7. Encyclopedia of Chemical Processing and Design, Volume 1, pgs. 216-240. McKetta, J. J.. Marcel Dekker, Inc., 1976.
8. Ions In Aqueous Systems, Moeller, T., and O'Conner, R., pg. 76-77. McGraw-Hill Book Company, 1972.