

Mass Transfer Studies in Wetted-Wall Extraction Towers

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WETTED-WALL extraction towers have been used extensively on a laboratory scale, because they offer an opportunity to estimate the interfacial contact area, which is not definitely known in other extractors like spray, packed, and perforated plate towers; for fundamental studies in liquid-liquid extraction, this is the decided advantage of this apparatus over other extractors.

Pioneer work in the field of wetted-wall extraction towers has been done by Fallah, Strang, Hunter, and Nash (6, 7, 13, 14), who investigated the hydrodynamic characteristics of this apparatus in order to establish the effect of turbulence on mass transfer. Treybal and Work (15) further studied the hydrodynamic aspect of this column, and showed that the interfacial velocity of the film was affected by the flow rates of both phases, and was always in the direction of the film liquid. In studying the extraction of acetic acid from an aqueous film to a benzene core, they observed that the flow rate of one phase might have a strong effect on the film coefficient of the other phase. Bergelin, Lockhart, and Brown (1) extracted isopropyl alcohol from tetrachloroethylene to water, by contacting the liquids in a horizontal extraction column, and showed from photographic recordings the effect of either flow rate on interfacial behavior.

Comings and Briggs (3) extracted several solutes between benzene and water in a wetted-wall extraction tower with variable tower heights and diameters, and found that the film liquid rate affects the resistances of both the liquid phases, while the core liquid rate has a negligible effect on the water film resistance. While extracting acetic acid from a methyl isobutyl ketone (4-methyl-2-pentanone) core to a water film, Brinsmade and Bliss (2) found that neither flow rate influenced the film transfer coefficient of the other phase. Recently Murdoch and Pratt (10) studied the extraction rates of uranyl nitrate between water and each of the solvents, methyl isobutyl ketone and dibutoxy diethyl ether [bis(2-butoxyethyl)ether], and interpreted the over-all extraction coefficients in terms of the phase velocities and interfacial resistance. They found that the film resistances were of the same order of magnitude, while the interfacial resistance was 5 to 20% of the over-all for the ether, and 10 to 35% for the ketone as core solvents.

In view of the wave motion observed in the wall liquid, and the presence of nonwetted patches at a few points on the tube surface, which somewhat reduce the available interfacial contact area and make it difficult to estimate it with greater precision, most workers in this field expressed the extraction data in terms of the volumetric mass transfer coefficients (as Ka) instead of area mass transfer coefficients.

In spite of the comparatively narrow range of flow rates obtainable and the lower efficiency of mass transfer realizable, wetted-wall extraction towers are very useful for fundamental studies in liquid extraction, as they are helpful for preliminary study of the effect of operating variables on mass transfer. With this object in view, the present investigation was undertaken.

APPARATUS

The accessory equipment for the extraction tower is the same as that used in the companion article (11), except that a globe valve was used in place of the atmospheric vent. The wetted-wall column follows the design of Treybal and

