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# Design and Control of a Two-Column Azeotropic Distillation System

ARTICLE *in* INDUSTRIAL & ENGINEERING CHEMISTRY PROCESS DESIGN AND DEVELOPMENT · JANUARY 1985

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variation of the column capacity. Nevertheless the SRR diminishes from 43% to 37%.

If these results are compared with those obtained from the ethylbenzene/styrene separation (example 2) in which the column rates are 60% of example 1, we come to the following conclusions.

1. The parameters having the most influence on the determination of the profitability of the vapor recompression heat pump applied to an existing distillation column are: (i) the temperature difference between top and bottom, (ii) the column capacity, and (iii) the need of preheating.

2. The smaller the temperature difference between top and bottom is, the more profitable the heat pump application will be, for the following reasons: (i) small temperature differences indicate difficult separations with high reflux ratios, and consequently, high steam and cooling water consumptions, and (ii) the pressure ratio and the compression power which are required increase with the increase of temperature difference.

3. The larger the column is, the more favorable will be the cost reduction of the main equipment referred to the proportional increase in energy saving.

4. The smaller the preheating is, the more interesting the heat pump application will be, because: (i) there is a reduction in the size and cost of E-1, (ii) the compression power decreases as the inlet temperature of the compressor decreases, and (iii) the steam consumption in E-6 decreases if the condensate is allowed to give more heat in the reboiler-condenser E-2 by subcooling.

#### Concluding Remarks

Calculation programs of simulation and optimization have been developed which allow us to analyze the eco-

nomical viability of substituting the conventional reboiler and condenser of a distillation column by a vapor recompression heat pump. For the two cases presented the POT is lower than 2.5 years.

The comparison among the above presented cases and other similar ones which have been studied by us (Flores, 1983), indicates that the parameters showing the position of the optimum for the same system are relatively stable in front of the variation of the column capacity, the ratio of energy prices/equipment costs, and the relation between cooling water and electricity cost. However, the value of the POT in the optimum is very sensitive to the variation of these factors, and therefore from this point of view it is necessary to take into the account the possible re-using of the conventional equipment to be substituted, with which the required additional investment would be reduced.

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Received for review July 6, 1983

Accepted March 26, 1984

This work was financially supported by Dow Chemical Iberica, S.A.

## Design and Control of a Two-Column Azeotropic Distillation System

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The steady-state design of a two-column azeotropic distillation system operating at two different pressures was studied with the objective of reducing energy consumption. The minimum-boiling, homogeneous binary azeotropic system tetrahydrofuran-water was used as a specific example. Energy consumption was reduced by a factor of 2 from conventional designs by using heat integration and feed preheat and by optimizing column pressures and overhead purities. The dynamics and control of the two-column, heat-integrated system was also explored. A control system was developed that effectively handled a variety of disturbances without any severe interactions between columns.

#### Introduction

The design of azeotropic distillation systems has been the subject of many papers and books (Hoffman, 1964). The use of two columns, operating at two different pressures, is one of the simplest and most economical techniques for separating binary azeotropes (Van Winkle, 1967), provided that a substantial shift in the composition of the azeotrope occurs when pressure is changed.

For a minimum-boiling homogeneous binary azeotrope the distillate from the low-pressure column is fed to the high-pressure column (see Figure 1). The distillate from the high-pressure column is recycled back to the low-

pressure column. These distillates have compositions that are close to the azeotropic compositions at their respective pressures.

More or less pure products are removed from the bottom of each column. In the tetrahydrofuran (THF)-water system, water is produced from the base of the low-pressure column. THF, the low-boiling component, is produced from the bottom of the high-pressure column.

While the steady-state design of these systems has been discussed, at least qualitatively, in the literature, there has been very little reported concerning the dynamics and control of the two-column system. Shinsky (1977) gave

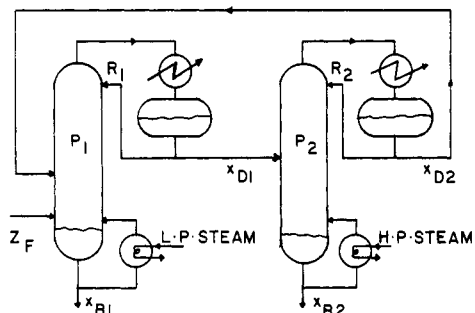


Figure 1. Minimum-boiling homogeneous azeotropic two-column system.

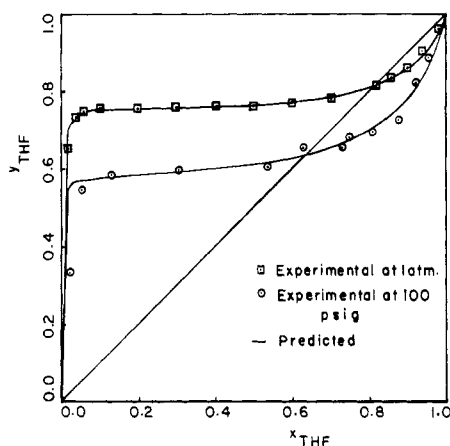


Figure 2. Experimental and predicted vapor-liquid equilibria for the THF-water system at 1 atm and 100 psig.

a qualitative discussion of the sensitivity of the column material balances to overhead compositions and warned of a "snowball" effect where the recycle flows between columns continued to grow until column flooding occurred. Shinskey proposed the use of steam to feed ratios to prevent this runaway problem, but no other details of the control system were given, nor was its performance quantitatively evaluated.

The purposes of this work are (1) to optimize the steady-state design of a two-column, THF-water system, primarily in terms of minimizing energy consumption, and (2) to develop and evaluate quantitatively via digital simulation a control system capable of handling a variety of disturbances.

### THF-Water System

THF is widely used in the chemical industry as a solvent and reaction medium. Typical THF recovery methods involve adsorption on activated carbon, followed by a steam stripping for regeneration of the bed. The resulting THF-water mixture cannot be separated into pure components in a single column because a minimum-boiling azeotrope is formed (5.3 wt % water at atmospheric pressure). Since the azeotropic composition shifts with pressure (12.0 wt % water at 100 psig), a two-column, two-pressure system is commonly used to produce very pure water and dry THF for recycle back to the process.

Physical property and vapor-liquid equilibrium data for the THF-water system at various pressures were obtained from literature and industrial sources (see Abu-Eishah, 1982, for details). The Wilson equation was used for estimating activity coefficients. Figure 2 compares predicted and experimental  $x$ - $y$  curves at 1 atm and 100 psig.

