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AN EXPERIMENTAL STUDY OF

THE SEPARATION OF MULTICOMPONENT MIXTURES VIA THERMAL PARAMETRIC PUMPING

BY

WAYNE WEYWEN LIN

A THESIS

PRESENTED IN PARTIAL FULFILIMENT OF

THE REQUIREMENTS FOR THE DEGREE

 \mathbf{OF}

MASTER OF SCIENCE IN CHEMICAL ENGINEERING

ΤA

NEWARK COLLEGE OF ENGINEERING

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Newark, New Jersey 1974

APPROVAL OF THESIS

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ВҮ

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FOR

DEPARTMENT OF CHEMICAL ENGINEERING NEWARK COLLEGE OF ENGINEERING

BY

FACLTY COMMITTEE

APPROVED

NEWARK, NEW JERSEY

MAY, 1974

ACKNOWLEDGMENT

I am deeply grateful to Dr. H.T.Chen, whose guidance, advice and assistance were essential in this research work. I am also thankful to Messers. J.D.Stokes and J.Park, for their help in setting up the experiment apparatus, running the experiments, and analyzing samples.

Part of this work was presented at the 4th Joint Meeting of AIChE and Canadian Society for Chemical Engineering, Vancouvor, Canada, September 1973, and has been published in AIChE Journal Vol. 20, 1974.

ABSTRACT

A thermal continuous parametric pump for separating multicomponent mixtures was experimentally investigated using the model system toluene-aniline-n-heptane on silica gel adsorbent. A simple method for predicting separations is presented and is found to be in good agreement with the experimental results. The method, based on an equilibrium theory, is under the assumption that a multicomponent mixture contains a series of pseudobinary systems, each system consisting of one of the solutes as one component and the common inert solvent as the other component.

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INTRODUCTION

Thermal parametric pumping is a phase-change separation process which depends for its operation on the coupling of periodic changes in temperature affecting the position of interphase equilibrium with synchronous periodic changes in flow direction. In the papers (Chen et al., (2,3)), separations of binary systems (single solute and its solvent) were experimentally investigated via continuous and semicontinuous parametric pumping. It has been shown that under certain conditions the pumps with feed at the enriched end have the capacity for complete removal of solute from one product stream and, at the same time, give arbitrarily large enrichment of solute in the other product stream.

In this thesis continuous parametric pumping is extended to the separations of multicomponent mixtures. The continuous pump is characterized by a steady flow of both feed and product streams during the hot upflow and cold downflow half-cycles. The system used is tolueneaniline-n-heptane on silica gel. A comparison is made between the experimental data and the calculated results based on the method proposed by Chen and Hill⁽¹⁾.

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THEORY

Figure 1 shows the continuous parametric pump model. Flow is upward during the hot half-cycle and downward during the cold half-cycle. Each half-cycle is $\frac{\pi}{\omega}$ time units in duration and the reservoir displacement volume is $Q(\frac{\pi}{\omega})$, where Q is the reservoir displacement rate. The pump has dead volumes V_T and V_Bfor the top and bottom reservoirs respectively. The feed is directed to the top of the column at the flow rate $(\phi_T + \phi_B)Q$. The top product flow rate is ϕ_TQ and the bottom product flow rate is ϕ_BQ , and ϕ_T and ϕ_B are the ratios of the top and bottom product rates to the reservoir displacement rate.

For processes inside the column we will assume, as did Pigford et al.⁽⁴⁾, that local interphase equilibrium exists with a linear distribution law having a temperature-dependent distribution coefficient. Also, there is negligible axial diffusion, temperature changes between hot and cold cycles are instantaneous, plug flow exists, and the fluid density is constant. We will assume further that the multicomponent mixture may be treated as n pairs of pseudo-binary systems. Each system includes one solute and the common inert solvent, and could be characterized by a dimensionless equilibrium parameter b₁ and corresponding values of the penetration distances of the hot and cold cycles, L₁₁ and L₂₁. L₁₁ and L₂₁

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can be expressed in terms of $\phi_{\rm B}$ and the equilibrium parameter b_i (Chen and Hill (1))

$$\frac{L_{1i}}{L_{2i}} = \frac{1 - \phi_B}{1 + \phi_B} \frac{1 + b_i}{1 - b_i}$$
(1)

$$L_{2i} = \frac{v_{o} (1 + \phi_{B})}{(1 + b_{i}) [1 + 0.5(m_{1i} + m_{2i})]} (\frac{\pi}{\omega})$$
(2)

where

$$^{m}i = \frac{\int s (1-\varepsilon) M_{i}}{\varepsilon}$$
(3)

and $M_i \left(= \frac{x_i}{y_i}\right)$ is the equilibrium distribution coefficient at temperature T. The quantity, b_i , is associated with a given two phase system when operated at two specific temperatures, and may be expressed as

$$b_{i} = \frac{0.5 \ (m_{2i} - m_{1i})}{1 - 0.5 \ (m_{1i} - m_{2i})}$$
(4)

The pump performance depends on the relative magnitudes of L_{1i}/L_{2i} and the height of the column, h. There are three possible regions of pump operations (see Figure 2) depending on L_{1i}/L_{2i} and h,

Region 1,
$$\frac{L_{1i}}{L_{2i}} \ge 1$$
 (or $\phi_B \le b_i$) and $L_{2i} \le h$ (5)

Region 2,
$$\frac{L_{1i}}{L_{2i}}$$
 <1 (or ϕ_B > b_i) and L_{1i} \leqslant h

Region 3, L_{1i} and $L_{2i} > h$

By treating the multicomponent mixture as a series of Pseudo-binary systems, the multicomponent separation could be predicted by the mathematical expressions for binary systems developed by Chen and Hill, ⁽¹⁾ and Chen et al. ^(2,3) It has been found that at steady state (n- $\rightarrow \infty$) solute removal from the bottom product stream, $\emptyset_B Q$, is complete in Region 1 and only partial in Regions 2 and 3.

Considering a mixture containing s solutes, each with its own b, and

$$b_1 > b_2 > \dots \quad b_k \gg \phi_B > b_k - 1 \dots > b_s$$
 (6)

were subscripts 1, 2, etc., refer to solutes 1, 2, etc. Furthermore,

 $L_{2i} \leqslant h$ when $i = 1, 2, \dots k$ (7)

 $L_{1i} \swarrow h$ when i = k - 1, --- sAt steady state the components, i = 1, 2, --- k for which the operations are indicated in Region 1, would appear only in the top product stream, and the remaining components (k + 1, --- s) would appear in both the top and bottom Product streams. In the extreme case where k = s the bottom product stream would consist only of pure solvent. By proper adjustment of \emptyset_B in Eq. 6 a solute split could be made which is analogous to that obtained by a multicomponent distillation column.

(a) Parametric Pumping Experiment

The experimental apparatus is shown schematically in Figure 3. Two dual infusion-withdrawal syringe pumps manufactured by the Harvard Apparatus Co., were used, one for the feed and one for the reservoirs. The jacked column had dimensions length 0.9 m, and inside diameter 0.01 m..Reservoirs at the two opposite ends of the column were two 50 cu. cm. glass syringes.

Prior to each run, a small magnetic stirrer was placed in each syringe to fulfill the required perfect mixing. Α micro-switch with stops was wired into the reservoir pump circuit to automatically reserve the action of the syringe plungers at the end of each half cycle. The jacked column was packed with 30 to 60 mesh chromatographic grade silica gel, and filled via the bottom reservoir syringe with the feed mixture of contrations, y_{o_1} , at room temperature. reservoir syringes were set to deliver about 40 cu. cm. The per half cycle with a minimum dead volume of approximately in 3 c.c. in each syringe. Hot and cold water baths were connected to the column jacket, and solenoid valves were wired to a dual timer to insure that hot water (333°K) was directed to the column during upflow and cold water (298°K) during downflow. Filled all lines and feed syringe with the feed mixture of contration, y_{0i} . After eleminating all of

air from the system, the bottom reservoir held 45 c.c. of the feed mixture and the top reservoir held 5 c.c.. The feed syringe was set to deliver 16 c.c. per half cycle.

To start the run, the feed and reservoir pumps were switched on and the timer was actived. The feed was delivered to the top of the column. The timer switched the solenoids to supply hot water $(333^{\circ}K)$ to the jacket. Bottom reservoir syringe pushed the fluid into the column, top reservoir received the fluid from the top of the column. Micrometer valves were used to regulate both the top and bottom product flows and impose a small back pressure on the system. The total cycle time was 2,400 sec. (1.200 sec. of up flow followed by 1,200 sec. of down flow). At the end of this half cycle, hot up flow half cycle, the microswitch reversed the reservoir pump movement. and the timer switched the solenoids to supply cold water (298°K) to the jacket. Subsequent to the cold down flow half cycle, the second cycle was started and the procedure was repeated until 14 cycles were completed.

Using the above procedures, twelve experimental runs were carried out. They were a aniline-n-heptane system, a toluene-n-heptane, and ten aniline-toluene-n-heptane systems in different operating conditions. All experimental data are given in Tables 2(A), 2(B), 3(A), 3(B) through 3(J). The experimental parameters for binary systems and ternary systems run 1 & run 2 are shown in Table 1.

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(b) Analysis of Product Streams

The product stream samples were taken at the end of each half cycle for analyzing.

To develop the procedure for analyzing ternary system (aniline-toluene-n-heptane) product, three previous experiments were made. First experiment, made a known concentration of aniline-n-heptane solution (aniline conc. = 2.743 x 10^{-1} k gmole/c.c.) diluted it with n-heptane to make different concentration solutions. Each diluted solution concentration was equal to 1/(Dilution Factor)of its original solution. Measuring each diluted solution by ultraviolet spectrometry, the absorbances , \mathcal{L} , at wave length , λ , of 263 m μ and 289 m μ were acquired. The data is shown on Table 4 and Plotted in Fig. 4 curves 1 and 4. For Second experiment, toluene-n-heptane solution (toluene conc.= 2.349 x 10^{-7} k gmole/c.c.) was made. Other procedures were the same as in experiment 1. The data is shown in Table 5, and Plotted in Fig. 4 curve 3. Made toluene-anilinen-heptane solution (aniline conc. = $2.743 \times 10^7 k$ gmole/c.c., toluene conc. = 2.349 x 10^7 k gmole/c.c.) for Third experiment. Same procedures as in First Experiment were carried out. The data is shown in Table 6, and Plotted in Fig. 4 curve 5. Samples for analysis were taken from the product streams at the end of each cycle and analyzed by ultraviolet spectrophotometry using the procedure illustrated in Fig. 4. То prevent the absorbance , \int , from over scale, each sample was

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diluted with n-heptane before analyzing to decrease its concentration to 1/(Dilution Factor) of that in original solution. For the runs involving binary systems (toluene or aniline in n-heptane) the analysis was straight forward and the concentration of solute was linearly proportional to the absorbance, l, at λ (wavelength) = 263 π or toluene and at $\lambda = 289 \pi^{\prime\prime}$ for aniline. This is clearly shown in curves 3 and 1 of Figure 4. In the case of the ternary system, aniline-toluene-n-heptane, the analysis was somewhat complicated. As shown in Fig.4, , I for aniline at $\lambda = 289 \, \text{mu}$ in toluene-n-heptane coincided with that in n-heptane alone and was found to be independent of the presence of toluene (curves 1 and 2). The l at $\lambda = 263 \, \text{m} M$ in toluene-anilinen-heptane (curve 5) was the sum of that in toluene-n-heptane (curve 3) and in aniline-n-heptane (curve 4). Therefore, for an unknown ternary mixture the aniline concentration was determined directly from l at $\lambda = 289 \text{ mu}$ regardless of the toluene concentration. Knowing the aniline concentration, ℓ for aniline at $\lambda = 263 \, m \, \text{M}$ was determined from curve 4. Subtraction of this from the total l obtained at l = 263 m din toluene-aniline-n-heptane gave the contribution of \mathcal{L} by toluene alone and hence its concentration from curve 3.