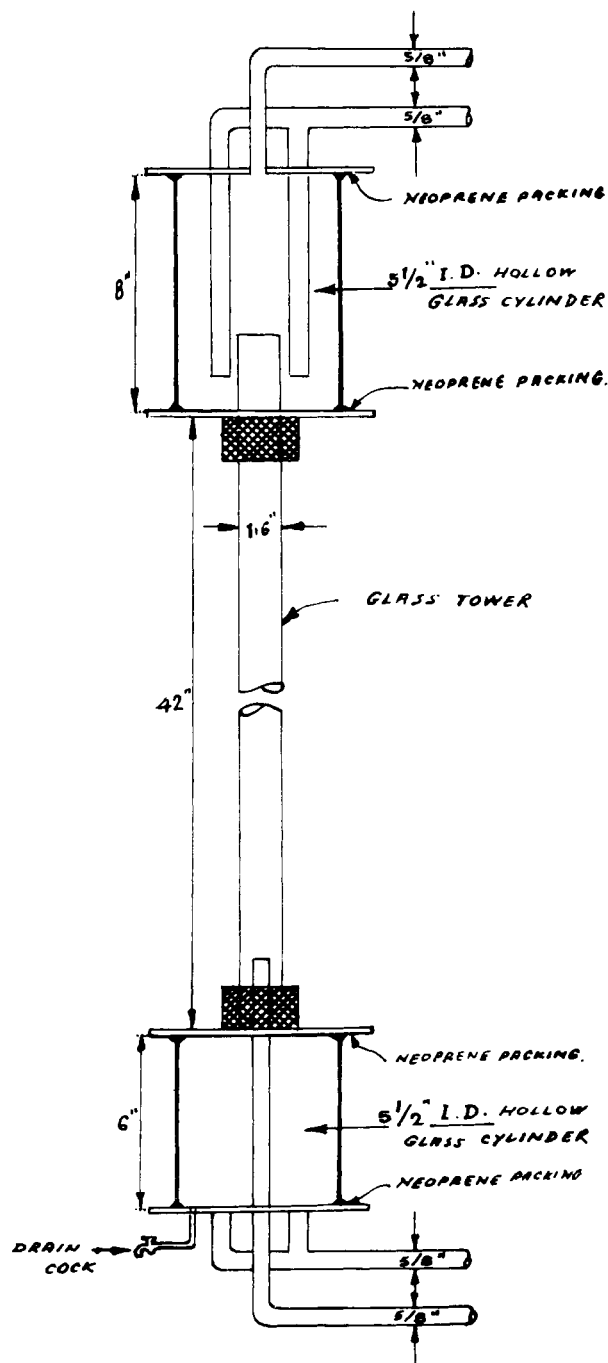


Figure 1. Details of wetted-wall tower and end sections

Wetted-wall tower, 1.6-inch diameter, 42 inches long

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Work (15) with slight modifications. A diagram of the column with the separating end sections is shown in Figure 1.

The 3-inch extension of the glass column into the upper end section of the column acts as a weir for the wall liquid, from which it flows down the inside surface of the column. To minimize transmission of the slight vibrations of the pumps to the column in operation, the tower end sections are connected to the flow lines by short pieces of neoprene tubing ($\frac{1}{2}$ -inch i.d.).

The core liquid feed line extends $1\frac{1}{2}$ inches above the gland nut of the bottom separating section (Figure 2). The main column, built of 1.6-inch i.d. borosilicate glass tubing, is 42 inches long (effective volume, 0.04890 cubic foot). A shorter column, 22.5 inches long but of the same diameter, was also used with the water-acetone-Pegasol system, with a view to studying the effect of column height on mass transfer (effective volume 0.02619 cubic foot). The separating end sections were $5\frac{1}{2}$ inches in inside diameter, with $\frac{3}{16}$ -inch walls. The bottom section was 6 inches long and the top section was 8 inches long.

The water from the Andhra University mains, supplied by Visakhapatnam Municipality, formed the wall (or film) liquid in all cases. A $\frac{3}{8}$ -inch globe valve is provided in the outlet water line; by adjusting it, the solvent-water interface is maintained about $\frac{1}{2}$ inch below the tip of the solvent inlet tube in all the runs.

Liquid-Liquid Systems. The following systems were chosen for the investigation, because the distribution of the solute between solvent and water in each case obeyed the simple distribution law fairly well in the range of concentrations studied, and because of the cheapness and availability of the chemicals.

1. Acetone-water-toluene
2. Acetone-water-Pegasol
3. Acetone-water-kerosine
4. Butyric acid-water-kerosine
5. Benzoic acid-water-kerosine
6. Benzoic acid-kerosine-0.1*N* sodium hydroxide solution.

Water, acetone, Pegasol, and toluene were from the same sources as in the companion article (11). Butyric acid (Naarden) was 99.8% pure, and benzoic acid (Baker's analyzed reagent grade) was 99.5% pure, determined by chemical analysis.

The equilibrium distribution data for these systems are available in the literature (8, 9, 11, 12) and are listed in Table I and were used in calculating mass transfer.

PROCEDURE

The glass column was thoroughly cleaned for each run with a strong solution of chromic acid; otherwise, the film of water would break away from portions of the glass surface and give rise to nonwetted patches. The tower was then flushed with tap water for about an hour. Water and solvent were mutually saturated with each other before being fed to the column. Water was admitted into the column at about the desired flow rate and the control valve in the outlet water line was adjusted until the lower interface remained about 1 inch below the tip of the solvent feed tube. The core liquid—i.e., the solvent solution having the desired solute concentration was then slowly introduced into the tower, and when it filled the tower, the water outlet valve was carefully adjusted to maintain the lower interface approximately constant ($\pm \frac{1}{2}$ inch on the desired level). The feed rates were gradually adjusted to the desired values. The $\frac{1}{4}$ -inch needle valves in parallel with the main control valves were used for final adjustment of flow rates.

Preliminary experiments have shown that steady conditions were obtained when the column contents were displaced at least four times. Consequently samples (about

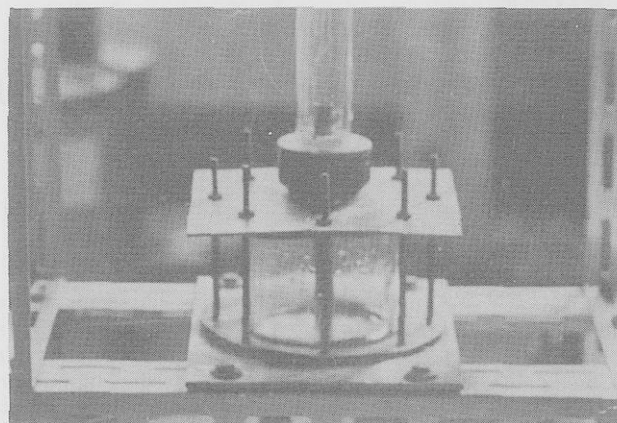


Figure 2. Bottom section of wetted-wall tower

Table I. Equilibrium Distribution Data for Acetone-Water-Solvents; Butyric Acid-Water-Kerosine, and Benzoic Acid-Water-Kerosine Systems