### Steady-State Optimization

Nonideal, non-equimolar overflow steady-state design and rating programs were developed to explore the effects

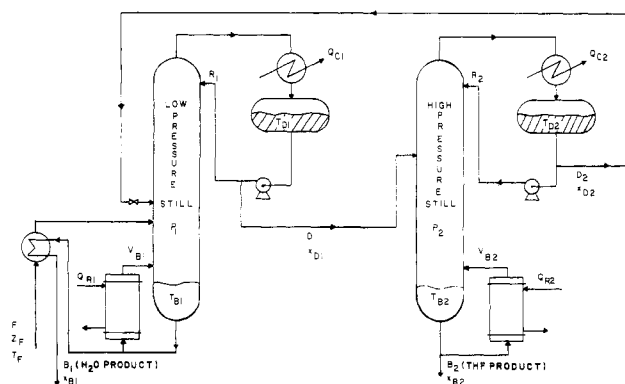


Figure 3. Typical conventional configuration.

of various design and operating parameters on energy consumption of the two-column system. Figure 3 shows the typical configuration which includes a feed preheater. The first column operates at  $P_1$ , while the second column operates at a higher pressure  $P_2$ . These two pressures are typically 1 atm and 100 psig, respectively, in present commercial plants.

**A. Overall Material Balances.** The flow rates  $D_1$  and  $D_2$  between the two columns are very sensitive to the overhead compositions. Using the nomenclature of Figure 3, total and component balances for each column and for the entire system can be written

column 1:

$$F + D_2 = D_1 + B_1 \quad (1)$$

$$FZ_F + D_2X_{D2} = D_1X_{D1} + B_1X_{B1} \quad (2)$$

column 2:

$$D_1 = D_2 + B_2 \quad (3)$$

$$D_1X_{D1} = D_2X_{D2} + B_2X_{B2} \quad (4)$$

total system:

$$F = B_1 + B_2 \quad (5)$$

$$FZ_F = B_1X_{B1} + B_2X_{B2} \quad (6)$$

Combining eq 5 and 6 gives

$$B_1 = F \frac{X_{B2} - Z_F}{X_{B2} - X_{B1}} \quad (7)$$

Combining eq 3 and 4 gives

$$D_2 = D_1 \frac{X_{D1} - X_{B2}}{X_{D2} - X_{B2}} \quad (8)$$

Now substituting eq 7 and 8 into eq 2 gives

$$D_1 = F \frac{(Z_F - X_{B1})(X_{D2} - X_{B2})}{(X_{B2} - X_{B1})(X_{D2} - X_{D1})} \quad (9)$$

For very pure products,  $X_{B1} = 0$  and  $X_{B2} = 1$ , which reduces eq 9 to

$$D_1 = FZ_F \frac{1 - X_{D2}}{X_{D1} - X_{D2}} \quad (10)$$

and eq 8 to

$$D_2 = FZ_F \frac{1 - X_{D1}}{X_{D1} - X_{D2}} \quad (11)$$

Notice that as  $X_{D1}$  approaches  $X_{D2}$ , the recycle flow rates between the columns become very large. This corresponds to high energy consumptions in each column ( $Q_{R1}$  and  $Q_{R2}$ ). Since  $X_{D1}$  and  $X_{D2}$  are fairly close to the azeotropic com-

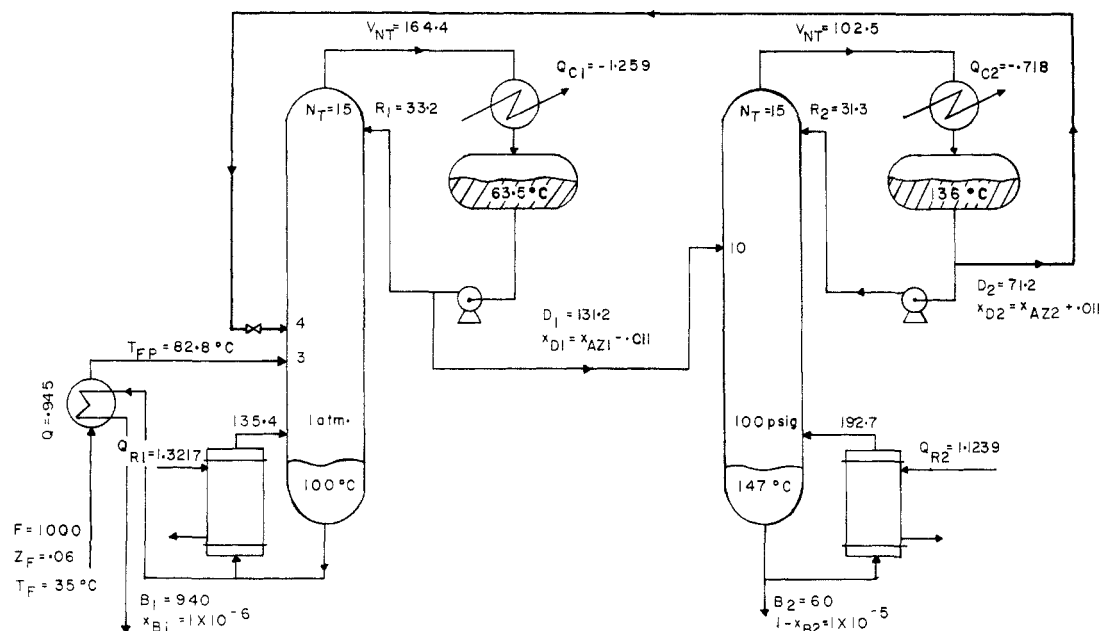


Figure 4. Flowsheet for the base case  $Q_T = 2.446 \times 10^6$  cal/h (all  $Q$ 's are in  $10^6$  cal/h).

Table I. Base Case Parameters for Rating Study

column 1	column 2
$P_1 = 760$ mmHg	$P_2 = 100$ psig
$F = 1000$ g-mol/h	$1 - X_{B2} = 1 \times 10^{-4}$ m.f. $H_2O^a$
$Z_F = 0.06$ m.f. THF <sup>a</sup>	
$T_F = 35$ °C (before preheater)	$N_T = 15$
$X_{B1} = 1 \times 10^{-6}$ m.f. THF <sup>a</sup>	$N_F = 10$
$N_T = 15$	
$N_{F1} = 3$	
$N_{F2} = 4$	

<sup>a</sup> m.f. = mole fraction.

positions,  $X_{AZ1}$  and  $X_{AZ2}$ , the pressures should be adjusted to maximize the difference between  $X_{AZ1}$  and  $X_{AZ2}$ . This suggests that the pressure in column 1 should be minimized and the pressure in column 2 should be maximized. We will show later that this is true up to a point. There is an optimum pressure in the high pressure column for a heat-integrated system.

**B. Effect of Parameters on Column Design.** The number of trays required in each column was calculated by the design program for various feed compositions, distillate purities, bottoms purities, column pressures, and ratios of actual to minimum reflux ratio (see Abu-Eishah, 1982, for details).