Using above procedureS, the product samples of twelve parametric pumping experiments, involving aniline-n-heptane system, toluene-n-heptane system and aniline-toluene-n-heptane in different operating conditions, were analyzed. Analysis results are shown in Tables 7(A), 7(B), and $\dot{8}(A)$ through 8(J).

RESULTS AND DISCUSSION

Among twelve experimental runs, the four typical runs — including two binary systems and two ternary systems, are employed in this section. The experimental parameters are shown in Table 1 and the data are plotted in Figures 5 and 6. The equations derived by Chen, et al.⁽²⁾ were used to calculate the concentration transients and the computed results corresponding to the experimental runs are also presented in Figures 5 and 6. These results compare resonably well with the observed values. In the computations for the ternary system (toluene-aniline-n-heptane), it was assumed that there are no interactions between solutes, and the system contains two pseudo-binaries, each binary consisting of one solute as one component and the common solvent as the other component, i.e., toluene-n-heptane and aniline-n-heptane.

When the pumps are operated in the Region 1, (i.e., $(L_{1i}/L_{2i}) \ge 1$ (or $\emptyset_B \le b_i$) and $L_{2i} \le h$), the bottom concentration transient is (Chen et al., 1972)

$$\frac{\langle y_{BP2} \rangle n}{y_{oi}} = \frac{1 - b_{i}}{1 + b_{i}} \left[\frac{1 - b_{i}}{1 + b_{i}} + C_{2} \right], \quad n \ge 1$$
(8)

and at steady state (n $\rightarrow \infty$),

$$\frac{\langle Y_{BP2} \rangle_{\infty}}{\gamma_{oi}} = 0$$
 (9)

Figure 5 illustrates $\langle y_{BP2} \rangle_n / \gamma_{oi}$ v.s. n for both toluene and aniline in both binary and ternary systems. one can see that $\langle y_{BP2} \rangle_n / y_{oi}$ decreases as n increases and, as the theory predicts, approaches zero as n becomes large. The slop, α , (of log $\langle y_{BP2} \rangle_n / y_{oi}$ v.s. n) of the solute i (toluene or aniline) in the binary mixture (toluene-n-heptane or aniline-n-heptane) is coincident with that in the ternary system (toluene-aniline-n-heptane). In other words, for a given C2, T1 and T2, the value of b; which can be calculated by Eq. 8 is essentially the same for both the binary from and ternary systems. It should be emphasized that the quantity b; is a measure of the extent of movement of solute i between phases as the result of a change in column temperature. Pump performance is enhanced by a large b_i. The particular b_i values considered here were determined to be 0.15 and 0.31 for toluene and aniline respectively.

Figure 6 shows the effects of L_{1i} , L_{2i} and h on the product concentrations. As long as L_{2i} is less than or equal to L_{1i} and h (Region 1), the separation factor, defined as the quotient of the top and bottom concentrations, approaches infinity as n becomes larger. If, in a pump originally operated in Region 1, L_{2i} is increased until it exceeds h or if L_{1i} becomes less than L_{2i} , switching points are encountered which cause the steady-state behavior of the pump to abruptly switch from a mode in which solute is completely removed from the bottom product stream to one in which solute removal is incomplete. One may visualize crossing the boundary L_{2i} =h as a result of increasing L_{2i} by increasing the reservoir displacement volume, $Q\frac{\pi}{\omega}$. Crossing of the boundary $L_{1i} = L_{2i}$ may be viewed as resulting from an increase of ϕ_B so that $\phi_B > b_i$, or $L_{2i} > L_{1i}$. At $T_{1} = 1$ 333 $\mathbf{\tilde{k}}$ and $T_2 = 298^{\circ} K$ the switching point for toluene corresponds to the condition $\phi_{B}=b_{toluene}=0.15$. In the case of aniline, the condition is $\phi_B = b_{aniline} =$ Thus, when $\not 0 \le 0.15$ (curves 3a and 3b), the 0.31. operation is in region 1 for both toluene and aniline, and solute removal from the bottom product stream may be complete at $n \rightarrow \infty$. If $\phi_{\rm B}$ is increased to the interval range, $0.15 < \phi \le 0.31$ (curves 4a and 4b), the operation switches to region 2 for toluene and, remains in region 1 for aniline, and the bottom product could eventually contain only toluene. If $\phi_{\rm R}$ is further increased, ϕ_{B} >0.31 = b_{aniline}, the operation is now in region 2 for both toluene and aniline, and both aniline and toluene would appear in the bottom product stream at n $\rightarrow \infty$. Over the internal $\emptyset_B \leq b_1 = \text{switch point of solute i,}$ then $\langle y_T p_2 \rangle_{\infty} / y_{0i} = 1 + \frac{\beta_B}{\rho_m}$

Beyond the switching points (see curve 4a, Figure 6),

 $< y_{BP2} \gg / y_{oi}$ and $< y_{TP2} \gg / y_{oi}$ can be calculated by the equations (Chen and Hill (1))

$$\frac{\langle y_{\rm TP2} \rangle_{\infty}}{y_{\rm oi}} = \frac{(\phi_{\rm T} + \phi_{\rm B}) (1 - b \phi_{\rm B})}{\phi_{\rm T} + \phi_{\rm B} - b (1 + \phi_{\rm T} \phi_{\rm B})}$$

$$\frac{\langle y_{BP2} \rangle}{y_{oi}} = \frac{(\phi_{B} - b) (\phi_{T} + \phi_{B})}{\phi_{B} [(\phi_{T} - \phi_{B}) - b (1 - \phi_{T} \phi_{B})]}$$

The method presented here is a means of predicting multicomponent separations by assuming that solutes do not interact with one another. In practice, the concentrations of solutes may be quite high and the high solute concentrations may cause competition for the adsorption sites. Therefore the maximum separation factors may never be obtained.

CONCLUSIONS

A simple means of predicting multicomponent separations in the continuous thermal parametric pump is presented. A multicomponent system is treated as a series of pseudo-binary systems, each binary system consisting of one of the solutes as one component and the common inert solvent as the other component. This approach permits the use of transient and steady state equations for binary systems developed by Chen and Hill⁽¹⁾ and Chen et. al.,⁽²⁾ Experimental data for the concentration transients agree reasonably well with the analytical predictions.

It is shown that the thermal parametric pump is capable of separating components in a multicomponent mixture and, as a theoretical limit, of attaining infinite separation factors. Also, the net movement of concentration fronts through the adsorption column is found to be important in determining the pump performance. Those solutes for which the net movement is upward would, at steady state, appear only in the top product. The remaining solutes would appear in both the top and bottom products. In the limiting case, it is possible for the bottom product to consist solely of pure solvent. This happens when all solutes in a mixture are very strongly adsorbed in a given cycle or when the flow rate of the bottom product is very small.

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NOTATION

	b		dimensionless equilibrium parameter defined
			by Equation (4)
	cl	=	V _T , dimensionless
			$Q \frac{\pi}{\omega}$
	C.		V _B dimensionless
	-2	-	$Q \frac{\pi}{\omega}$
	h ·	-	column height, m
	L	=	penetration distances defined by Equation (1)
			or (2), m
	М	=	$\frac{x}{y}$, k gmoles/g.
	m	=	dimensionless equilibrium parameter defined
			by Eq. 3
	n	=	number of cycle of pump operation
	Q		reservoir displacement rate, cm ³ /s
	vo	=	interstital velocity based on the reservoir
			displacement rate, m/s
	۷ _T	Ŧ	top reservoir dead volume, cm ³
	v _B	_	bottom reservoir dead volume, cm ³
	x	=	concentration of solute in the solid phase,
			k gmole/g of adsorbent
	У	=	concentration of solute in the liquid phase,
		5	k gmoles/cm ³ or k gmoles/k gmoles of fluids.
<	<_>	=	average value

.

	Greek	Lette	ers
	ø	=	product volumetric flow rate/reservoir
•	$\frac{\pi}{\omega}$	=	displacement rate, dimensionless
			duration of half cycle,s
	3	=	void fraction in packing, dimensionless
	۴f	=	density of fluids k gmoles/cm ³
	fs	_	density of adsorbent, kg./cm ³
	Subsci	ripts	
	0	=	initial condition
	1	-	upflow
	2	=	downflow
	В		stream from or to bottom of the column
	i	-	solute i
	P	-	product stream
.'	Т	=	stream from or to top of the column
	BP	=	bottom product
	TP	=	top product

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Figure 1



Figure 2



Fig. 3



Feed pump











.



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$\frac{\pi}{\omega}$	=12(00 Sec.,	Tl = 33	3 [°] K,	T ₂ =	298 ⁰ K,	h =	0.9m,	$\phi_{\rm T} + \phi_{\rm f}$	_B =0.4
		Conc.	Vol.%	Ø _B	C	с ₂	^b i	$L_{li}(m)$	L _(m)	Region
A.		2.5	0	0.07	0.12	0.13	0.15	0.51	0.43	1
Β.		0	2.5	0.10	0.11	0.13	0.31	0.70	0.45	1
C.		2.5	2.5	0.09	0.12	0.13				
	C-1						0.15	0.49	0.44	l
	C-2						0.31	0.71	0.45	1
D.		2.5	2.5	0.22	0.12	0.13				
	D-1						0.15	0.42	0.49	2
	D-2						0.31	0.62	0.51	1

A. Toluene-n-heptane system

B. Aniline-n-heptane system

C. *Aniline-toluene-n-heptane system experiment, Run 1
 C-1 Toluene-n-heptane system

C-2 Aniline-n-heptane system

- D. *Aniline-toluene-n-heptane system experiment, Run 2 D-1 Toluene-n-heptane system
 D. Aniline - heptane system
 - D-2 Aniline-n-heptane system
- * Assume that a ternary system contains two pseudo-binaries

TABLE 1 Experimental and Model Parameter

TABLE 2(A)

4

. -

T _l = 333°K	Т <mark>2</mark> = 298 [°] К
$\frac{\pi}{\omega}$ = 1200 Sec.	$\phi_{\mathtt{T}}$ + $\phi_{\mathtt{B}}$ = 0.40
$Q\frac{\pi}{\omega}$ = 40 c.c.	\$ B= 0.1

		Feed	i	Rese rv Readin	sir g	Reservoi Displac	ir ement	Product	
C	ycle	Syringe	Volume	Bottom	Top	Bottom	Top	Bottom	Top
-	n n	(c.c.)	(c.c.)	<u>(c.c.)</u>	(c.c.)	(c.c.)	(c.c.)	(c.c.)	<u>(c.c.</u>)
		42		46.0	5.0				
1	Up	26	16.0	5.0	45.0	41.0	40.0	4.0	11.0
1	Down	10	16.0	46.0	4.0	41.0	40.0	3.7	8.0
		43	Feed s	yringe	was ref	Cilled w	ith fee	ed	
2	Up	27	16.0	4.0	46.0	42.0	42.0	4.0	11.6
2	Down	11	16.0	46.0	4.0	42.0	42.0	3.6	9.0
		45	Feed s	yringe	was rei	Cilled w	ith fee	ed	
3	Up	28	17.0	5.0	48.0	41.0	44.0	3.8 .	11.8
3	Down	12	16.0	46.0	4.0	41.0	44.0	3.9	10.2
		47	Feed a	yringe	was rei	filled w	ith fee	ed	
4	Up	3 3	14.0	4.0	46.0	42.0	42.0	4.0	11.0
4	Down	17	16.0	42.0	7.0	38.0	39.0	3.6	11.6
5	Up	3	14.0	4.0	43.0	38.0	36.0	3.8	10.7
		47	Feed a	syringe	was re:	filled w	ith fe	ed	
5	Down	31	16.0	42.0	4.0	38.0	39.0	3.8	9.3