(Pound moles per cubic foot)

Series No.	C_W , Acetone-Water	C_T , ^a Acetone-Toluene	Series No.	C_W , Acid-Water	C_K , Acid-Kerosine
Distribution of Acetone between Toluene and Water at 30° C.			Distribution of Butyric Acid between Kerosine and Water at 30° C. ^b		
1	0.00992	0.00730	1	0.001993	0.0001528
2	0.02741	0.01985	2	0.003063	0.0003378
3	0.04439	0.03265	3	0.005368	0.0009802
4	0.05821	0.04529	4	0.007935	0.002010
Distribution of Acetone between Pegasol and Water at 30° C.			5	0.01025	0.003281
6	0.01444	0.006302	6	0.01444	0.006302
7	0.01659	0.008196	7	0.01659	0.008196
8	0.02000	0.01169	8	0.02000	0.01169
			Distribution of Benzoic Acid between Kerosine and Water at 29° C. ^c		
			1	0.00043	0.00042
			2	0.00068	0.00107
			3	0.00101	0.00236
			4	0.00116	0.00308
			5	0.00127	0.00368
			6	0.00136	0.00428
			7	0.00146	0.00500
			8	0.00156	0.00562
			9	0.00164	0.00636
			C_P , Acetone-Pegasol C_K , Acetone-Kerosine		
1	0.02985	0.00515			
2	0.06439	0.01155			
3	0.1115	0.02078			
4	0.1595	0.03233			
Distribution of Acetone between Kerosine and Water at 30° C.					
1	0.03932	0.00545			
2	0.06618	0.00965			
3	0.1045	0.01478			
4	0.2176	0.03389			
5	0.3265	0.05804			
6	0.3470	0.06385			

^a Data of Jayarama Rao (8).

^b Data of Krishna Murty (9).

^c Data of present authors (12).

Table II. Summary of Results of Mass Transfer in Wetted-Wall Extraction Tower

(Tower 1.6-inch i.d., 42 inches long)

System and Direction of Extraction	Series	Number of Runs	
		Total	Rejected ^a
Acetone-toluene-water (T → W)	A	31	...
Acetone-toluene-water (W → T)	B	15	1
Acetone-Pegasol-water (P → W)	C	27	...
Acetone-Pegasol-water (P → W) (tube length = 22.5 inches)	D	19	...
Acetone-kerosine-water (K → W)	E	28	1
Butyric acid-kerosine-water (K → W)	F	38	3
Benzoic acid-kerosine-water (K → W)	G	51	6
Benzoic acid-kerosine-0.1 <i>N</i> sodium hydroxide (K → 0.1 <i>N</i> NaOH)	H	24	1

^a Runs rejected because of poor material balances.

250 ml. each) of outlet water, inlet solvent, and outlet solvent streams were taken 10 minutes after attainment of the steady state. The flow rate of each phase was checked periodically, once every 5 minutes, by measuring the volumes of the effluent streams for a known interval of time. The solvent feed was discontinued, the solvent in the tower displaced by water, and then the whole tower drained and thoroughly cleaned before the next run.

The acid solutes (butyric and benzoic acids) were estimated by titration of the sample with standard alkali, and the solute acetone was determined by the hydroxylamine method.

All the runs were taken at room temperature, which varied from 29° to 32° C. during the experimental work. This slight variation of temperature had a negligible effect on the equilibrium distribution curves of the systems studied. The wall liquid rate was varied between the lower limit below which the tube surface was not completely wetted, and the upper limit above which the film flow no longer persisted and the film showed a greater tendency

to tear away from the tube wall and fall in thick streams.

Measurements of wall liquid film thickness were not made, as they were not particularly satisfactory, because of the observed phenomena of wave motion and nonwetted patches, which were rather difficult to eliminate completely.

Besides flow rates, other operating variables studied included direction of extraction, column height, and extraction, followed by chemical reaction at the interface. Acetone was extracted from toluene (core) to water (film) and vice versa, to find the effect of the direction of extraction on mass transfer. Acetone was extracted from Pegasol (core) to water (film) in two columns 42 and 22.5 inches high, respectively. Benzoic acid was extracted from kerosine (core) to water (film), and the film liquid, water, was replaced by 0.1N sodium hydroxide, for extracting the acid from the kerosine core to study extraction accompanied by chemical reaction.

Runs were taken at different core liquid rates for each wall liquid rate. With satisfactory and stable operation, the core liquid rate could be varied over a fivefold range (10 to