The effect of all these parameters on the total number of trays required in each column was fairly small. The number of trays ranged from 10 to 20 unless very low reflux ratios or very high purities were specified.

Therefore, 15 theoretical tray columns were used in the rating program. This corresponded to using a 1.2 ratio of actual to minimum reflux ratio. Table I gives details of the base case parameters for the rating study.  $\Delta X_1$  and  $\Delta X_2$  are the absolute differences between the specified distillate compositions and the azeotrope compositions for column 1 and column 2, respectively.

**C. Distillate Composition.** With the composition of the distillate in column 2 fixed at  $\Delta X_2 = 1.1$  mol %, as the distillate composition of the first column becomes purer (closer to the azeotrope), more reflux and heat input are required in column 1 (see Table II). However, the distillate flow rates from both columns decrease, which reduces the heat input to column 2. Therefore, the total heat input  $Q_T$  has a minimum.

$$Q_T = Q_{R1} + Q_{R2} \quad (12)$$

Table II shows that the minimum occurs at a value of  $\Delta X_1$  of 1.1 mol % away from the azeotrope. Notice that this minimum is quite flat, implying that overhead purity is not too critical.

Table III shows that the optimum column 2 overhead purity is also 1.1 mol %, with a quite flat optimum. Figure 4 gives process conditions for this "base case" with  $\Delta X_1 = \Delta X_2 = 1.1$  mol %. These are the optimum overhead purities with  $P_1 = 760$  mmHg and  $P_2 = 100$  psig. The effect of pressure on these optimum purities will be discussed later.

**D. Feed Composition.** Table IV shows that all flow rates, except the bottoms from column 1 ( $B_1$ ), increase as

Table II. Effect of Column 1 Distillate Composition (with  $\Delta X_2 = 1.1$  mol %)

column 1				column 2			
$\Delta X_1$ , mol %	$R_1$ , g-mol/h	$D_1$ , g-mol/h	$Q_{R1}$ , $10^6$ cal/h	$R_2$ , g-mol/h	$D_2$ , g-mol/h	$Q_{R2}$ , $10^6$ cal/h	$Q_T$ , $10^6$ cal/h
0.8	39.6	128.8	1.3571	30.9	68.8	1.0981	2.4552
0.9	37.2	129.6	1.3433	31.0	69.6	1.1066	2.4499
1.0	35.1	130.4	1.3317	31.2	70.4	1.1152	2.4469
1.1	33.2	131.2	1.3217	31.3	71.2	1.1239	2.4456 <sup>a</sup>
1.2	31.4	132.1	1.3131	31.4	72.1	1.1327	2.4458
1.3	29.8	132.9	1.3057	31.6	72.9	1.1416	2.4483
1.4	28.4	133.8	1.2993	31.7	73.7	1.1507	2.4500
1.5	27.0	134.6	1.2938	31.8	74.6	1.1598	2.4536

<sup>a</sup> Minimum total energy required.

Table III. Effect of Column 2 Distillate Composition (with  $\Delta X_1 = 1.1$  mol %)

$\Delta X_2$ , mol %	column 1			column 2			
	$R_1$ , g-mol/h	$D_1$ , g-mol/h	$Q_{R1}$ , $10^6$ cal/h	$R_2$ , g-mol/h	$D_2$ , g-mol/h	$Q_{R2}$ , $10^6$ cal/h	$Q_T$ , $10^6$ cal/h
0.8	32.85	129.93	1.3130	34.85	69.93	1.1368	2.4498
0.9	32.95	130.36	1.3159	33.54	70.36	1.1316	2.4475
1.0	33.05	130.80	1.3188	32.37	70.80	1.1274	2.4462
1.1	33.15	131.24	1.3217	31.29	71.24	1.1239	2.4456 <sup>a</sup>
1.2	33.26	131.69	1.3246	30.30	71.68	1.1211	2.4457
1.3	33.37	132.14	1.3276	29.37	72.14	1.1187	2.4463
1.4	33.47	132.60	1.3306	28.49	72.60	1.1168	2.4474
1.5	33.58	133.07	1.3337	27.65	73.06	1.1152	2.4489

<sup>a</sup> Minimum total energy required.

Table IV. Effect of Feed Composition

	$Z_F$ , m.f. THF		
	0.06	0.15	0.30
$Q_{R1}$ , $10^6$ cal/h	1.322	3.024	5.862
$Q_{R2}$ , $10^6$ cal/h	1.124	2.810	5.620
$Q_T$ , $10^6$ cal/h	2.446	5.834	11.482
$Q_T/B_2$	0.0408	0.0389	0.0383
$T_{FP}$ , °C	82.8	73.8	62.4
$B_1$ , g-mol/h	940.0	850.0	700.0
$R_1$ , g-mol/h	33.2	82.1	163.6
$D_1$ , g-mol/h	131.2	328.1	656.2
$B_2$ , g-mol/h	60.0	150.0	300.0
$R_2$ , g-mol/h	31.3	78.2	156.5
$D_2$ , g-mol/h	71.2	178.1	356.2

Table V. Effect of Reduction of Column 1 Pressure ( $P_2 = 100$  psig and  $\Delta X_1 = \Delta X_2 = 1.1$  mol %)

	$P_1$ , mmHg	
	760	350
$Q_{R1}$ , $10^6$ cal/h	1.322	1.036
$Q_{R2}$ , $10^6$ cal/h	1.124	0.867
$Q_T$ , $10^6$ cal/h	2.446	1.903
$X_{D1}$ , m.f. THF	0.8097	0.8570
$R_1$ , g-mol/h	33.2	26.1
$D_1$ , g-mol/h	131.2	101.4
$R_2$ , g-mol/h	31.3	23.3
$D_2$ , g-mol/h	71.2	41.4

feed composition  $Z_F$  increases. Since  $B_1$  is used to preheat the feed, the temperature of the feed after the preheater ( $T_{FP}$ ) decreases as  $Z_F$  increases. It should be noted that the energy required ( $Q_T$ ) per mole of THF product ( $B_2$ ) decreases as  $Z_F$  increases.

**E. Column Pressures.** As mentioned earlier, reducing column 1 pressure should decrease total energy consumption. This pressure can be reduced to the point where it begins to become difficult to be able to use cooling water

in the overhead condenser. A reflux drum temperature of 42.6 °C is a practical limit with 35 °C mid-summer cooling water temperatures. This corresponds to a minimum column 1 pressure of 350 mmHg. Table V compares the 760 and 350 mmHg cases with column 2 pressure fixed at 100 psig. Distillate composition  $X_{D1}$  increases from 0.8097 to 0.8570 mole fraction of THF. This reduces distillate flow rate, reflux flow rate, and heat input in both columns.