•

Pood		Reservoir		Reservoir Displacement		Product		
Cycle	Syringe	Volume	Bottom	Top	Bottom	Top	Bottom	Top
No. <u>n</u>	Reading (c.c.)	Fed (c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)
6 Up	17	16.0	5.0	41.0	37.0	37.0	3.8	11.0
6 Down	1	16.0	42.0	4.0	37.0	37.0	3.8	10.9
	47	Feed	syringe	was re	filled	with fe	eed	
7 Up	32.5	15.5	4.0	41.0	38.0	37.0	3.9	12.0
7 Down	17	15.5	41.0	3.5	37.0	37.5	3.7	10.2
8 Up	3	14.0	5.0	41.0	36.0	37.5	3.9	11.0
	46	Feed	syringe	was re	efilled	with fo	eed	
8 Down	29	17.0	42.0	5.0	37.0	36.0	3.6	12.2
9 Up	13	16.0	5.0	42.0	37.0	37.0	4.0	12.8
	47	Feed	syringe	was re	efilled	with f	eed	
9 Down	32	15.0	44.0	3.0	39,0	39.0	3.4	11.2
10 Up	17	15.0	5.0	42.0	39.0	39.0	3.7	13.0
10 Dow	n 2	15.0	42.0	3.0	37.0	39.0	3.7	11.0
wa ##	47	Feed	syringe	was r	efilled	with f	eed	
ll Up	31	16.0	4.0	42.0	38.0	39.0	4.0	12.2
ll Dow	n 14	17.0	45.0	3.0	41.0	39.0	3.8	11.8
	47	Feed	syringe	was r	efilled	with f	eed	
12 Up	31	16.0	6.0	43.0	39.0	40.0	4.0	12.8
12 Dow	n 14	17.0	46.0	2.0	40.0	41.0	3.8	13.0
	45	Feed	syringe	was r	efilled	with f	eed	
13 Up	30	15.0	4.0	47.0	42.0	45.0	3.7	11.8
13 Dow	n 13	17.0	47.0	5.0	43.0	42.0	3.5	12.6
							,	

	Fee	d	Reservo Reading	ir	Reservoi <u>Displace</u>	r ement	Product	
Cycle	Syringe Reading	Volume Fed	Bottom	Тор	Bottom	Тор	Bottom	Top
<u>n</u>	(c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)	(c.c.)
	46	Feed a	syringe w	as ret	illed wi	th fee	d	
14 Up	30	16	6.0	47.0	41.0	42.0	3.8	11.6
14 Dov	vn 4	16	47.0	5.0	41.0	42.0	3.8	11.0

TABLE 2(B)

System: Tolnene - n-Heptane

Feed: Toluene Conc.= 2.349 x 10^{-7} k gmole/c.c.= 2.5 vol.% Operating Condition: $T_1 = 333^{\circ}$ K $\phi_B + \phi_T = 0.4$

 $T_1 = 555 \text{ K}$ $\varphi_B + \varphi_I = 0.4$
 $T_2 = 298^{\circ} \text{ K}$ $\varphi_B = 0.05 \text{ (Experimental)}$
 $\frac{\pi}{\omega} = 1200 \text{ Sec.}$ $Q\frac{\pi}{\omega} = 40 \text{ c.c.}$

Cycle	Fee Syringe	d Volume	Reservo <u>Reading</u> Bottom	ir Top	Reserve Displac Bottom	oir cement Top	Product Bottom	t Top
No. n	Reading (c.c.)	Fed (c.c.)	<u>(c.c.</u>)	(c.c.)	<u>(c.c.)</u>	(c.c.)	<u>(c.c.)</u>	(c.c.)
	47		46.0	6.0				
l Up	31	16	5.0	43.0	41.0	37.0	2.0	12.0
1 Down	15	16	42.0	5.0	37.0	38.0	1.9	12.0
	48	Feed	syringe	was re	filled	with fe	ed	
2 Up	34	14	4.0	43.0	36.0	38.0	2.0	14.0
2 Down	18	16	42.0	5.0	38.0	38.0	1.8	7.0
3 Up	2	16	5.0	40.0	37.0	35.0	2.2	15.6
	49	Feed	syringe	was re	filled	with fe	ed	
3 Down	33	16	45.0	5.0	40.0	35.0	1.9	12.0
4 Up	17	16	5.0	43.0	40 .0	38.0	2.4	15.0
	48	Feed	syringe	was re	filled	with fe	e d	
4 Down	32	16	46.0	5.0	41.0	38.0	1.9	12.4
5 Up	16	16	5.0	46.0	41.0	41.0	2.0	16.0
5 Down	1	15	45.0	5.0	40.0	41.0	2.0	12.0
	48	Feed	syringe	was re	filled	with fe	ed	
6 Up	32	16	5.0	46.0	40.0	41.0	2.0	16.0

	Fee	d	Reservo Reading	5	Reservo Displac	oir ement	Product	<u>t</u>
Cycle	Syringe	Volume	Bottom	Top	Bottom	Тор	Bottom	Top
<u>n</u>	(c.c.)	(c.c.)	(c.c.)	<u>(c.c.)</u>	(c.c.)	(c.c.)	(c.c.)	<u>(c.c.)</u>
6 Down	16	16	45.0	5.0	40.0	41.0	1.8	12.6
7 Up	0	16	5.0	46.0	40.0	41.0	2.0	17.0
	49	Feed a	syringe w	vas ref	illed wi	ith feed	L	
7 Down	32	17	46.0	6.0	41.0	4 0. 0	1.9	12.6
8 Up	16	16	5.0	48.0	41.0	42.0	2.0	15.0
8 Down	0	16	45.0	5.0	40.0	43.0	2.0	13.4
	49	Feed	syringe	was ref	illed w	ith feed	1	
9 Up	33	16	5.0	47.0	40.0	42.0	2.0	12.8
9 Down	17	16	45.0	5.0	40.0	42.0	2.0	14.2
10 Up	1	16	5.0	46.0	40.0	41.0	2.0	15.0
	49	Feed	syringe	was ref	'i lle d w	ith feed	d	
10 Down	a 33	16	45.0	5.0	40.0	41.0	2.0	12.4
ll Up	17	16	5.0	45.0	40.0	40.0	2.0	15.2
ll Down	1 1	16	45.0	5.0	40.0	40.0	2.0	13.2
	48	Feed	syringe	was ref	illed w	ith fee	d	
12 Up	32	16	5.0	45.0	40.0	40.0	2.0	14.7
12 Dowr	n 16	16	45.0	5.0	40.0	40.0	2.0	14.5
13 Up	0	16	5.0	45.0	40.0	40.0	2.2	15.0
	48	Feed	syringe	was ref	filled w	ith fee	d	
13 Dowr	n 32	16	45.0	5.0	40.0	40.0	2.0	12.0
14 Up	16	16	5.0	45.0	40,0	40.0	2.0	16.0
14 Down	n O	16	45.0	5.0	40.0	40.0	2.0	12.8

TABLE 3(A) Parametric Pumping Experiment Run 1

System : Aniline - Toluene - n-Heptane Feed : Aniline Conc. = $2.743 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Toluene Conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Operating Condition : $T_1 = 333 \text{ K}$ $T_2 = 298 \text{ K}$ $\phi_B + \phi_T = 0.4$ $\phi_B = 0.05 \text{ (Experimental)}$ $\frac{\pi}{\omega} = 1200 \text{ Sec.}$ $Q_{\overline{w}} = 40 \text{ c.c.}$

	. Fee	d	Reservoir Reading		Reservo Displac	ir ement	: Produc	Product	
Cycle	Syringe	Volume	Botm.	Top	Botm.	Тор	Botm.	Top	
<u>n</u>	(c.c.)	(c.c.)	(c.c.)	(c.c	<u>.)(c.c.)</u>	(<u>c.c.</u>)	(c.c.)	<u>(c.c.)</u>	
	32		47	·7					
l Up	16	16	7	47	40	40	2.0	13.0	
2 Dn.	0	16	47	7	40	40	2.3	11.0	
	48	Feed	syringe	was	r efil led	with	feed		
2 Up	32	16	7	47	40	40	2.3	17.0	
2 Dn.	16	16	47	7	40	40	2.0	17.0	
3 Up	0	16	7	47	40	40	2.3	16.0	
	48	Feed	syringe	was	refilled	with	feed	·	
3 Dn.	32	16	47	7	40	40	2.0	12.5	
4 Up	16	16	7	47	40	40	2.3	16.0	
4 Dn.	0	16	47	7	40	40	2.1	11.0	
	50	Feed	syringe	was	refilled	with	feed		
5 Up	34	16	7	47	40	40	2.4	15.0	
5 Dn.	18	16	47	. 7	40	40	2.2	12.0	
6 Up	2	16	7	47	40	40	2.3	17.0	

	·	Fee	d	Reserv Readin	oir g	Reservo Displac	ir ement	Produc	ct ·
i.	Cycle	Syringe Reading	Volume	Both.	Top	Botm.	Top	Botm.	Тор
	<u>n</u>	(c.c.)	<u>(c.c.)</u>	(c.c.)	<u>(c.c.</u>	<u>)(c.c.</u>)(<u>c.c.)</u>	<u>(c.c.)</u>	(c.c.)
		50	Feed	s yringe	was	refilled	with	teed	
	6 Dn.	37	13	47	7	40	40	2.0	10.0
	7 Up	21	16	7	47	40	40	2.3	16.0
	7 Dn.	5	16	47	7	40	40	2.2	13.0
		51	Feed	syringe	was	refilled	with	feed	
	8 Up	36	15	7	47	40	40	2.2	15.2
	8 Dn.	20	16	47	7	40	40	2,2	12.0
	9 Up	. 4	16	7	47	40	40	2.2	16.0
		50	Feed	syringe	was	refilled	. with	feed	
	9 Dn.	34	16	47	7	40	40	2.3	12.0
	10 Up	18	16	7	47	40	40	2.2	15.5
	10 Dn.	2	16	47	7	40	40	2.5	13.0
		50	Feed	syringe	was	refilled	with	feed	
	ll Up	34	16	7	47	40	40	2,2	15.0
	ll Dn.	18	16	47	7	40	40	2.5	11.0
	12 Up	2	16	7	47	40	40	2.3	15.0
		50	Feed	syringe	was	refilled	l with	feed	
	12 Dn.	33	17	47	7	40	40	2.8	15.0
	13 Up	17	16	7	47	40	40	2.0	15.0
·	13 Dn.	l	16 -	47	7	40	40	2.3	13.0
	يە بەر	50	Feed	syringe	e was	refilled	l with	feed	
	14 Up	34	16	7	47	40	40	2.2	15.0
	14 Dn.	18	16	47	7	40	40	2.3	16.0
	•								

Feed			Reserv oir Reading		Reserv oir Displacement		Product		
Cycle No.	Syringe Reading	Volume ; Fed	Botm.	Тор	Botm.	Тор	Botm.	Top	
<u>n</u>	<u>(c.c.</u>)	<u>(c.c.)</u>	(c.c.)	<u>(c.c.</u>) <u>(c.c.</u>) <u>(c.c.)</u>	(c.c.)	(c.c.)	
15 Up	28	16	7	47	40	40	2.2	15.0	
15 Dn.	12	16	47	7	40	40	2.0	13.0	