Table III. Over-all Mass Transfer Data for Set of Representative Runs

Run No.	V_W	V_C	C_{W1}	C_{W2}	C_{S2}	C_{S1}	N_{av}	MBE	K_{Wa}	K_{Sa}	HTU _{OW}	HTU _{OS}
Series A												
2	145.5	35.5	...	0.001916	0.01701	0.008753	0.003987	-4.93	4.919	6.704	29.58	5.295
5	145.5	10.7	...	0.000932	0.01704	0.003452	0.001956	-6.77	3.446	4.700	42.22	2.271
9	284.1	14.5	...	0.000740	0.01863	0.003270	0.003017	-5.73	5.090	6.943	55.82	2.09
11	277.2	48.3	...	0.001889	0.01767	0.006870	0.007289	+0.29	9.762	13.32	28.40	3.63
14	230.8	38.1	...	0.001665	0.01665	0.006970	0.005251	+4.06	7.211	9.833	32.00	3.88
17	230.6	42.7	...	0.001823	0.01678	0.006474	0.005998	-4.48	8.495	11.59	27.17	3.68
20	185.7	37.2	...	0.001797	0.01638	0.007184	0.004748	-2.54	6.520	8.881	28.48	4.19
25	180.2	18.1	...	0.001149	0.01698	0.004955	0.002962	-5.13	4.572	6.236	39.40	2.91
31	284.8	24.3	...	0.001123	0.01797	0.004575	0.004489	-1.89	6.896	9.406	41.20	2.58
Series B												
4	140.0	47.3	0.01506	0.01311	0.00059	0.00614	0.003730	+3.85	8.070	11.01	17.3	4.3
5	138.6	14.6	0.01731	0.01635	0.00072	0.00938	0.001809	+4.70	4.080	5.560	34.0	2.6
7	180.3	30.8	0.01361	0.01248	0.00089	0.00720	0.002766	+4.03	8.036	10.96	22.43	2.81
13	230.8	30.8	0.01460	0.01354	0.00053	0.00796	0.003297	+6.34	8.876	12.11	25.97	2.55
Series C												
1	284.1	45.8	...	0.001691	0.01552	0.004591	0.006872	-4.02	2.75	15.36	103.2	2.98
4	282.5	19.8	...	0.000713	0.01315	0.002444	0.002885	-5.17	1.63	9.09	173.4	2.18
22	138.6	44.5	...	0.003184	0.01566	0.005879	0.006118	+1.35	2.23	12.46	62.1	3.57
Series D												
6	145.5	19.4	...	0.001229	0.01526	0.005450	0.002574	-6.08	1.866	10.41	77.97	1.86
13	232.1	30.4	...	0.001242	0.01599	0.006342	0.004051	-1.51	2.685	14.98	86.45	2.03
19	235.0	45.1	...	0.001691	0.01731	0.008059	0.005691	-4.94	3.252	18.15	72.26	2.49
Series E												
4	145.0	24.1	...	0.001480	0.01414	0.004955	0.003030	-2.53	1.025	6.970	141.5	3.45
7	149.0	47.3	...	0.002219	0.01328	0.006177	0.004645	-1.44	1.488	10.12	100.1	4.67
16	227.2	39.7	...	0.001612	0.01351	0.004690	0.005000	+4.52	1.773	12.06	128.1	3.29
26	280.6	46.2	...	0.002140	0.01889	0.005714	0.008428	-1.36	2.265	15.40	123.9	3.00
Series F												
4	114.6	42.4	...	0.0004000	0.004094	0.003036	0.000631	+1.11	1.221	3.543	93.7	11.97
6	214.8	41.3	...	0.000269	0.003787	0.002336	0.000821	-3.74	1.890	5.484	113.80	7.53
8	146.9	13.7	...	0.000150	0.003666	0.001897	0.000323	-9.85	0.820	2.379	179.10	5.76
14	145.5	49.7	...	0.000358	0.004079	0.002999	0.000738	-3.22	1.451	4.206	100.10	11.82
20	256.4	12.5	...	0.000141	0.004050	0.000964	0.000522	-6.15	1.720	4.989	149.30	2.50
34	277.2	19.9	...	0.000166	0.003914	0.001597	0.000641	-0.50	1.758	5.093	157.7	3.91
37	282.7	51.9	...	0.000291	0.004172	0.002434	0.001201	-9.18	2.580	7.463	109.8	6.95
Series G												
2	138.6	55.2	...	0.000346	0.004130	0.003950	0.000675	-0.26	11.72			
4	231.0	57.8	...	0.000274	0.003880	0.002763	0.000892	-0.58	16.47			
9	284.1	11.5	...	0.000096	0.004395	0.001625	0.000412	-9.11	8.400			
24	277.2	46.6	...	0.000217	0.006235	0.004405	0.001009	-8.44	15.48			
37	79.7	26.0	...	0.000272	0.004310	0.003262	0.000341	-4.74	6.450			
42	83.2	42.8	...	0.000341	0.004725	0.003925	0.000437	-1.55	7.750			
Series H												
2	114.6	26.0	...	0.000784	0.006632	0.003519	0.001190	+5.16	16.58			
10	203.1	44.8	...	0.000624	0.006189	0.003548	0.001710	+3.03	24.29			
16	180.2	39.9	...	0.000569	0.005892	0.003392	0.001412	+1.06	20.52			
20	84.1	41.7	...	0.000768	0.006381	0.004734	0.000930	-1.50	12.33			

50 feet per hour) and the wall liquid rate about a threefold range (90 to 280 feet per hour). About 240 runs were taken altogether (Tables II and III).