Increasing column 2 pressure should have the same effect. The limit on how high  $P_2$  can be raised is the availability of high-temperature heat source. No high-temperature sensitivity of THF has been reported. Table VI gives the effect of  $P_2$  on flow rates and heat inputs with  $P_1$  fixed at 760 mmHg. The higher the pressure in column 2, the lower the total energy consumption  $Q_T$ .

The optimum overhead purities were calculated for the new pressures. The optimum  $\Delta X_2$  was found to be essentially independent of  $P_2$  and varied only with  $P_1$ . At  $P_1 = 350$  mmHg, the optimum  $\Delta X_2$  was 1.5 mol %. The optimum  $\Delta X_1$  increased with  $P_2$  as shown in Table VII.

In summary, we have seen that there are optimum overhead purities where total energy required by the system is minimized. Reduction of column 1 pressure and increasing column 2 pressure have major effects on minimizing total energy consumption. In the next section we will explore the optimum combinations of these pressures when energy conservation schemes are used.

**F. Heat Integration.** Since column 2 overhead temperature is higher than column 1 base temperature, it is obvious that the overhead vapor from column 2 can be used to provide at least a portion of the heat required in column 1. For example, as shown in Figure 4 for the base case ( $P_1 = 760$  mmHg and  $P_2 = 100$  psig), the condenser heat duty in column 2 ( $0.718 \times 10^6$  cal/h) at 136 °C could be transferred into the base of column 1 at 100 °C. Since

Table VI. Effect of Column 2 Pressure (with  $P_1 = 760$  mmHg and  $\Delta X_1 = \Delta X_2 = 1.1$  mol %)

$P_2$ , psig	$R_1$ , g-mol/h	$D_1$ , g-mol/h	$Q_{R1}$ , $10^6$ cal/h	$R_2$ , g-mol/h	$D_2$ , g-mol/h	$Q_{R2}$ , $10^6$ cal/h	$Q_T$ , $10^6$ cal/h
50	42.1	171.2	1.6772	57.1	111.2	1.5687	3.2459
100	33.2	131.2	1.3217	31.3	71.2	1.1239	2.4456
150	30.1	118.0	1.2027	20.6	58.0	0.9882	2.1909
200	28.5	111.3	1.1411	14.7	51.3	0.9315	2.0726
250	27.5	107.2	1.1026	10.9	47.2	0.9059	2.0085
300	26.9	104.4	1.0759	8.3	44.4	0.8954	1.9713

Table VII. Optimum Distillate Purities at  $P_1 = 350$  mmHg and  $\Delta X_2 = 1.5$  mol %

$P_2$ , psig	$X_{D1}$ , m.f. THF	$X_{D2}$ , m.f. THF	opt. $\Delta X_1$ , m.f. THF	$Q_{R1}$ , $10^6$ cal/h	$Q_{R2}$ , $10^6$ cal/h	$Q_T$ , $10^6$ cal/h
50	0.856	0.681	1.2	1.1768	1.0801	2.2569
100	0.852	0.624	1.6	1.0008	0.8898	1.8906
150	0.850	0.587	1.8	0.9321	0.8339	1.7660
200	0.846	0.542	2.0	0.8907	0.8204	1.7111
250	0.845	0.527	2.1	0.8657	0.8192	1.6849

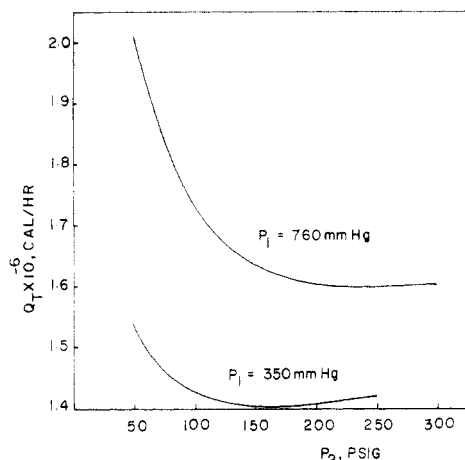


Figure 5. Total energy required with heat integration as a function of pressure.

the total heat required in column 1 is  $1.3217 \times 10^6$  cal/h, the net energy that would have to be provided in the steam reboiler would be reduced from  $1.3217 \times 10^6$  to  $0.6037 \times 10^6$  cal/h by heat integration.

Table VIII gives energy consumptions for various column pressures for the heat integrated columns when  $Q_{c2}$  is subtracted from  $Q_{R1} + Q_{R2}$  to get  $Q_T$ .

Notice that there is an optimum column 2 pressure at 150 psig for the case where  $P_1 = 350$  mmHg. This occurs because of the reduction of the  $Q_{c2}/Q_{R1}$  ratio as  $P_2$  is increased. Although the temperature of the overhead vapor from column 2 increases as  $P_2$  increases, the flow rate decreases. Thus the energy in column 2 overhead that is available to reboil column 1 decreases as  $P_2$  is increased. There are two opposing effects. As  $P_2$  is increased, the energy required in each reboiler decreases, but the energy available for heat integration also decreases. This leads to an optimum  $P_2$  where total net energy is minimized. Figure 5 shows this result graphically. We will call this  $P_1 = 350$  mmHg and  $P_2 = 150$  psig case the "optimum pressure" case. The optimum overhead purities for this case are  $\Delta X_1 = 1.8$  mol % and  $\Delta X_2 = 1.5$  mol %.

**G. Heat Economizers.** Up to this point we have considered only a single feed preheater using column 1

Table VIII. Energy Consumption of the Heat Integrated System as a Function of Column 2 Pressure (at Optimum  $\Delta X_1$ )

$P_2$ , psig	$Q_{R1}$ , $10^6$ cal/h	$Q_{c2}$ , $10^6$ cal/h	$Q_{c2}/Q_{R1}$	$Q_T$ , $10^6$ cal/h
(a) $P_1 = 350$ mmHg, $\Delta X_2 = 1.5\%$				
50	1.1768	-0.7216	0.6132	1.5353
100	1.0008	-0.4642	0.4638	1.4263
150	0.9321	-0.3572	0.3832	1.4088 <sup>a</sup>
200	0.8907	-0.3001	0.3369	1.4110
250	0.8657	-0.2615	0.3021	1.4234
(b) $P_1 = 760$ mmHg, $\Delta X_2 = 1.1\%$				
50	1.7228	-1.1636	0.6754	2.0631
100	1.3217	-0.7179	0.5432	1.7277
150	1.1788	-0.5493	0.4660	1.6376
200	1.1044	-0.4563	0.4132	1.6075
250	1.0600	-0.3945	0.3722	1.6010 <sup>a</sup>
300	1.0279	-0.3512	0.3417	1.6036

<sup>a</sup> Minimum total energy required with heat integration.

bottoms. More energy can be saved by using additional heat economizers.