TABLE 3(B) Farametric Fumping Experiment Run 2

System : Aniline - Toluene - n Heptane

Feed : Aniline 18c.c.+ Toluene 18c.c.+ n-Heptane 684c.c.Aniline Conc. = 2.743×10^{-7} k gmole/c.c. = 2.5 Vol.% Toluene Conc. = 2.349×10^{-7} k gmole/c.c. = 2.5 Vol.% Operating Condition :

$T_1 = 333^{\circ} K$	$\phi_{\rm B} + \phi_{\rm T} = 0.4$
$T_2 = 298^{\circ} K$	$\phi_{\rm B}$ = 0.218 (Experimental)
$\frac{\pi}{\omega}$ = 1200 Sec.	Q

Cycle	Fe	ed Volume	Reserv <u>Readir</u> Botm.	oir _{ng} Top	Reservo Displac Botm.	ir <u>ement</u> Top	Produc Botm.	et Top
$\frac{n}{n}$	$\frac{(c.c.)}{(c.c.)}$	<u>(c.c.)</u>	(c.c.)	<u>(c.</u> c	<u>.)(c.c.)</u>	<u>(c.c.</u>) <u>(c.c.)</u>	(c.c.)
	50		48	7				
l Up	35	15	8	47	40	40	8.7	7.6
l Dn.	20	15	48	7	40	40	8.7	5.3
2 Up	4	16	7	47	41	40	8.7	11.0
	49	Feed	syringe	was	refilled	with	feed	
2 Dn.	32	17	47	7	40	40	8.6	5.6
3 Up	16	16	7	47	40	40	8.6	9.4
	50	Feed	syringe	was	refilled	with	feed	
3 Dn.	34	16	47	7	40	40	8.6	6.0
4 Up	17	17	7	47	40	40	8.6	10.6
4 Dn.	1	16	47	7	40	40	8.6	5.6
	50	Feed	syringe	was	refilled	with	feed	
5 Up	34	16	7	47	40	40	8.6	8.6

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Cucle	Fee	ed Volume	Reserv Readir Botm	noir ng Top	Reservo Displac	ir ement Top	Produce Botm.	t Top
No. n	Reading (c.c.)	Fed $(c.c.)$	<u>(c.c.)</u>	(c.c.) <u>(c.c.)</u> (<u>c.c.</u>)	(c.c.)	(c.c.)
5 Dn.	18	16	47	- 7	40	40	8.7	6.6
6 Up	2	16	7	47	40	40	8.6	8.8
	51	Feed	syringe	was r	efilled	with :	feed	
6 Dn.	35	16	47	7	40	40	8.7	5.8
7 Up	19	16	7	47	40	40	8.6	9.2
7 Dn.	3	16	47	7	40	40	. 8.7	7.4
· ,	53	Feed	syringe	was r	efilled	with :	feed	
8 Up	37	16	7	47	40	40	8.7	7.8
8 Dn.	21	16	47	7	40	40	8.7	5.8
9 Up	5	16	7	47	40	40	8.7	8,8
<u></u>	50	Feed	syringe	was r	refilled	with	feed	
9 Dn.	34	16	47	7	40	40	8.8	5.9
10 Up	18	16	7	47	40	40	8.7	8.2
10 Dn.	2	16	47	7	40	40	8.7	6.6
	51 ,	Feed	syringe	was 1	refilled	with	feed	
ll Up	35	16	7	47	40	40	8.8	8.4
ll Dn.	19	16	47	7	40	40	8.7	6.2
12 Up	3	16	7	47	40	40	8.7	8.6
	49	Feed	syringe	was	refilled	with	feed	
12 Dn.	33	16	47	7	40	40	8.7	6.2
13 Up	17	16	7	47	40	40	8.7	9.0
13 Dn.	1	16	47	7	40	40	8.7	5.8
			-					

Cycle No.	Fe Syringe Reading	ed Volume Fed	Reser <u>Readi</u> Botm.	voir ng Top	Reserve <u>Displac</u> Botm.	oir <u>cement</u> Top	Produc Botm.	t Top
<u>n</u>	(c.c.)	(<u>c.c.)</u>	(c.c.)	(c.c.) <u>(c.c.)</u>	(<u>c.c.)</u>	<u>(c.c.)</u> (<u>c.c.</u>)
	40	reed sy	yringe	was Re	filled	with f	eed	
14 Up	24	16	7	47	40	40	8.7	9.4
14 Dn.	. 8	16	47	7	40	40	8.7	6.0

TABLE 4. Absorbance of Aniline Solution

Solution : Aniline 2.5c.c. + n-Heptane 97.5c.c. Conc. of Aniline= 2.743 x 10⁷k gmole/c.c.

No.	(Dilution) Factor	Toluene Conc.x10 <u>k gmole/c.c.</u>	<u>Yi</u> Y _R	Absorbance, L at 263 mµ	Absorbance, L at 289 mm
l.	242	11.33	1.031	67.2	
2.	363	7.56	0.689	44.0	
3.	484	5.67	0.516	33.6	
4.	726	3.78	0.344	22.8	72.0
5.	1029	2.67	0.243	16.0	
6.	1331	2.06	0.188	13.5	40.5
7.	1815	1.51	0.138	11.0	28.5
8.	2178	1.26	0.115	7.5	
9.	2904	0.95	0.086	5.5	
		· · · ·			

Where $y_{R} = 10.977 \times 10^{-10}$

TABLE 5. Absorbance of Toluene Solution

Solution : Toluene 2.5c.c. + n-Heptane 97.5c.c. Conc. of Toluene = 2.349 x 10^7 k gmole/c.c.

No.	(Dilution) Factor	Toluene conc.x10 k gmole/c.c.	<u> </u>	Absorbance, at 263 mm	Absorbance, L at 289 mm
l.	242	9.71	1.031	23.6	0
2.	363	6.47	0.688	15.5	0
3.	484	4.85	0.516	11.0	0
4.	726	3.24	0.344	7.2	0
÷ 5.	1331	1.76	0.188	4.0	0

Where $y_{R} = 9.413 \times 10^{-10}$

TABLE 6. Absorbance of Aniline and Toluene Solution

.

Solution : Aniline 2.5 c.c. + Toluene + n-Heptane 95.0 c.c.

Conc. of Aniline = 2.743×10^7 k gmole/c.c. Conc. of Toluene = $2.349 \times 10^7 k$ gmole/c.c.

No.	(Dilution) Factor	Aniline Conc.xl0 ¹⁰ <u>k_gmole/c.c</u> .	Toluene Conc.x10 <u>k gmole/c.c</u> .	$\frac{y_i}{y_R} \xrightarrow{\text{Asorb.}, j}_{\text{at 289}m}$	Asorb., L u at 263 mu
1.	242	11.33	9.71	1.031	89.0
2.	484	5.67	4.85	0.516	43.8
3.	726	3.78	3.24	0.344 71.0	30.0
4.	1029	2.67	2.29	0.243 51.0	21.0
5.	1331	2.06	1.76	0.188 39.4	-
6.	1815	1.51	1.29	0.138	12.0
7.	2178	1.26	1.08	0.115 23.5	
8.	2904	0.95	0.81	0.086 17.5	7.4

Where

 $y_{R} = 9.413 \times 10^{-10}$ for Toluene conc. $y_{R} = 10.977 \times 10^{-10}$

for Aniline conc.

For the Parametric Pumping Experiment :

System : Aniline - n-Heptane

Feed : Aniline Conc. = $2.743 \times 10^7 \text{k}$ gmole/c.c. = 2.5 Vol.% $\phi_B = 0.1$

Sa ((ample No. Cycle No.) n	(Dilution)	Absorbance, at289 <i>m</i> µ	Sample Conc. k gmole/c.c. x 10	<y<sub>BPz>n Yoi</y<sub>
Fe	ed	726	70.6	2.743	1.00
1	Bot.,Down	242	84.2	1.089	0.0397
2	Bot.,Down	121	42.8	0.277	0.101
4	Bot.,Down	63	50.4	0.170	0.0619
6	Bot.,Down	16	52.5	0.045	0.0164
, 8	Bot.,Down	10	52.8	0.0283	0.0103
10	Bot.,Down	11	31.2	0.0184	0.0067
12	Bot.,Down	6	43.6	0.0140	0.0051

	11.11.11.11.11.11.11.11.11.11.11.11.11.				<u><y<sub>TB2>n Yoi</y<sub></u>
4	Top, Down	1331	38.9	2.773	1.01
5	Top, Down	1331	41.9	2.990	1.09
6	Top,Down	1331	44.3	3.155	1.15
7	Top, Down	1331	44.3	3.155	1.15
8	Top,Down	1331	39.3	2.806	1.02
10	Top,Down	1331	42.4	3.023	1.10
		-			

TABLE 7(B).Analyses of Product Stream Samples

For the Parametric Pumping Experiment:

System: Toluene -n-Heptane

Feed: Toluene.conc.= 2.349 x 10^{-7} k gmole/c.c. = 2.5 Vol.%

 $\phi_B = 0.05$

Sample No. (I (Cycle No.) n	Dilution Factor	Absorb. at 263 mµ	Sample Conc.x 10 ⁷ k gmole/c.c.	<y<sub>Bpz>n Yoi</y<sub>
Feed	6 6	84.0	2.349	1.00
3 Botm. Down	48	62.1	1.264	0.54
4 Botm. Down	40	55.5	0.939	0.40
6 Botm. Down	24	55.4	0.564	0.24
8 Botm. Down	11	57.0	0.312	0.133
9 Botm. Down	8	62.5	0,212	0.09
10 Botm. Down	6	54,5	0.139	0.059
ll Botm. Down	6	36,6	0.093	0.0397
12 Botm. Down	4	41.2	0.070	0.0297
13 Botm. Down	3	38.9	0.049	0.021
14 Botm. Down	2	39,5	0.033	0.014
				<u>< Y_Tpz>n Yoi</u>
4 Top, Down	121	50,0	2.56	1.09
6 Top, Down	121	53,5	2.75	1.17
8 Top, Down	121	58,2	2.98	1.27
10 Top, Down	121	60.8	3.12	1.33
12 Top, Down	121	57.2	2.94	1.25
14 Top, Down	121	59.0	3.03	1.29

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TABLE 8(A). Analyses of Product Stream Samples

For the Parametric Pumping Experiment : Run 1

System: Aniline - Toluene - n-Heptane

Feed : Aniline Conc. = $2.743 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Toluene Conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$

 $\phi_B = 0.05$ (Experimental Value)

Sa ((ample No. Vole No.)	(Dilution)	Absorbance at 289 mll	Absorbance at 263 mH	<u> </u>	pz>n oì
	n				Aniline	Toluene
1	Bot.,Down	363	49.1	27.3		0.845
2	Bot.,Down	363	49.1		0.348	
3	Bot.,Down	242	31.3	22.8	0.148	0.606
4	Bot.,Down	88	58.7	51.5	0.101	0.560
5	Bot.,Down	40	63.5	71.0	0.0496	0.390
7	Bot.,Down	36	16.4	31.5	0.0115	0.180
8	Bot.,Down	20	24.8	42.0	0.0097	0.130
9	Bot.,Down	11	28.8	53.2	0.0062	0.092
10	Bot.,Down	7	32.9	60.0	0.0045	0.066
11	Bot.,Down	6	22.2	45.5	0.0026	0.044
12	Bot.,Down	6	13.6	28.0	0.0016.	0.027
13	Bot.,Down	4	19.2	23.8	0.0015	0.021
14	Bot.,Down	4	12.0	23.5	0.00094	0.015
15	Bot.,Down	3	8.5	27.5	0.00049	0.014
Fe	eed	726	70.5	28.8	1.00	1.00