CALCULATION OF MASS TRANSFER

Values of over-all coefficients, and over-all heights of transfer units were calculated from the equation used in the companion article (11).

For the runs using butyric acid, values of $K_K a$ and $(HTU)_{OK}$ were evaluated graphically. For all the runs in which aqueous sodium hydroxide solution was brought in contact with kerosine-benzoic acid solutions, the over-all coefficients were calculated, based on the concentration differences in pure water. The concentration difference at any point is equal to the concentration of benzoic acid in water which is in equilibrium with the measured concentration in the kerosine, the concentration in sodium hydroxide solution being zero.

COLUMN BEHAVIOR IN OPERATION

Nonwetting regions appear to form at few points even on the thoroughly cleaned tube surface, especially at low wall liquid rates and higher core liquid rates, and the tube surface remains completely wetted during operation when the wall liquid rate is maintained at least five times the core liquid rate. Because such nonwetted areas would somewhat reduce the effective area for mass transfer, the wall liquid rates are maintained high above the rates where such phenomena occurred.

Wave motion was observed in the wall liquid, and was

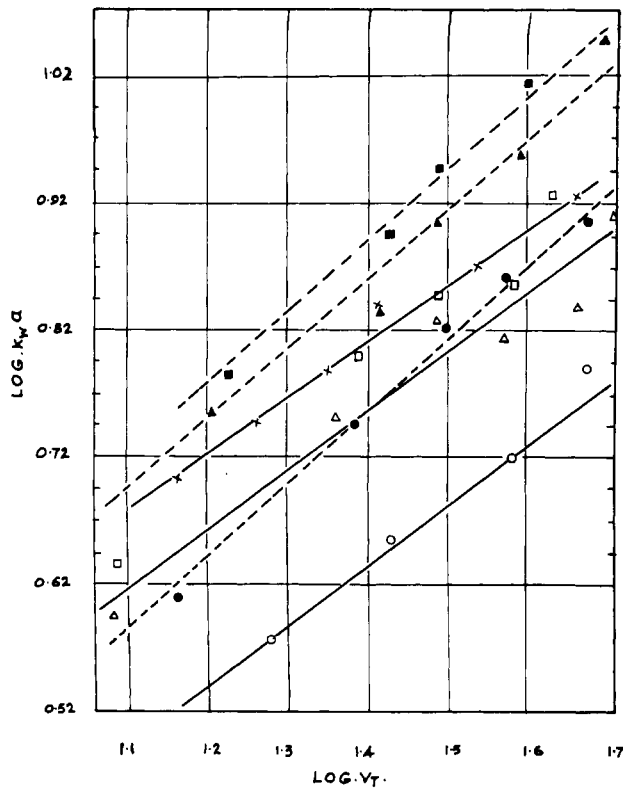


Figure 3. Extraction of acetone from toluene (core) to water (film) and in reverse direction

V_w , Ft./Hr.	Direction of Extraction	
	T → W	W → T
140	○	●
180	△	■
230	□	▲
280	×	

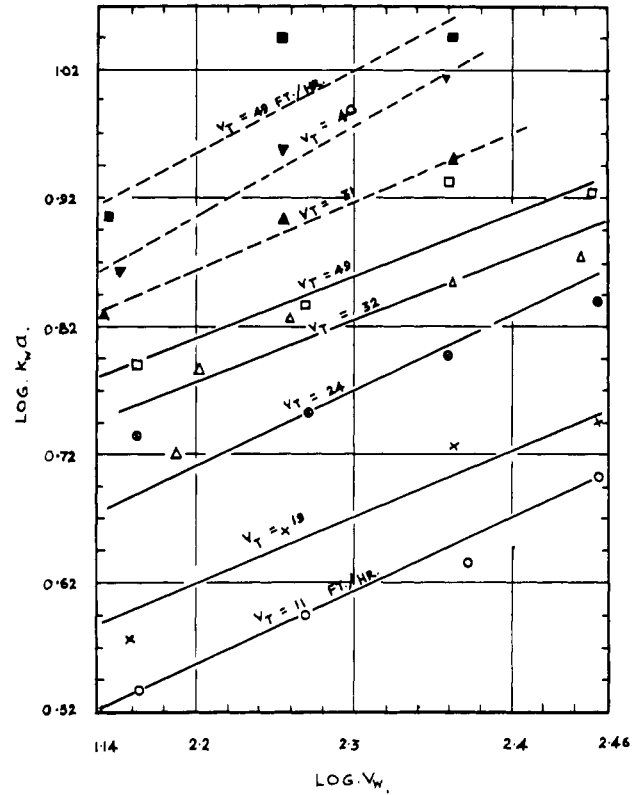


Figure 4. Extraction of acetone from toluene (core) to water (film) and in reverse direction

V_T , Ft./Hr.	Direction of Extraction	
	T → W	W → T
11	○	
19	×	
24	⊗	
31-32	△	▲
40		▼
49	□	■