For the base case ( $P_1 = 760$  mmHg and  $P_2 = 100$  psig), column 1 feed can also be preheated by column 1 overhead vapor (see Figure 6). Column 2 bottoms can also be used to preheat column 2 feed. Total energy consumption is reduced from  $1.73 \times 10^6$  to  $1.48 \times 10^6$  cal/h by use of these additional preheaters in addition to heat integration.

For the optimum pressure case ( $P_1 = 350$  mmHg and  $P_2 = 150$  psig), column 1 overhead vapor cannot be used economically to preheat the feed because of the low temperature differential ( $T_F$  is  $35^\circ\text{C}$  and column 1 reflux drum temperature is  $42.6^\circ\text{C}$ ). Figure 7 shows that a preheater on column 2 can be used, reducing total energy consumption from  $1.409 \times 10^6$  to  $1.11 \times 10^6$  cal/h with heat integration.

Abu-Eishah (1982) also evaluated the use of vapor recompression but found that this alternative was less attractive than heat integration.

In summary, the use of optimum pressures, heat integration, and heat economizers has reduced the energy consumption from the typical base case level of  $2.446 \times 10^6$  cal/h to  $1.11 \times 10^6$  cal/h, a reduction of more than 50%. In the next section we will explore the dynamics and

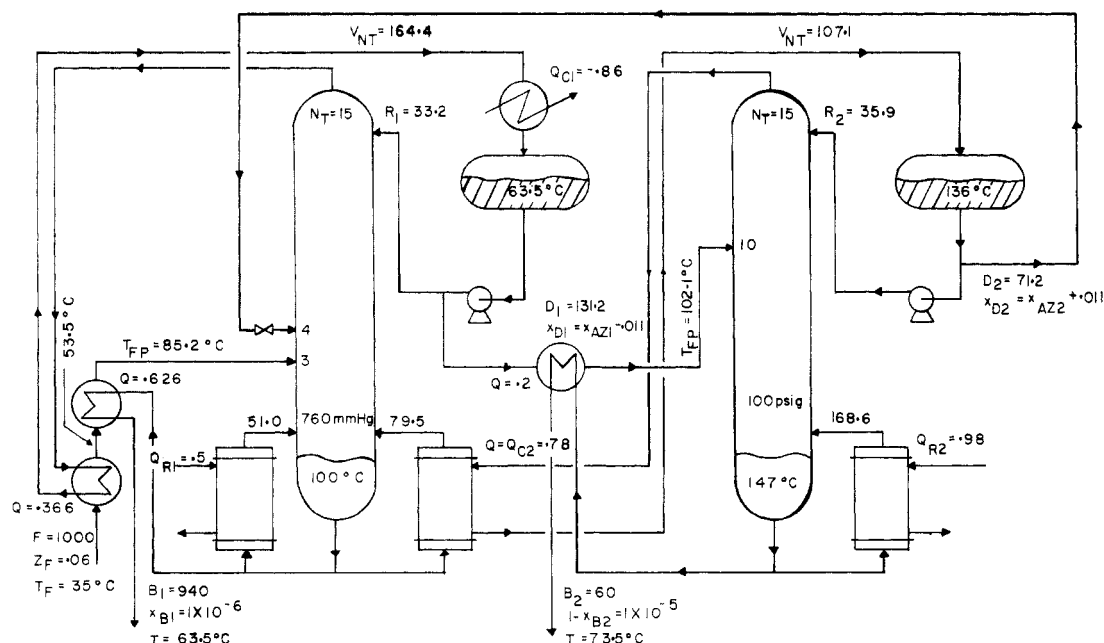


Figure 6. Flowsheet and energy requirement for base case ( $P_1 = 760$  mmHg,  $P_2 = 100$  psig) using both energy integration and heat economizers,  $Q_T = 1.480 \times 10^6$  cal/h.

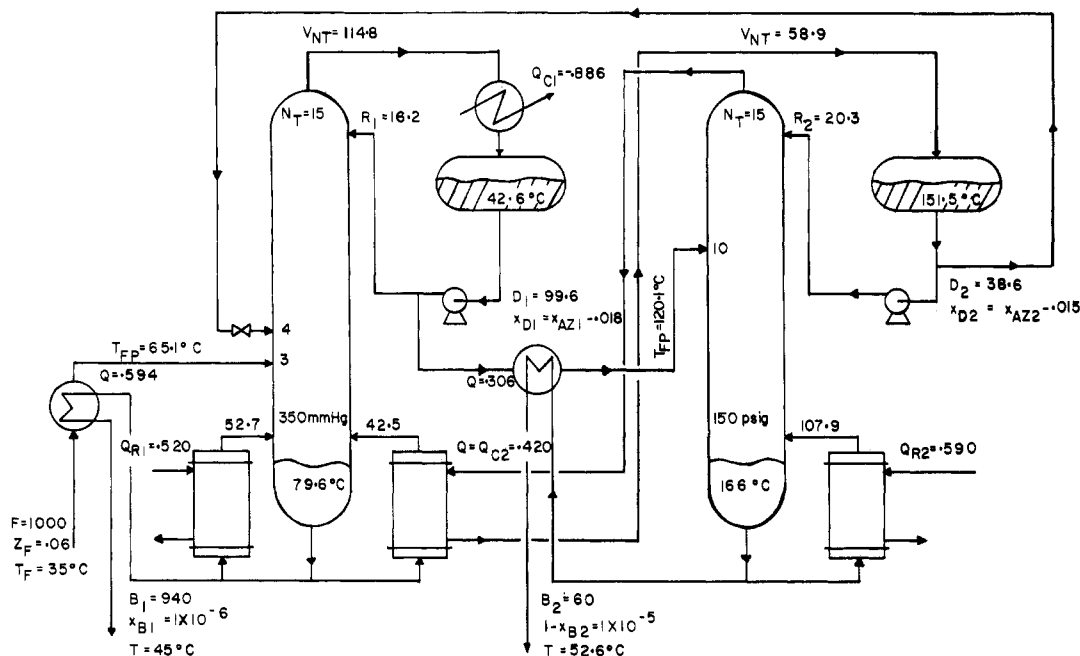


Figure 7. Flowsheet and energy requirement for optimum case ( $P_1 = 350$  mmHg,  $P_2 = 150$  psig) using both energy integration and heat economizers,  $Q_T = 1.110 \times 10^6$  cal/h.