Sample No. (Cycle No.) n	(Dilution)-	Absorbance, at 289 mM	Absorbance, at 263 m M	<u> </u>	ρ₂> i Toluene
Feed	1331	38.5	15.8	1.00	1.00
9 Top, Down	1331	38.5	18.8	1.00	1.75
ll Top,Down	1331	38.5	17.7	1.00	1.50
12 Top,Down	1331	38.5	17.8	1.00	1.52
15 Top, Down	1331	40.4	17.2	1.051	1.22

TABLE 8(B). Analyses of Product Stream Samples

For the Parametric Pumping Experiment : Run 2

System : Aniline - Toluene - n-Heptane

Feed : Aniline Conc. = $2.743 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Toluene Conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ $\phi_{\text{B}} = 0.218$

Sample No. (Cycle No.) n	(Dilution) Factor	Absorbance at 289 mm	_	<u>(YBPZ>n</u> Yoi Aniline
Feed	605	84.8		1.00
2 Botm.,Down	121	53.0		0.125
3 Botm., Down	121	27.5		0.065
5 Botm., Down	44	3 7.5		0.032
6 Botm.,Down	20	60.5		0.0236
7 Botm.,Down	10	82.0	·	0.016
8 Botm., Down	10	32.8		0.0064
9 Botm.,Down	6	55.5		0.0065
10 Botm.,Down	6	40.2		0.0047
ll Botm.,Down	8	63.5		0.0033
12 Botm.,Down	5	63.4		0.00206
13 Botm.,Down	5 5	53.5		0.00174
14 Botm.,Down	1	64.5		0.00126

(continued on next page)

TABLE 8(B). (Continued)

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S: (0	ample No. Cycle No.) n	(Dilution) Factor	Absorbance at 289 mm	Absorbance at 263 m M	 ✓yep₂>n yoi Toluene
Fe	eed	616	84.0	34.6	1.00
2	Botm.,Down	88	74.0	67.0	0.71
3	Botm., Down	66	51.0	60.5	0,54
4	Botm.,Down	44	44.0	67.0	0.43
5	Botm.,Down	44	38.5	51.5	0.32
6	Botm.,Down	44	28.0	45.0	0.29
7	Botm.,Down	33	.25.2	60.0	0.31
8	Botm.,Down	22	15.0	71.5	0.267
10	Botm., Down	22	10.5	70.5	0.27
11	Botm.,Down	3 3	5.0	66.5	0.39
12	Botm.,Down	33	3.2	77.5	0.46
13	Botm.,Down	33	1.0	80.0	0.48
14	Botm.,Down	44	0,5	80.0	0.64

Top product stream samples were analyzed by gas chromatograph

			0
Cycle No. n	<u> </u>	۹,	
	Aniline	<u>Toluene</u>	
14	1.860	1.806	•
13	1.866	1.741	
12	1.833	1.806	
11	1.333	1.774	
10	1.400	1.483	

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APPENDIX

Among 10 ternary system experimental runs, 8 runs (run 3 through run 10) are attached in the appendix here. Their experimental data, product streams analysis, and plotted figures are following:

Run No.	Experimental	** Data	Product	Analysis ^{**}	Plotted	** Figures
3	Table 3(C)		Table	8(C)	Fig.	10
4	3(D)			8(D)		11
5	3(E)			8(E)		12
6	3(F)			8(F)		13
7	3(G)			8(G)		14
8	3(H)			8(H)		15
9	3(I)			8(I)		16
10	3(J)			8(J)		17

**Note: Refer to Pages 16 and 17 for notations and Greek letters shown on Tables and Figures.

TABLE 3(C). Parametric Pumping Experiment, Run 3

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = $2.743 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Operating condition :

$T_1 = 333$ [°] K	$\phi_{\rm T} + \phi_{\rm B} = 0.4$
$T_2 = 298^{\circ} K$	$\phi_{\rm B} = 0.088$
$\frac{\pi}{\omega}$ = 1200 Sec.	$Q\frac{\pi}{\omega} = 40$ c.c.

Feed			Reserv	Öir ø	Reservo Displac	ir ement	Product		
Cycle	Syringe	Volume	Botm.	Top	Botm.	Top	Botm.	Тор	
<u>n</u>	(c.c.)	(c.c.)	(c.c.)	<u>(c.c.</u>) <u>(c.c.)</u> (<u>C.c.)</u>	(<u>c.c.)</u>	<u>(c.c.)</u>	
100 100	43		47	7					
l Up	27	16	. 7	47	40	40	3.5	13.0	
l Dn.	11	16	47	7	40	40	3.5	9.0	
-	48	Feed	syringe	was	refilled	with	feed		
2 Up	32	16	7	47	40	40	3.5	14.0	
2 Dn.	16	16	47	7	40	40	3.5	10.4	
3 Up	0	16	7	47	40	40	3.5	14.5	
	50	Feed	syringe	was	refilled	with	feed	· · · · ·	
3 Dn.	34	16	47	7	40	40	3.5	10.0	
4 Up	18	16	9	47	38	40	3.5	13/5	
4 Dn.	2	16	48	- 7	39	40	3.4	12.0	
	50	Feed	syringe	was	refilled	with	feed		
5 Up	33	17	7	4 7	41	40	3.5	17.0	
5 Dn.	17	16	47	7	40	40	3.5	10.6	

Feed				Reservoir <u>Reading</u>		Reservoir <u>Displacement</u> Retmon		Product	
	Cycle No.	Syringe Reading	volume Fed	Botm.	Тор	Botm. T	op	Botm.	Тор
	<u>n</u>	<u>(c.c.)</u>	<u>(c.c.)</u>	<u>(c.c.)(c</u>	<u>.c.</u>)	<u>(c.c.)(c.</u>	<u>c.) (</u>	<u>c.c.)</u>	(c.c.)
	6 Up	1	16	7	47	40	40	3.5	16.2
		51	Feed	syringe	was	refilled	with	feed	
	6 Dn.	36	15	46	7	39	40	3.5	9.0
	7 Up	22	14	7	47	41	40	3.5	13.5
	7 Dn.	6	16	47	7	40	40	3.5	9.8
		50	Feed	syringe	was	refilled	with	feed	
	8 Up	34	16	7	47	40	40	3.7	13.0
	8 Dn.	18	16	46	7	39	40	3.5	10.5
	9 Up	2	16	7	7	39	40	3.5	14.0
		50	Feed	syringe	was	refilled	with	feed	
	9 Dn.	34	16	47	7	40	40	3.5	10.4
	10 Up	18	16	7	47	40	40	3.5	15.2
	10 Dn.	3	15	47	7	40	40	3.5	9.0
		50	Feed	syringe	was	refilled	with	feed	
	ll_Up	34	16	7	47	40	40	3.5	15.8
	ll Dn.	18	16	47	7	40	40	3.5	9.0
		50	Feed	syringe	was	refilled	with	feed	
	12 Up	34	16	7	47	40	40	3.5	13.0
	12 Dn.	18	16	47	7	40	40	3.5	11.2
	, 	46	Feed	syringe	was	refilled	with	feed	
	13 Up	30	16	7	47	40	40	3.5	15.0
	13 Dn.	14	16	47	7.	40	40	3.5	10.2

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			Reserv	voir	oir Reservo			
	I	reed	Readir	ıg	Displac	cement	: <u>Produ</u>	ct
Cycle	Syringe	e Volume	Botm.	Top	Botm.	Top	Botm.	Top
No.	Reading	g Fed	<i>/</i>	,		,		<i>(</i>)
<u>n</u>	<u>(c.c.</u>)	<u>(c.c.</u>)	<u>(c.c.</u>)	(c.c	.)(c.c.)	(C.C.) <u>(c.c.</u>)	(c.c.)
60	47	Feed	syringe	was	refilled	with	feed	
14 Up	31	16	7	47	40	40	3.5	14.6
14 Dn.	15	16	47	7	40	40	3.5	10.4
	50	Feed	syringe	was	refilled	with	feed	
15 Up	34	16	47	7	40	40	3.5	14.6
15 Dn.	18	16	7	47	40	40	3.5	10.0

.

TABLE 3(D). Parametric Pumping Experiment Run 4

System : Aniline - Toluene - n-Heptane Feed : Aniline Conc. = 0.6585×10^7 k gmole/c.c. = 0.6 Vol.% Toluene Conc. = 2.349×10^7 k gmole/c.c. = 2.5 Vol.% Operating Conditions :

$T_1 = 333^{\circ} K$	$\phi_{\rm B} + \phi_{\rm T} = 0.4$
$T_2 = 298^{\circ} K$	$\phi_{\rm B} = 0.05$
$\frac{\pi}{\omega} = 1200$ Sec.	$Q \frac{\pi}{w} = 40 \text{ c.c.}$

Feed			Reserv Readin	oir ng	Reservo Displac	oir Sement	Product	
Cycle	Syringe	Volume	Botm.	Top	Botm.	Тор	Botm.	Top
n n	(c.c.)	<u>(c.c.)</u>	(c.c.)	<u>(c.c</u>	<u>.)(c.c.)</u>	(<u>c.c.)</u>	<u>(c.c.</u>)	(c.c.)
	40		47	7				
l up	24	16	7	47	40	40	2.0	17.0
l Dn.	8	16	47	7	40	40	2.0	12.0
	50	Feed	syringe	was	refilled	with	feed	
2 Up	34	16	9	47	38	40	2.2	14.2
2 Dn.	17	17	47	7	38	40	2.2	13.0
3 Up	1	16	6	47	41	40	2.3	17.2
	50	Feed	syringe	was	refilled	with	feed	
3. Dn.	34	16	47	7	41	40	2.2	12.0
4 Up	18	16	7	47	40	40	2.1	16.8
4 Dn.	2	16	47	. 7	40	40	1.8	11.2
	50	Feed	syringe	was	refilled	with	feed	
5 Up	34	16	7	47	40	40	2.0	17.8
5 Dn.	18	16	47	7	40	40	2.1	10.0

		Feed		Reserv <u>Readin</u>	Reservoir Reading		ir <u>ement</u>	Product	
C J	rcle	Syringe Reading	Volume	Botm.	Тор	Botm.	Тор	Botm.	Тор
	<u>n</u>	(<u>c.c.)</u>	<u>(c.c.)</u>	<u>(c.c.</u>)	(c.c	.) <u>(c.c.</u>)(<u>c.c.</u>)	<u>(c.c.)</u>	<u>(c.c.</u>)
6	Up	2	16	. 7	47	40 ·	40	2.4	17.4
	- -4	50	Feed	syringe	was	refilled	with	feed	
6	Dn.	34	16	47	7	40	40	2.0	11.0
7	Up	18	16	7	47	40	40	2.4	16.6
7	Dn.	2	16	47	7	40	40	2.2	12.0
-		50	Feed	syringe	was	refilled	with	feed	
8	Up	34	16	7	47	40	40	2.1	17.0
8	Dn.	18	16	47	- 7	40	40	2.0	15.0
9	Up	2	16	7	47	40	40	2.2	16.2
		50	Feed	syringe	was	refilled	with	feed	
9	Dn.	34	16	47	7	40	40	2.2	12.0
10	Up	18	16	7	47	40	40	2.3	16.2
10	Dn.	2	16	47	7	40	40	2.1	12.6
-	-	50	Feed	syringe	was	refilled	with	feed	•,
11	Up	34	16	7	47	40	40	2.2	16.0
11	Dn.	18	16	47	7	40	40	2.0	13.8
12	Up	2	16	7	47	40	40	2,2	16.0
-	-	50	Feed	syringe	was	refilled	with	feed	
12	Dn.	34	16	47	7	40	40	2.0	13.0
13	Up	18	16	7	47	40	40	2.4	16.0
13	Dn.	2	16	47	7	40 [`]	40	2.0	12.0