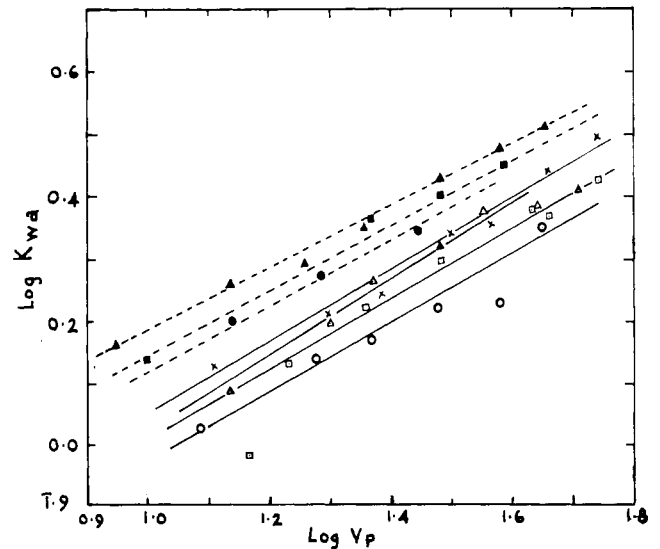


Figure 5. Extraction of acetone from Pegasol (core) to water (film) in 42.0- and 22.5-inch columns

V_w , Ft./Hr.	Tower Length, Inches	
	42	22.5
145	○	●
180	□	■
230	△	▲
280	×	

found to be a strong function of the wall liquid rate and tube length. At fairly low wall liquid rates ($V_W = 140$ feet per hour) there was no appreciable wave motion up to 15 inches down the weir, and at moderate rates ($V_W = 180$ to 200 feet per hour) rippling commenced 10 to 12 inches

below the weir. At higher rates of wall liquid, however, waves were set up as near as 4 to 6 inches down the weir. At any of the flow rates, this wave motion became increasingly marked as the tower bottom was approached. In view of the observed wave motion in the film liquid and

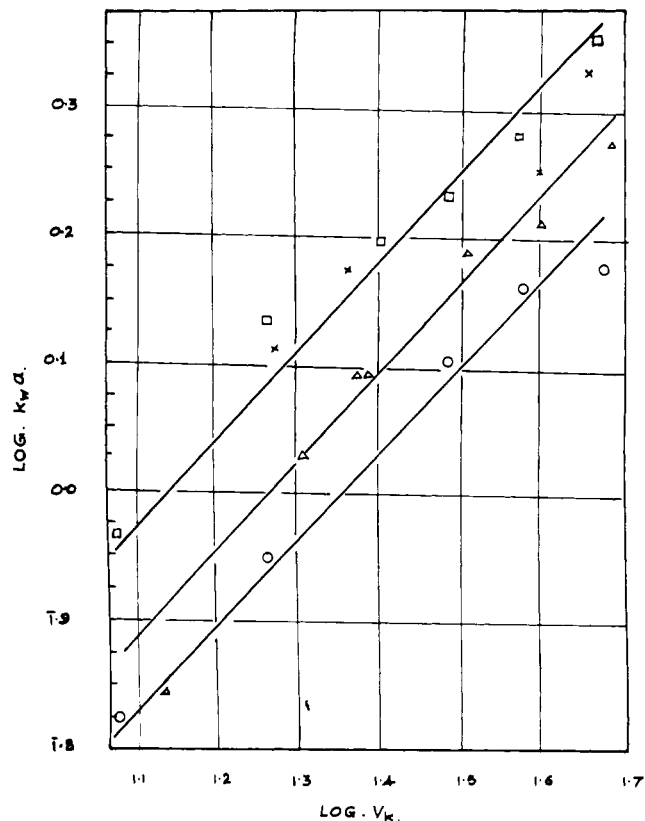


Figure 6. Extraction of acetone from kerosine (core) to water (film)

V_w , Ft./Hr.	V_w , Ft./Hr.
○ 140	× 230
△ 180	□ 280

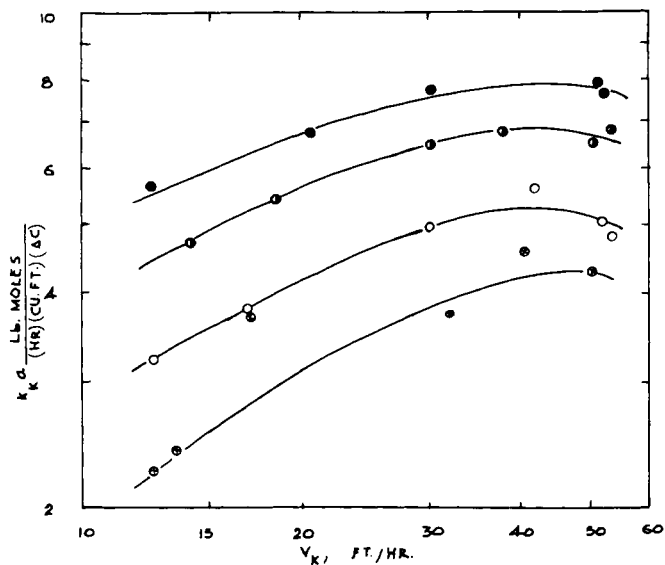


Figure 7. Extraction of butyric acid from kerosine (core) to water (film)