Table IX. Columns and Tray Specifications

	column 1	column 2
column pressure, psig	0	100
feed: temperature, °C	85.20	102.1
composition, m.f. THF	0.06	0.8097
recycle: temperature, °C	135.85	—
composition, m.f. THF	0.6495	—
column diameter, cm	161.74	118.24
tray weir length, cm	117.17	85.13
tray weir height, cm	5.08	5.08
range of tray holdups, kg mol	1.69–8.93	0.95–1.05
holdup in reflux drum, kg mol	26.60	17.55
holdup in the column base, kg mol	178.43	39.36

control of the heat integrated system.

### Dynamics and Control

The two-column system studied in this work is highly intercoupled and interacting. Any disturbance that occurs in one column is directly transmitted into the other column through distillate flows. For the heat-integrated columns, there is the additional coupling of energy inputs between column 2 and column 1.

The dynamic mathematical models of the two columns had to be solved simultaneously (see Abu-Eishah, 1982, for details on modeling and simulation procedures followed). A 15-s temperature measurement lag was assumed. Holdup times of 5 min were used in reflux drums and column bases.

Table IX gives steady-state data and dimensions for the system chosen to be studied which was the base case with heat integration and heat economizer (Figure 6). This case was selected because it is the state of the art in industry where the results of this study could be applied. We expect little difference in dynamics and controllability from the optimum pressure case which also features heat integration and economizers.

**A. Overall Control Objectives.** The objectives of the control system are to keep the compositions of the two bottoms products (water from column 1 and THF from column 2) near or above specification purity levels, while using as little energy as possible. The control system must give stable and efficient operation of this interacting

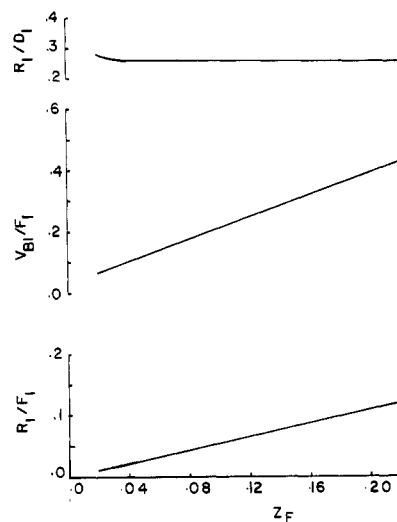


Figure 8. Calculated  $R/F$ ,  $V/F$ , and  $R/D$  ratios for column 1 as a function of its feed composition.

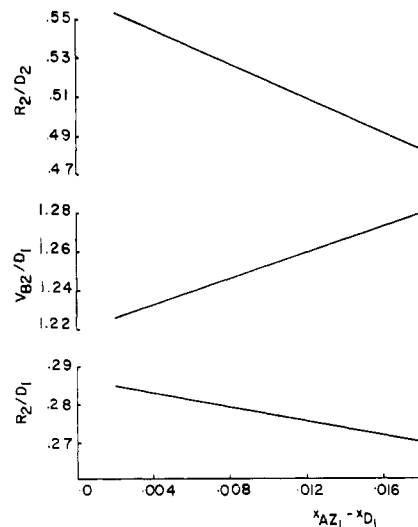


Figure 9. Calculated  $R/F$ ,  $V/F$ , and  $R/D$  ratios for column 2 as a function of its feed composition.

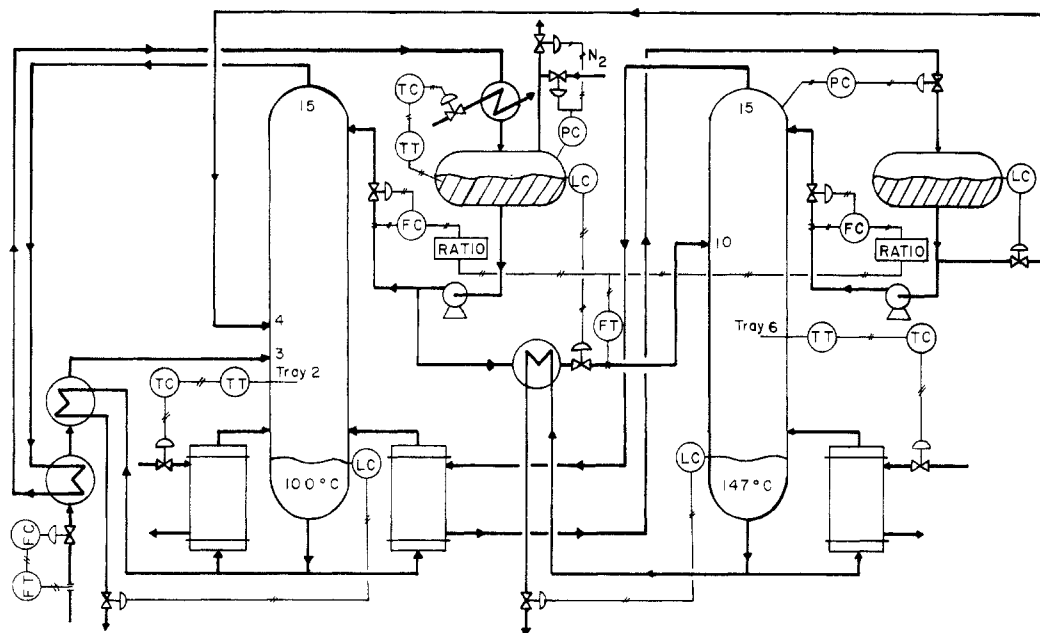


Figure 10. Constant  $R_1/D_1$  and  $R_2/D_1$  control scheme.

system for a variety of fairly large disturbances.

There are two basic variables to be controlled,  $X_{B1}$  and  $X_{B2}$ . Therefore, at least two manipulated variables are required, but there are more than two manipulated variables available: heat inputs ( $Q_{R1}$  and  $Q_{R2}$ ) and reflux flow rates ( $R_1$  and  $R_2$ ). We assumed that the two distillate flows would be used to control the two reflux drum levels with proportional-only controllers because of the low reflux ratios in both columns.

We could control the distillate compositions of each column at their optimum values ( $\Delta X_1 = \Delta X_2 = 1.1$  mol % for the base case with  $P_1 = 760$  mmHg and  $P_2 = 100$  psig). This would require dual composition control systems on each column.

As a more practical alternative, it may be almost as energy efficient to control just one composition for each column (or temperature) and maintain constant reflux to feed rate ratio, vapor boilup to feed rate ratio, or reflux ratio as suggested by Luyben (1975). The effect of these alternatives on energy consumption was determined from steady-state analysis.