· _	Feed	i	Reser Readin	voir ng	Reserv	voir acement	Produ	et
Cycle No.	Syringe Reading	Volume	Botm.	Top	Botm.	Тор	Botm.	Тор
<u>n</u>	(<u>c.c.)</u>	<u>(c.c.</u>)	<u>(c.c.</u>)	<u>(c.c.</u>) <u>(c.c.</u>)) <u>(c.c.</u>)	(c.c.)	(c.c.)
	50	Feed s	yringe -	was re	filled	with fo	eed	
14 Up	34	16	7	47	40	40	2.2	15.8
14 Dn.	18	16	47	7	40	40	2.0	13.0

TABLE 3(E). Parametric Pumping Experiment <u>Run 5</u>

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = $0.5486 \times 10^7 \text{k gmole/c.c.} = 0.5 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Operating condition :

$$T_1 = 333 \text{ K}$$

 $T_2 = 298 \text{ K}$
 $\frac{\pi}{\omega} = 1200 \text{ Sec.}$
 $\phi_T + \phi_B = 0.4$
 $\phi_B = 0.05$

		F	eed	Reserv Readir	voir	Reservo Displac	oir cement	: Produ	Product	
Сĭ	vcle	Syringe	Volume	Botm.	Top	Botm.	Тор	Botm.	Top	
r 	<u>n</u>	(c.c.)	(c.c.)	<u>(c.c.)(</u>	c.c.) <u>(c.c.</u>)(c	<u>)</u>	(c.c.)	<u>(c.c.)</u>	
		50		47	8					
1	Up	34	16	9 *	47	38	38	2.0	12.0	
1	Dn.	18	16	47	8	38	39	2.2	10.0	
2	Up	2	16	8	48	39	40	2.0	14.0	
		50	Feed	syringe	was	refilled	with	feed		
2	Dn.	34	16	47	8	39	40	2.0	12.0	
3	Up	18	16	9	47	38	39	2.2	16.0	
3	Dn.	1	17	48	8	39	3 9	2.0	11.0	
		32	Feed	syringe	was	refilled	with	feed		
4	Up	15	17	9	45	39	37	2.3	16.0	
		49	Feed	syringe	was	refilled	with	feed		
4	Dn.	33	16	47	- 7	38	38	2.0	11.0	
5	UP	17	16	7	45	40	38	2.3	17.0	
5	Dn.	l	16	47	5	40	40	2.0	10.0	
		50	Feed	syringe	was	refilled	with	feed		

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		Fe	ed	Reserv	voir	Reserv Displa	oir cement	Produc	et
Ċ	ycle	Syringe	Volume	Botm.	Top	Botm.	Тор	Botm.	Top
-	<u>n</u>	$(\underline{c.c.})$	(c.c.)	<u>(c.c.</u>)	<u>(c.c.</u>)(<u>c.c.</u>)	(<u>c.c.)</u>	<u>(c.c.</u>)	<u>(c.c.</u>)
e	5 Up	33	17	7	45	40	40	2.0	17.0
e e	5 Dn.	17	16	44	7	37	38	2.0	12.0
•	7 Up	1	16	7	46	37	39	2.2	15.0
		50	Feed s	yringe	was r	efilled	with f	eed	
-	7 Dn.	34	16	44	7	37	39	2.2	12.0
	3 Up	17	17	8	45	36	38	2.2	16.0
8	B Dn.	2	15	44	8	36	37	2.0	12.0
		50	Feed a	syringe	was r	efilled	With 1	leed	
ç	9 Up	34	16	8	45	36	37	2.2	16.0
() Dn.	18	16	46	6	38	39	2.0	13.0
10) Up	2	16	. 9	45	37	39	2.2	15.4
		50	Feed	syringe	was n	refilled	with 3	feed	
10	Dn.	34	16	45	9	36	36	2.0	12.0
1	L Up	18	16	9	45	36	36	2.3	15.4
1	L Dn.	2	16	45	9	36	36	2.0	12.0
		49	Feed	syringe	was :	refilled	with	feed	
1	2 Up	3 3	16	9	45	36	36	2.2	16.0
1	2 Dn.	17	16	45	9	36	36	2.2	12.2
1	3 Up	l	16	9	45	36	36	2.3	15.6
		50	Feed	syringe	was ;	refilled	with	feed	
1	3 Dn.	34	16	45	9	36	36	2.2	11.2
1	4 Up	18	16	9	45	36	36	2.2	16 0
1	4 Dn.	2	16	47	8	38	37	2.2	12.0

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TABLE 3(F). Parametric Pumping Experiment Run 6

System : Aniline - Toluene - n-Heptane

Feed; Aniline conc. = $0.329 \times 10^{-7} \text{k gmole/c.c.} = 0.3 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^{-7} \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Operating condition :

 $T_1 = 333 \text{ K}$ $T_2 = 298 \text{ K}$ $\overline{W} = 1200 \text{ Sec.}$ $\varphi_T + \varphi_B = 0.4$ $\varphi_B = 0.05$

	म	Reservoir		Reservo	Reservoir		Product	
Cycle	Syring	e Volume	Botm.	Top	Botm.	Top	Botm.	Top
<u>n</u>	(<u>c.c.</u>)	<u>(c.c.</u>)	<u>(c.c.</u>)	<u>(c.c</u>	<u>.)(c.c.)(</u>	(c.c.)	<u>(c.c.)</u> (<u>c.c.)</u>
	49	***	47	10				
l Up	38	11	9	43	38	33	2.4	6.4
l Dn.	27	11	44	8	35	35	2.2	3.0
2 Up	17	10	10	47	34	39	2.2	8.0
2 Dn.	7	10	46	10	36	37	2.0	3.5
	50	r'eed	syringe	was	refilled	with	feed	
3 U p	40	10	10	48	36	38	2.2	10.0
3 Dn.	30	10	47	12	37	36	2.0	· 4.0
4 Up	20	10	9	48	38	36	2.2	12.5
4 Dn.	10	10	46	10	37	38	2.2	6.7
5 Up	0	10	. 7	.48	39	38	2.0	13.0
** **	50	Feed	syringe	was	refilled	with	feed	
5 Dn.	40	10	46	9	39	39	2.2	. 3.1
6 Up	30	10	9	46	37	37	2.0	10.4
6 Dn.	20	10	46	9	37	37	2.2	5.0

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CJ	ycle	Feed	Volume	Reserv Readin Botm.	roir ng Top	Reservo Displac Botm.	ir <u>eme</u> nt Top	<u>Produc</u> Botm.	t Top
۲ 	<u>n</u>	$\frac{(c.c.)}{(c.c.)}$	rea c.c.)	(c.c.)	(c.c	<u>.)(c.c.)</u> (<u>c.c.)</u>	(c.c.)	<u>(c.c.)</u>
7	Up	10	10	9	46	37	37	2.1	12.2
7	Dn.	0	10	46	9	37	37	2.0	4.0
. •	• ••	50	Feed	syringe	was	refilled	with	feed	
8	Up	40	10	8	47	38	38	2.1	13.0
8	Dn.	30	10	47	9	39	38	2.0	4.5
9	Up	20	10	9	47	38	38	. 2.2	12.5
9	Dn.	10	10	47	9	38	38	2.0	4.0
10	Up	0	10	9	47	38	38	2.2	13.0
· ••••		51	Feed	syringe	was	refilled	with	feed	
10	Dn.	40	11	47	9	38	38	2.1	4.0
11	Up	30	10	9	47	. 38	38	2.0	12.0
11	Dn.	20	10	47	9	38	38	2.1	4.0
12	Up	10	10	9	47	38	38	2.0	11.5
12	Dn.	0	10	47	9	38	38	2.0	5.0
-		50	Feed	syringe	was	refilled	with	feed	•
13	Up	40	10	9	47	38	38	2.0	12.0
13	Dn.	30	10	47	9	38	38	2.0	4.5
14	Up	20	10	9	47	38	38	2.0	12.5
14	Dn.	10	10	47	. 9	38	38	2.0	3.5

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TABLE 3(G). Parametric Pumping Experiment Run 7

System : Aniline - Toluene - n-Heptane Feed : Aniline conc. = $0.329 \times 10^7 \text{k gmole/c.c.} = 0.3 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Operating condition :

$$T_1 = 333 \text{ K}$$

 $T_2 = 298^{\circ} \text{K}$
 $\frac{\pi}{\omega} = 1200 \text{ Sec.}$
 $\phi_{\mathrm{B}} = 0.05$

Feed			Reservoir Reading		Reservoir Displacement		Product	
Cycle No.	Syringe Reading	Volume Fed	Botm.	Top	Botm.	Top	Botm.	Top
<u>n</u>	(c.c.)	(c.c.)	<u>(c.c.)</u>	(c.c.) <u>(c.c.</u>)(<u>c.c.)</u>	(c.c.)	(c.c.)
· •••	41		45	10				
l Up	25	16	9	47	36	37	1.8	15.4
l Dn.	9	16	48	9	39	38	1.0	10.0
	51	Feed	syringe	was	refilled	with	feed	
2 Up	35	16	11	47	37	38	2.0	13.8
2 Dn.	19	16	46	7	3 5	40	2.0	14.0
3 Up	3	16	7	46	39	39	2.0	15.0
-	51	Feed	syringe	was	refilled	with	feed	•
3 Dn.	35	16	46	8	39	38	2.0	11.2
4 Up	19	16	8	46	38	38	2.2	16.0
4 Dn.	3	16	46	8	38	38	2.0	12.0
	50	Feed	syringe	was	refilled	with	feed	
5 Up	34	16	8	46	38	38	2.0	15.6
5 Dn.	18	16	46	9	38	37	1.8	12.5
6 Up	2	16	9	47	37	38	2.0	14.6

		Food		Reserv	<i>v</i> oir	Reservoir		Droduot	
CN	rcle	Svringe	Volume	Botm.	ig Tor	Botm.	Top	Botm.	Top
N N	10.	Reading	Fed				<u>r</u>		<u>F</u>
	<u>n</u>	<u>(c.c.</u>)	<u>(c.c.</u>)	(<u>c.</u> c.)(<u>c.</u> c	<u>.)(c.c.)(</u>	<u>c.c.</u>)	<u>(c.c)</u>	(c.c.)
-	• •	50	Feed	syringe	was	refilled	with	feed	
6	Dn.	34	16	46	9	37	38	2.2	12.0
7	Up	18	16	10	46	36	37	1.8	16.2
7	Dn.	2	16	46	9	36	37	2.0	12.8
		51	Feed	syringe	was	refilled	with	feed	
8	Up	35	16	10	46	36	37	1.8	15.2
8	Dn.	19	16	46	10	36	36	1.9	12.4
9	Up	3	16	10	46	36	36	1.8	16.0
		50	Feed	syringe	was	refilled	with	feed	
9	Dn.	34	16	46	. 9	36	37	2.0	12.5
10	Up	18	16	9	47	37	38	1.8	16.0
10	Dn.	2	16	46	10	37	37	1.9	12.6
	·	50	Feed	syringe	was	refilled	with	feed	
11	Up	34	.16	10	46	36	36	1.8	15.0
11	Dn.	18	16	46	9	36	37	1.8	14.0
12	Up	2	16	10	47	36	38	2,2	15.0
		50	Feed	syringe	was	refilled	with	feed	
12	Dn.	34	16	46	10	36	37	1.8	12.0
13	Up	18	16	9	45	37	35	2.0	17.0
13	Dn.	2	16	45	9	36	36	2.0	12.0
TABLE 3(G).(Continued)