V_w , Ft./Hr.	V_w , Ft./Hr.
⊙ 140	● 230
○ 180	● 280

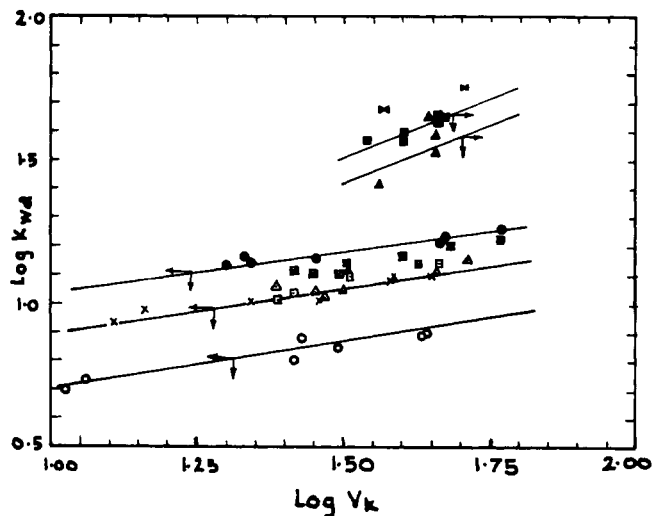


Figure 8. Extraction of benzoic acid from kerosine (core) to films of water and 0.1N sodium hydroxide

V_w , Ft./Hr.	Wall Liquid	
	Water	0.1N NaOH
80	○	
115	×	
140	△	▲
180	□	■
230	⊠	◆
230		◊
280	●	◈

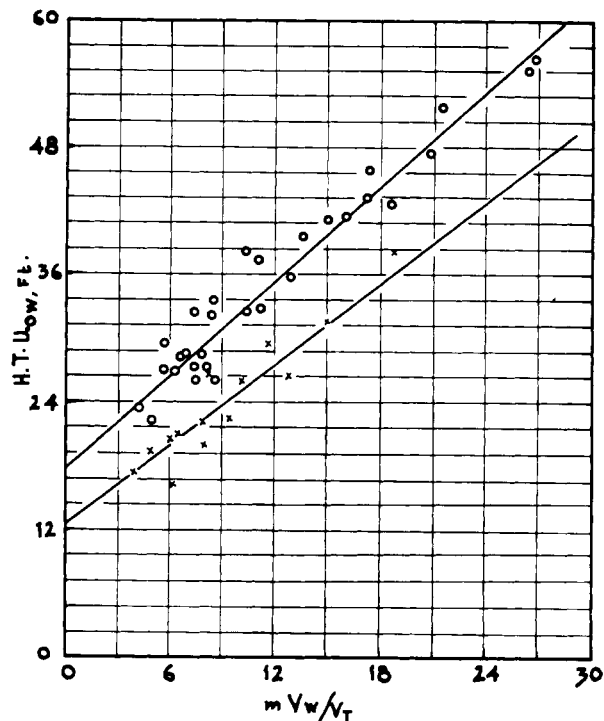


Figure 9. HTU correlation for extraction of acetone between toluene and water

Direction of Extraction	V_w , Ft./Hr.	V_T , Ft./Hr.
×	140-280	12-50

Table IV. Empirical Correlation of Capacity Coefficients with Flow Rates for Wetted-Wall Extraction Tower

System	Equation
Acetone-toluene-water (T → W)	$K_{Wa} = 0.072 V_W^{0.54} V_T^{0.47}$
Acetone-toluene-water (W → T)	$K_{Wa} = 0.060 V_W^{0.56} V_T^{0.56}$
Acetone-Pegasol-water (22.5-inch column)	$K_{Wa} = 0.06 V_W^{0.36} V_P^{0.54}$
Acetone-Pegasol-water (42.0-inch column)	$K_{Wa} = 0.04 V_W^{0.36} V_P^{0.56}$
Acetone-kerosine-water	$K_{Wa} = 0.0085 V_W^{0.56} V_K^{0.63}$
Butyric acid-kerosine-water $V_K = 12$ ft./hr.	$K_{Ka} = 0.0017 V_W^{1.25} V_K^{0.46}$
$V_K = 12$ to 40 ft./hr.	$K_{Ka} = 0.0010 V_W^{0.89} V_K^{0.46}$
Benzoic acid-kerosine-water Wall liquid water	$K_{Wa} = 0.120 V_W^{0.65} V_K^{0.33}$
Wall liquid, 0.1N NaOH	$K_{Wa} = 0.047 V_W^{0.65} V_K^{0.90}$

Table V. Extraction of Acetone from Toluene Core to Water Film

(1.6-in. diameter and 42-in. long wetted wall extraction tower.)