The steady-state rating program was used to calculate the heat input and reflux required to maintain constant distillate and bottoms compositions in each column as feed composition varied. These results are given in Figures 8 and 9. They suggest that (a) column 1 could be run with a fixed reflux ratio ( $R_1/D_1$ ) and waste very little energy since this ratio does not vary much with feed composition, and (b) column 2 could be run with a fixed reflux to feed ratio ( $R_2/D_1$ ) for the same reason. A small amount of energy is wasted since the columns are over refluxed in order to handle the worst-case feed composition.

The final control system is shown in Figure 10. The temperatures on tray 2 in column 1 and on tray 6 in column 2 are controlled by heat inputs to the two reboilers. These trays were selected on the basis of their being near the bottom of the columns but in regions where the temperature profiles are sensitive to product composition changes (Figures 11 and 12).

As shown in Figure 10, the pressure in the lower pressure column is held by a vent-bleed system. The pressure in the high-pressure column is held by a control valve in the liquid line from the condenser. This is a flooded condenser system in which the area available for condensation is

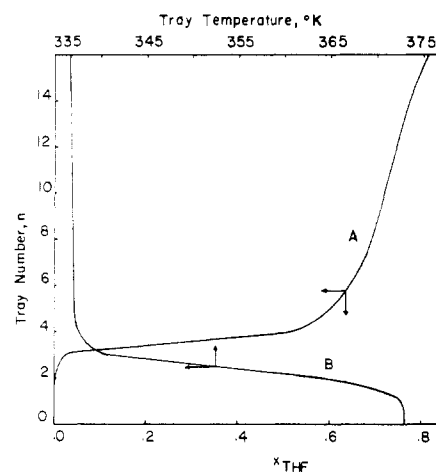


Figure 11. Composition and temperature profiles for column 1.

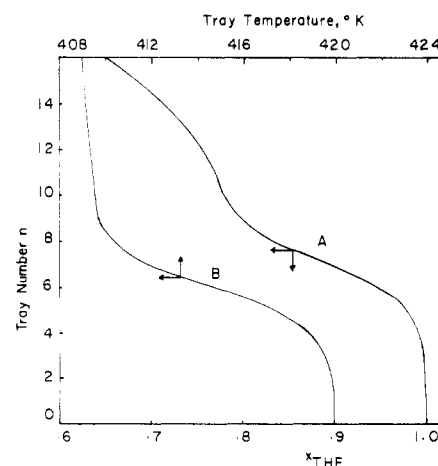


Figure 12. Composition and temperature profiles for column 2.

varied by changing the liquid height in the shell side of the reboiler/condenser to cover or expose more tubes.

Transfer functions for the two by two system were obtained by pulse testing the mathematical model. The two temperature controllers were tuned using the frequency-domain technique for proportional-integral controllers. Table X gives process transfer function data and controller



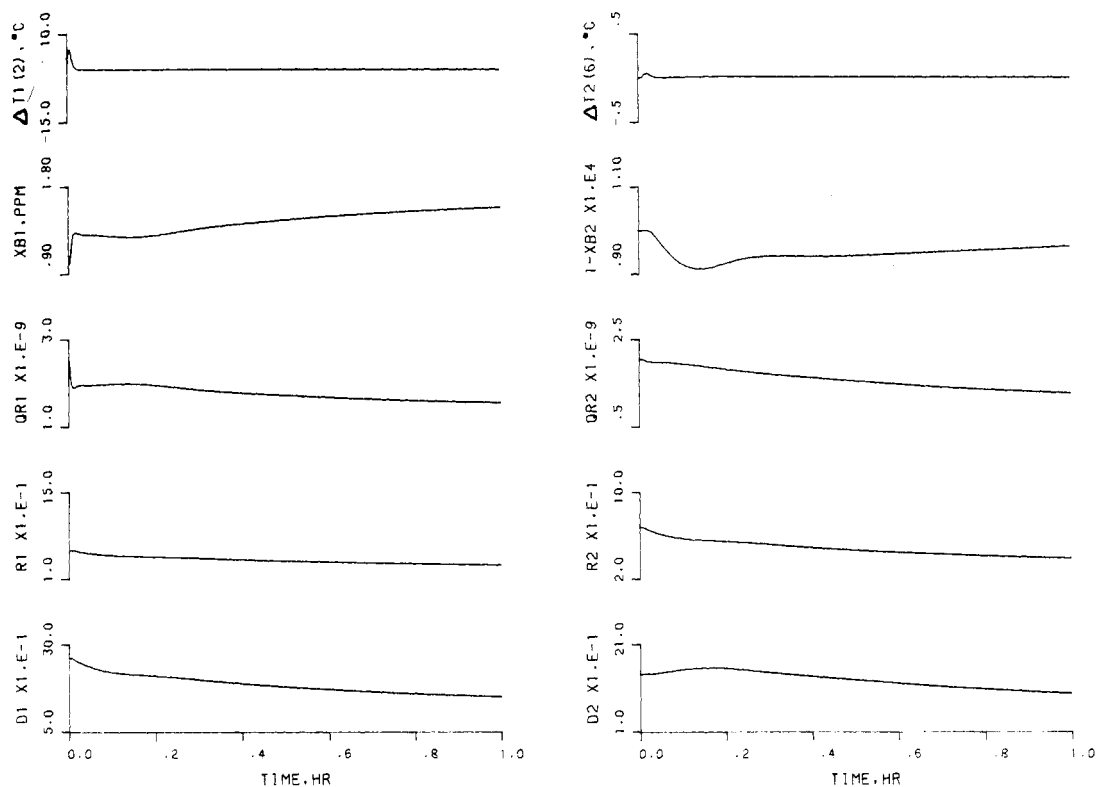


Figure 13. Response of the two-column system to -50% change in feed composition.