			Reserv	voir	Reservo	bir	Deceder	.
Cycle	Syring Readin	eea e Volume g Fed	Botm.	ng Top	Botm.	Top	Botm.	Top
<u>n</u>	(c.c)	(c.c.)	<u>(c.c.)</u>	(c.c	:.) <u>(c.c.</u>)	(<u>c.c.)</u>	(c.c.)	<u>(c.c.)</u>
	51	Feed	syringe	was	refilled	with	feed	
14 Up	35	16	9	45	36	36	2.0	16.0
14 Dr	. 19	16	45	9	36	36	2.0	9.0
15 Up	3	16	9	45	36	36	2.0	16.0
	50	Feed	syringe	was	refilled	with	feed	
15 Dr	. 34	16	45	9	36	36	2.0	10.0

TABLE 3(H).Parametric Pumping Experiment Run 8

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = $2.743 \times 10^7 \text{k}$ gmole/c.c. = 2.5 Vol.%Toluene conc. = $2.349 \times 10^7 \text{k}$ gmole/c.c. = 2.5 Vol.%Operating condition :

T₁ = 333°K

 $T_2 = 298^{\circ} K \qquad \qquad \phi_T + \phi_B = 0.4$ $\frac{\pi}{\omega} = 1200 \text{ Sec.} \qquad \qquad \phi_B = 0.1$

·	Fee	ed	Reserv Readin	oir g	Reservoi Displac	lr ement	Produ	ct
Cycle	Syringe Reading	Volume	Botm.	Top	Botm.	Гор	Botm.	Top
<u>n</u>	(c.c.)	(c.c.)	(c.c.)	<u>(c.c.</u>) <u>(c.c.)(</u>	<u>c.c.)</u>	<u>(c.c.</u>)	(c.c.)
	48		46	5	-			· · · · · · · · · · · · · · · · · · ·
l Up	32	16	5	44	41	39	2.2	9.6
l Dn.	16	16	45	5	40	39	1.7	5.0
2 Up	0	16	5	45	40	40	4.0	13.0
-	49	Feed	syringe	e was	refilled	with	feed	
2 Dn.	33	16	45	5	40	40	3.2	9.0
3 Up	17	16	5	45	40	40	4.0	14.0
3 Dn.	1	16	45	5	40	40	3.6	11.0
-	49	Feed	syringe	was	refilled	with	feed	
4 Up	33	16	5	45	40	40	4.0	13.4
4 Dn.	17	16	45	5	40	40	3.5	9.4
5 Up	1	16	5	46	40	41	4.0	13.6
	49.5	Feed	syringe	was	refilled	with	feed	
5 Dn.	33.5	16	46	5	41	41	2.8	9.6
6 Up	17.5	16	5	46	41	41	4.0	13.2

Feed			Reserv	voir	Reserve	oir	Product		
Cycle	Syringe	Volume	Botm.	Top	Botm.	Top	Botm	. Top	
No. 	Reading <u>(c.c.)</u>	Fed (c.c.)	(c.c.)	<u>(c.c</u>	.)(<u>c.c.)</u> ((c.c.)	<u>(c.c.</u>)(<u>c.c.</u>)	
6 Dn.	2.5	16	45	5	40	41	4.0	10.4	
	49	Feed	syringe	was	refilled	with	feed		
7 Up	33	16	5	46	40	41	4.0	12.6	
7 Dn.	.17	16	45	5	40	41	4.0	10.6	
8 Up	1	16	5	46	40	41	4.0	11.6	
Br m	50	Feed	syringe	was	refilled	with	feed		
8 Dn.	34	16	45	5	40	41	4.0	8.0	
9 Up	18	16	5	46	40	41	4.0	13.7	
9 Dn.	2	16	45	- 5	40	41	4.0	12.0	
	49	Feed	syringe	was	refilled	with	feed		
10 Up	33	16	5	45	40	40	4.0	14.0	
10 Dn.	17	16	45	. 55	40	40	4.0	11.0	
11 UP	1	16	5	45	40	40	4.0	13.4	
1449 644-	49	Feed	syringe	was	refilled	with	feed		
11.Dn.	33	16	45	5	40	40	3.5	10.0	
12 Up	17	16	5	45	40	40	4.0	13.0	
12 Dn.	l	16	45	5	40	40	4.0	10.0	
	49	Feed	syringe	was	refilled	with	feed		
13 Up	33	16	5	45	40	40	4.0	13.5	
13 Dn.	17	16	45	5	40	40	4.0	10.0	
14 Up	l	16	-5	45	40	40	4.0	9.8	
	26	F≙ed	syringe	was	refilled	with	feed		
14 Dn.	10	16	45	5	40	40	4.0	9.8	

TABLE3(I). Parametric Pumping Experiment Run 9

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = 2.743×10^7 k gmole/c.c. = 2.5 Vol.% Toluene conc. = 2.349×10^7 k gmole/c.c. = 2.5 Vol.% Operating Condition :

$T_1 = 333^{\circ} K$	
$T_2 = 298^{\circ} K$	$\phi_{\rm T} + \phi_{\rm B} = 0.4$
$\frac{\pi}{\omega} = 1200$ Sec.	$\phi_{\rm B} = 0.05$

		Feed	Reserv	voir	Reserv Displa	oir cement	Produ	ct ·
Cycle	Syring	ge Volume	Botm.	Top	Botm.	Тор	Botm.	Top
n	(c.c.) <u>(c.c.)</u>	(c.c.)	<u>(c.c.</u>)(<u>c.c.)</u>	(<u>c.c.)</u>	(c.c.)	(<u>c.c.)</u>
	48		48	8	-			
l Up	32	16	5 *	46	43	38	2.0	3.8
l Dn.	16	16	. 45	5	40	41	2.2	3.7
2 Up	. 0	16	5	45	40	40	2.0	12.0
	50	Feed s	yringe v	was re	filled	with fe	ed	
2 Dn.	34	16	48	7	43	38	2.0	5.0
3 Up	18	16	8	48	40	41	2.0	13.5
3 Dn.	2	16	47	6	39	42	1.8	15.0
	50	Feed s	yringe	was re	filled	with fe	eed	
4 Up	34	16	8	47	39	41	1.8	13.2
4 Dn.	18	16	48	7	40	40	1.8	12.0
5 Up	2	16	8	47	40	40	1.8	18.0
	50	Feed s	yringe 1	was re	filled	with fe	eed	
5 Dn.	34	16	47	7	39	40	1.9	11.2
6 Up	18	16	9	47	38	40	1.9	14.2

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		_	Reserv	oir	Reservo	ir		•
Cuolo	Fee	ed Volume	Readin Botm.	ug Tron	Displac	ement Top	Produ Botm	Top
No.	Reading (c.c.)	Fed $(c.c.)$	<u>(c.c.)(</u>	<u>c.c.</u>) <u>(c.c.)(c</u>	<u>.c.</u>)	<u>(c.c.</u>)	<u>(c.c.)</u>
6 Dn.	2	16	48	8	39	39	1.8	12.0
	50	Feed	syringe	was	refilled	with	feed	
7 Up	34	16	8	48	40	40	1.8	17.0
7 Dn.	18	16	47	9	39	39	2.0	10.5
8 Up	2	16	9	48	38	39	2.0	16.0
	50	Feed	syringe	was	refilled	with	ľeed	
8 Dn.	34	16	47	9.	5 38	38.5	1.8	11.2
9 Up	18	16	10	47	37	37.5	2.0	17.0
9 Dn.	2	16	47	7	37	40	1.9	13.5
· • •	50	Fe ed	syringe	was	refilled	with	feed	
10 Up	34	16	7	47	40	40	2.0	18.0
10 Dn.	18	16	47	7	40	40	1.8	10.6
ll Up	2	16	6	48	41	41	2.0	17.4
~ -	50	Feed	syringe	was	refilled	with	feed	
ll Dn.	34	16	47	7	41	41	1.8	10.6
12 Up	17	17	7	47	40	40	2.0	17.4
12 Dn.	1	16	47	7	40	40	2.0	11.0
70 km	50	Feed	syringe	was	refilled	with	feed	
13 Up	34	16	7	47	40	40	2.0	17.0
13 Dn.	18	16	47	7	40	40	1.7	10.0
14 Up	2	16	7	49	40	42	1.8	13.4
	25	Feed	syringe	was	refilled	with	feed	
14 Dn.	9	16	48	7.5	5 41	41.5	1.8	12.0

TABLE 3(J) Parametric Pumping Experiment Run 10

System : Aniline - Toluene - n-Heptane

Feed : Aniline Conc. = $0.914 \times 10^7 \text{k gmole/c.c.} = 0.8 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Operating condition :

$$T_1 = 333^{\circ} K$$

 $T_2 = 298^{\circ} K$
 $\phi_B + \phi_T = 0.4$
 $\frac{\pi}{\omega} = 1200 \text{ Sec.}$
 $\phi_B = 0.05$

Feed			Reserv	Reservoir Reading		Reservoir Displacement		:t
Cycle	Syringe	Volume	Botm.	Top	Botm.	Тор	Botm.	Тор
<u>n</u>	(c.c.) (rea (c.c.)	<u>(c.c.</u>)	(c.c.) <u>(c.c.</u>)	(c.c.)	(c.c.) ((e.c.)
	48		48	7				
l Up	32	16	8	47	40	40	1.8	11.5
l Dn.	16	16	48	7	40	40	2.0	10.2
2 Up	Ö	16	7	47	41	40	2.0	15.6
	48	Feed	syringe	was r	efilled	with 1	feed	
2 Dn.	32	16	47	8	40	39	1.8	6.8
3 Up	16	16	8	47	39	- 39	2.2	13.5
3 Dn.	0	16	48	7	40	40	1.8	12.0
	48	Feed	syringe	was r	efilled	with 1	feed	
4 Up	32	16	8	47	40	40	1.8	16.5
4 Dn.	16	16	47	7	39	40	1.6	11.2
5 Up	0	16	8	- 47	39	40	1.8	15.4
	47	Feed	syringe	was r	refilled	with	feed	
5 Dn.	31	16	47	7	39	40	1.3	11.2
-								

Cycle	Fee Syringe	ed Volume	Reservo <u>Reading</u> Botm.	oir Top	Reserv Displa Botm.	roir <u>cement</u> Top	Produc Botm.	ot Top
n <u>n</u>	(c.c.)	(c.c.)	<u>(c.c.)(</u>	<u></u>)	(<u>c.c.</u>)(<u>c.c.</u>)	(c.c.)	(c.c.)
6 Up	15	16	8	47	39	40	1.9	15.4
	48	Feed	syringe	was	refille	d with	feed	
6 Dn.	32	16	47	7	39	40	1.5	11.5
7 Up	16	16	8	47	39	40	1.6	15.0
7 Dn.	0	16	47	8	39	40	2.0	10.0
	48	Feed	syringe	was	refille	d with	feed	
gU 8	32	16	8	47	39	40	2.0	15.2
8 Dn.	16	16	48	7	40	40	2.0	11.8
9 Up	0	16	8	47	40	40	2.0	14.6
	48	Feed	syringe	was	refille	ed with	feed	4
9 Dn.	3 2	16	48	7	40	40	2.0	11.2
10 Up	16	16	8	47	40	40	2.2	15.0
10 Dn.	0	16	48	7	40	40	1.6	12.0
	49	Feed	syringe	was	refille	ed with	feed	
ll Up	33	16	8	47	40	40	2.2	16.0
ll Dn.	17	16	48	7	40	40	1.9	8.0
12 Up	1	16	8	47	40	40	2.1	15.1
	48	Feed	syringe	was	refille	ed with	feed	
12 Dn.	32	16	47	8	39	39	2.2	10.5
13 Up	17	15	7	48	40	40	2.5	13.2
13 Dn.	1	16	47	8	40	40	2.2	11.0