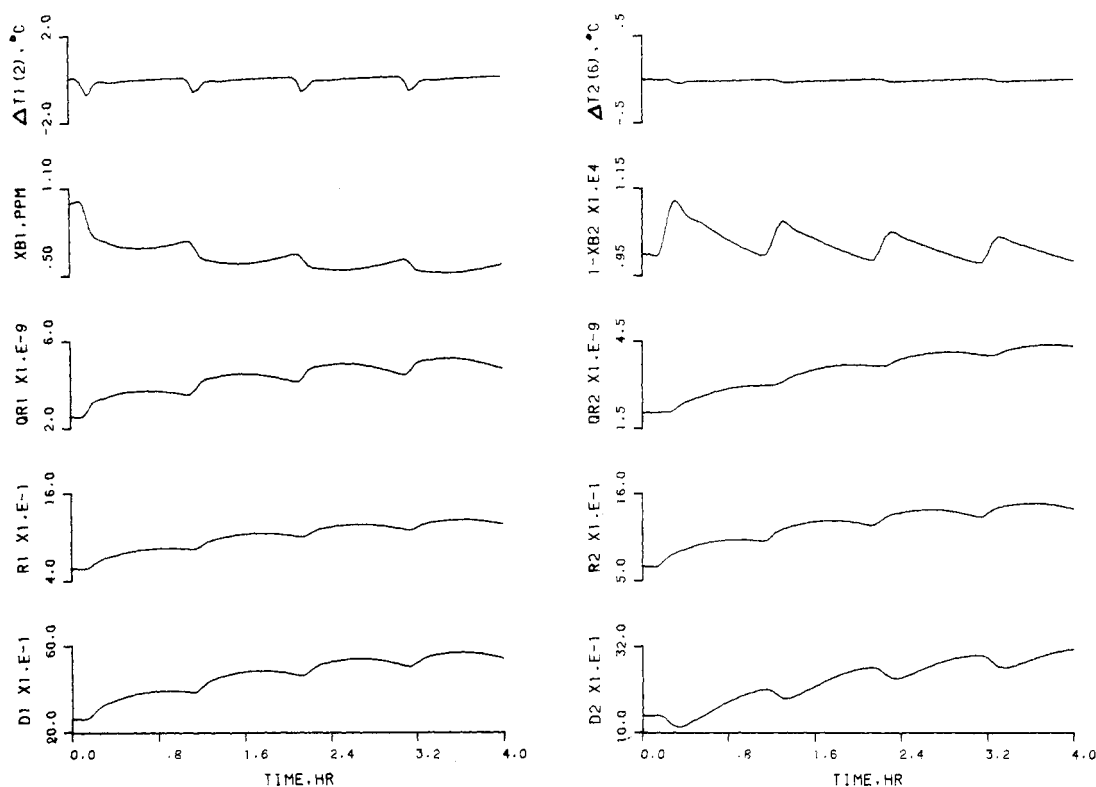


Figure 14. Response of the two-column system to a periodic change in feed composition (for a feed tank holdup of 1.5 h).

settings. All process and controller gains were made dimensionless by assuming temperature transmitter spans of 50 °C and valve gains equal to the steady-state values of reboiler duty.

The performance of the control system is shown in Figure 13 for a 50% reduction in feed composition and in Figure 14 for a periodic disturbance in feed composition that would be typical in batch regeneration by steaming out a fixed-bed THF adsorption bed.

The proposed control system handled these large disturbances very effectively. The performance of Shinskey's material balance scheme was also evaluated on the dynamic model. Tray temperatures were controlled by distillate flow rates. The performance was very poor. Only very small disturbances could be made without running into instability problems. This is to be expected because of the large delay time between a change in distillate flow rate and the response of a tray near the bottom of the

**Table X. Summary of Pulse Test Results and Controller Settings**

	column 1		column 2	
process gain	12.857		4.864	
process time constant, min	2.22		14.3	
process dead time, min	0.1048		0.1495	
ultimate gain	3.096		30.76	
ultimate frequency, rad/min	15.27		10.55	
ultimate period, min	0.412		0.596	
PI-Controller Settings				
	gain	reset	gain	reset
Ziegler-Nichols	1.408	0.343	13.843	0.537
design	1.189	0.103	13.482	0.163

column. These tray temperatures respond quickly, however, to heat input changes.

### Nomenclature

$B$  = bottoms flow rate, g-mol/h  
 $D$  = distillate flow rate, g-mol/h  
 $F$  = feed flow rate, g-mol/h  
 $N_F$  = feed tray  
 $N_T$  = total number of trays

$P$  = column pressure, mmHg or psig  
 $Q_c$  = condenser heat duty,  $10^6$  cal/h  
 $Q_R$  = reboiler heat duty,  $10^6$  cal/h  
 $Q_T$  = total energy consumption,  $10^6$  cal/h  
 $T_F$  = temperature feed before preheater, °C  
 $T_{FP}$  = temperature feed after preheater, °C  
THF = tetrahydrofuran  
 $X_{AZ}$  = azeotropic composition, mole fraction of THF  
 $X_B$  = bottoms composition, mole fraction of THF  
 $X_D$  = distillate composition, mole fraction of THF  
 $Z_F$  = feed composition, mole fraction of THF  
 $\Delta X$  = difference between azeotropic composition and distillate composition, mol % THF  
**Registry No.** THF, 109-99-9.

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Received for review July 8, 1983

Accepted January 31, 1984

## Rapid Procedures for the Prediction of Fixed-Bed Adsorber Behavior. 3. Isothermal Sorption of Two Solutes from Gases and Liquids

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We extend a simple method previously used to describe single-solute adiabatic sorption operations to *binary isothermal* systems. The method separates equilibrium and kinetic effects by first constructing the effective equilibrium pathways, followed by the application of solution charts for single-solute *nonequilibrium* sorption. Construction of the effective equilibrium curves involves the solution of a single nonlinear algebraic or differential equation. The solution charts are valid for both developing and broadening solute fronts. The procedure is used successfully in predicting solute breakthrough from clean or preloaded isothermal adsorber and ion-exchange beds taking binary gaseous and liquid feeds. The relation to existing methods of describing column behavior is established and procedures are outlined for arriving at quick design estimates and analyzing the effect of various parameters.

### Introduction

In part 2 of this series (Basmadjian, 1980a), we had used "effective equilibrium curves" in conjunction with known solutions of single-solute isothermal nonequilibrium sorption to predict actual behavior of adiabatic columns taking a feed containing one sorbable component. The curves in question are in essence solutions of the equilibrium and conservation equations of equilibrium theory; they can be obtained in simple fashion by the numerical or graphical solution of algebraic or differential equations.

Although the general validity of this approach still lacks a formal proof, we were encouraged by its ability to predict adiabatic fixed-bed adsorption and regeneration for widely diverse systems with reasonable accuracy (Basmadjian, 1980a,b; 1981). In addition to its simplicity, the method has the advantage of separating equilibrium and kinetic effects—a feature usually lacking in numerical solutions of the full PDE model—thus allowing the analyst to

identify sources of unsatisfactory performance and suggest parameter changes for process improvement. Once the effective equilibrium curves have been established for a particular feed/bed condition, design charts for the single-solute isothermal case can be applied in a few minutes' work to derive profiles and breakthrough curves or required bed weight and regenerant. Changes in feed rate, bed length, cycle time, or particle size and diffusivity are accommodated by a simple adjustment of the dimensionless chart parameters. However, the method presently is not able to accommodate variable feed conditions and nonuniform initial bed distributions.

### Theory

**1. Derivation of Effective Equilibrium Curves or Pathways.** Equations for the effective equilibrium curves are derived from the solute conservation and equilibrium equations which apply to a column with prevailing local equilibrium. The relevant relations have been given in the