TABLE 8(C). Analyses of Product Stream Samples

For The Parametric Pumping Experiment : Run 3

System. : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = 2.743×10^{-7} k gmole/c.c. = 2.5 Vol.% Toluene conc. = 2.349×10^{-7} k gmole/c.c. = 2.5 Vol.% $\phi_{\rm B} = 0.088$

2	ample No.	(Dilution)	Absorb.	Absorb.	<Увр: Уот	2>n
(0	ycle No.)	\ Factor /	at289ml	at263mjl	<u>Aniline</u>	<u>Toluen</u> e
Fe	eed	605	86.0	35.4	1.00	1.00
1	Botm.Down	121	81.0	57.0	0.188	0.710
2	Botm.Down	55	78.5	72.2	0.083	0.482
3	Botm.Down	36	84.0	82.0	0.058	0.369
4	Botm.Down	24	65.0	70.0	0.0299	0.214
5	Botm.Down	24	43.0	49.5	0.0198	0.159
6	Botm.Down	20	37.0	39.5	0.0142	0.103
7	Botm.Down	11	62.8	65.8	0.0132	0.0932
8	Botm.Down	8	59.5	76.7	0.0091	0.0851
9	Botm.Down	5	63.0	7 3. 2	0.0061	0.049
10	Botm.Down	4	58 .0	58.3	0.0045	0.0295
11	Botm.Down	3	60.0	55.0	0.0035	0.0201
12	Botm.Down	3	46.0	43.4	0.0027	0.0160
13	Botm.Down	3	36.5	35.5	0.0021	0.0132
14	Botm.Down	1	68.0	51.0	0.0013	0.0055
15	Botm.Down	1	58.0	43.6	0.0011	0.0047

TABLE8(D). Analyses of Product Stream Samples of Run 4

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = 0.6585×10^7 k gmole/c.c. = 0.6 Vol.% Toluene conc. = 2.349×10^7 k gmole/c.c. = 2.5 Vol.% $\phi_B = 0.05$

Sample	No. (Dilution)	Absorbance	Absorbance	<u>< YBP</u> Voi	<u>2>n</u>
<u>n</u>				Aniline	Toluene
Feed	242	54.0	40.0	1.00	1.00
1	88	46.0	61.4	0.03095	0.724
2	88	24.2	42.4	0.1630	0.542
3	55	23.8	55.0	0.100	0.461
4	40	19.5	43.0	0.0597	0.261
6	11	33.2	82.5	0.0279	0.140
7	11	21.5	60.0	0.0181	0.086
8	4	44.5	96.0	0.0137	0.058
9	3	34.2	82.5	0.00785	0.038
10	3	28.0	60.0	0.00642	0.027
11	3	20.0	42.0	0.0046	0.019
12	3	11.8	23.5	0.00271	0.0102
13	3	7.5	12.0	0.00172	0.0051
14	3	5.0	9.5	0.00115	0.0042

TABLE 8(E). Analyses of Product Stream Samples

For the Parametric Pumping Experiment : Run 5

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = $0.5486 \times 10^7 \text{k gmole/c.c.} = 0.5 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ $\phi_{\rm B} = 0.05$

Sa ((ample No.	(Dilution) ⁷	Absorb.	Absorb.	<u>-<Ув</u> У•	P2> i
	n n			at 20)m	Aniline	Toluene
Fe	ed	121	85.6	71.8	1.00	1.00
2	Botm.Down	100	33.8	53. 6	0.326	0.79
3	Botm.Down	66	38.5	72.4	0.245	0.73
4	Botm.Down	44	43.3	91.7	0.184	0.63
6	Botm.Down	33	16.4	73.0	0.0684	0.41
8	Botm.Down	16	21.5	92.2	0.037	0.25
9	Botm.Down	11	22.0	98.0	0.0233	0.185
10	Botm.Down	11	22.4	83.8	0.0237	0.155
11	Botm.Down	7	19.0	94.0	0.0128	0.113
12	Botm.Down	6	17.6	90.2	0.0102	0.093
13	Botm.Down	4	18.4	. 95.8	0.0071	0.066
14	Botm.Down	3	20.0	95.4	0.0058	0.049

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TABLE 8(F). Analyses of Product Stream Samples of Exp. Run 6

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = 0.329 x 10^7 k gmole/c.c. = 0.3 Vol.% Toluene conc. = 0.349 x 10^7 k gmole/c.c. = 2.5 Vol.% ϕ_B =0.05

(Dilution)	Absorb.	Absorb.	<ybpz>n Voi</ybpz>		
(Pac tor /	at 289mm	at 200mja	Aniline	Toluene	
121	51.6	61	1.00	1.00	
36	31	92	0.180	0.54	
36	13	39.5	0.075	0.234	
30	24.0	56.0	0.12	0.267	
16	37.8	90.0	0.096	0.23	
10	38.0	79.0	0.060	0.123	
10	28.5	45.0	0.046	0.066	
10	18.5	30.8	0.030	0.046	
9	27.0	22.5	0.039	0.0234	
4	74.0	52.5	0.047	0.0 2 2	
4	30.0	23.2	0.0192	0.0103	
4	36.0	23.0	0.023	0.0088	
4	24.5	21.8	0.0157	0.011	
	(Dilution) Factor	$ \begin{array}{c c} \text{Dilution} \\ \text{Factor} \\ \begin{array}{c} \text{Absorb.} \\ \text{at 289m} \\ \text{at 289m} \\ \text{at 289m} \\ \begin{array}{c} \text{at 289m} \\ \text{at 289m} \\ \begin{array}{c} \text{at 289m} \\ \begin{array}{c} \text{at 289m} \\ \begin{array}{c} \text{at 289m} \\ \begin{array}{c} \text{at 289m} \\ \end{array} \\ \begin{array}{c} 121 \\ \begin{array}{c} 51.6 \\ \end{array} \\ \begin{array}{c} 36 \\ 13 \\ \end{array} \\ \begin{array}{c} 36 \\ 13 \\ \end{array} \\ \begin{array}{c} 36 \\ 13 \\ \end{array} \\ \begin{array}{c} 30 \\ 24.0 \\ \end{array} \\ \begin{array}{c} 16 \\ 37.8 \\ \end{array} \\ \begin{array}{c} 10 \\ 38.0 \\ \end{array} \\ \begin{array}{c} 10 \\ 28.5 \\ \end{array} \\ \begin{array}{c} 10 \\ 18.5 \\ \end{array} \\ \begin{array}{c} 9 \\ 27.0 \\ \end{array} \\ \begin{array}{c} 4 \\ 74.0 \\ \end{array} \\ \begin{array}{c} 4 \\ 30.0 \\ \end{array} \\ \begin{array}{c} 4 \\ 36.0 \\ \end{array} \\ \begin{array}{c} 4 \\ 24.5 \end{array} $ } \end{array}	$ \begin{array}{c c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{$	$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	

TABLE8(G). Analyses of Product Stream Samples

For the Parametric Pumping Experiment Run 7

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = $0.329 \times 10^{-7} \text{k gmole/c.c.} = 0.3 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^{-7} \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ $\phi_{\text{B}} = 0.05$

Sample No. (cycle No.)	(Dilution)	Absorb. at 289mM	Absorb. at 263ml	<u><ybpz>n</ybpz></u> Yoi	
<u>n</u>				<u>Aniline</u>	Toluene
Feed	121	50	62	1.00	1.00
2	54	44.6	86.0	0.40	0.69
3	30	27.0	76.0	0.134	0.36
4	30	12.2	42.0	0.061	0.203
5	16	18.8	56.2	0.050	0.143
6	11	26.2	60.5	0.047	0.102
7	6	33.5	68.5	0.033	0.062
8	4	43.0	60.0	0.028	0.033
9	4	40.0	42.2	0.026	0.021
10	4	27.5	30.5	0.018	0.016
11	4	26.8	22.5	0.018	0.010
13	5	25.5	18.5	0.021	0.0095
14	3	32.5	23.5	0.016	0.0072
15	3	22.5	17.0	0.011	0 .0 054

TABLE8(H).Analyses of Product Stream Sample

For The Parametric Pumping Experiment Run 8

System : Aniline - Toluene - n-Heptane Feed : Aniline conc. = $2.743 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ $\phi_{\text{B}} = 0.1$

Sample No.		(Dilution) At	Absorb.	Absorb.	 	
	n				Aniline	Toluene
Fe	eed	726	72.0	30.0	1.00	1.00
2	Botm.Down	242	71.0	38.4	0.33	0.735
3	Botm.Down	264	60.0	34.0	0.304	0175
6	Botm.Down	44	63.0	86.8	0.052	0.54
8	Botm.Down	32	38.2	74.2	0.0232	0.36
10	Botm.Down	18	29.0	92.5	0.0098	0.27
12	Botm.Down	14	22.2	83.0	0.006	0.19
14	Botm.Down	9	21.0	93.5	0.00 35	0.142
		•				

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TABLE 8(I). Analyses of Product Stream Samples

For The Parametric Pumping Experiment : Run 9

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = $2.743 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ Toluene conc. = $2.349 \times 10^7 \text{k gmole/c.c.} = 2.5 \text{ Vol.\%}$ $\Phi_B = 0.05$

Sample No. (Cycle No.)	No. (Dilution)	Absorb. at 289mp	Absorb.	<ybpz>n</ybpz>	
	No.) (Factor/		at 263mm	Aniline Toluene	
<u> </u>		<u></u>		MITTHC	TOTACHE
Feed	605	86.5	35.5	1.00	1.00
2	242	47.5	24.2	0.22	0.43
4	121	70.0	42.2	0.16	0.46
6	55	76.0	68.2	0.08	0.45
8	40	56.2	66.2	0.043	0.356
10	27	38.8	67.8	0.02	0.275
11	22	35.6	69.4	0.015	0.234
12	18	34.8	74.2	0.012	0,208
13	15	29.0	74.0	0.0083	0.178
14	12	33.2	81.2	0.0076	0.155

For The Parametric Pumping Experiment : Run 10

System : Aniline - Toluene - n-Heptane

Feed : Aniline conc. = 0.914 x 10^7 k gmole/c.c. = 0.8 Vol.% Toluene conc. = 2.349 x 10^7 k gmole/c.c. = 2.5 Vol.% $\phi_B = 0.05$

Sample	No. (Dilution)	Absorb.	Absorb.	<>Bp2>n	
<u>n</u>	NO.) (Factor / at 209mp at 209mp	at 200mm	Aniline Aniline	Toluene	
Feed	242	72.0	44.6	1.00	1.00
2	66	91.4	91.4	0.346	0.76
3	55	58.3	83.8	0.184	0.66
4	36	51.4	96.6	0.106	0.530
5	36	36.8	77.2	0.076	0.432
6	25	30.6	83.0	0.044	0.334
7	24	22.5	67.8	0.031	0.266
8	20	21.0	68.8	0.024	0.227
9	18	17.5	58.8	0.018	0.175
11	11	15.8	60.0	0.010	0.11
12	7	17.4	77.5	0.007	0.092
13	5	17.4	84.5	0.005	0.072

TABLE8(J). (Continued)

Cycle No.	<u><ytb2>n</ytb2></u> Yoi			
	Aniline	Toluene		
8	1.466	1.451		
6	1.333	1.870		
4	1.500	1.354		
•				

Figure 10



Figure 11



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Figure 15



Figure 16