V_W , Ft./Hr.	V_T , Ft./Hr.	Over-all Transfer Coefficient, $K_S a$	
		Exptl.	Calcd.
277.2	34.3	10.17	9.84
284.1	25.9	9.48	8.65
228.7	24.3	8.76	7.96
185.7	37.2	8.88	8.96
186.4	22.9	7.80	7.15
145.5	47.6	8.45	8.85
145.5	23.2	7.62	6.75

the nonwetted patches at a few points on the tube surface, it was difficult to estimate the interfacial surface area, a , with accuracy, and hence all the mass transfer results were expressed only as Ka and not as K .

The short extraction tower (22.5 inches long) behaved much like the upper portion of the long tower (42 inches long), and the wave motion toward the tower bottom was much less than that with the long tower. Similar observations regarding the effect of tube length on wave motion in wetted-wall towers were made by several investigators (2, 4, 5, 15).

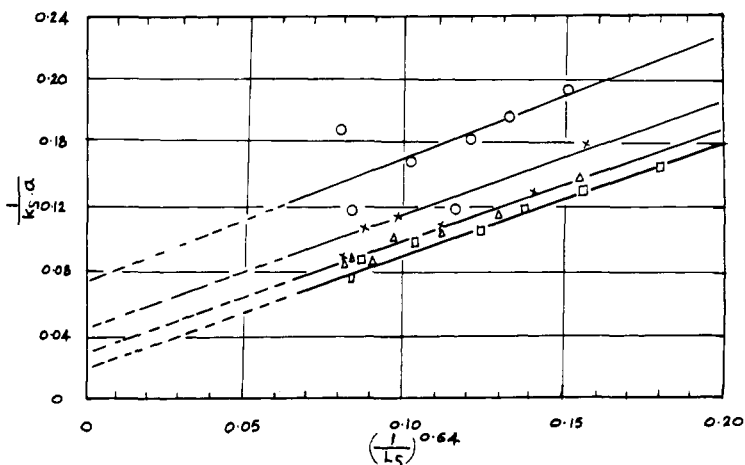


Figure 10. Individual film resistances in extraction of acetone from toluene (core) to water (film)

Method of Brinsmade and Bliss

	V_W , Ft./Hr.	V_T , Ft./Hr.
○	140	12-50
×	180	
△	230	
□	280	

RESULTS

The data are summarized in Tables II and III and shown in Figures 3 to 9. The empirical correlations of capacity coefficients with the flow rates are listed in Table IV, and the values of the film transfer units, obtained by resolving the over-all values, are listed in Table V for acetone-water-solvent systems.

Flow Rates. The over-all transfer coefficients have been found to be a function of both the wall liquid and core liquid flow rates and were empirically correlated as $Ka = \alpha V_W^m \times V_S^n$. Examination of Table IV reveals that the exponent of the flow rates is less than unity for all except the butyric acid-water-kerosine system, where the exponent of V_W is 1.25 at low values of V_K . The actual values of these exponents (m and n) and the constant (α) are somewhat different for each system, with m ranging from 0.45 to 0.65 and n from 0.35 to 0.65.

For similar conditions of flow rates, the over-all coefficients were nearly equal for acetone-water-kerosine and acetone-water-Pegasol systems, while those of the water-acetone-toluene system were four to five times greater. This may be due to the fact that the distribution coefficient of acetone is closer to unity in the toluene system, a condition which gives equal importance to both film coefficients and causes each phase flow rate to affect its film coefficient, with the ultimate result of increased over-all coefficient with increasing flow rates.

To evaluate the relative influences of flow rates on the individual film resistances, the mass transfer data of the acetone-water-toluene system, expressed as over-all transfer coefficients, were resolved into the film values, by following the graphical procedure of Brinsmade and Bliss (2). The final form of the equation deduced from graphical treatment of the data (Figure 10) could be written as

$$1/K_S a = (0.710/L_S^{0.64}) + (7.7/L_W) \quad (1)$$

which obviously showed that neither flow rate affected the film transfer coefficient of the other phase. This equation permitted calculation of the over-all extraction coefficients, as indicated in Table VI.

HTU's. The over-all heights of transfer units (HTU_{OW}),

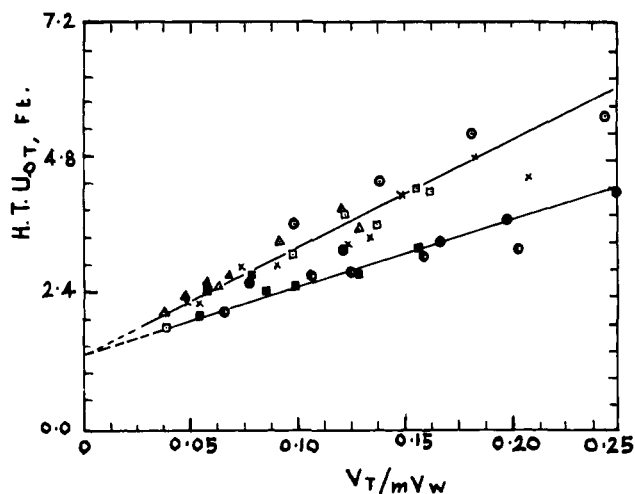


Figure 11. HTU correlation for extraction of acetone between toluene and water

Direction of Extraction	Symbol	V_W , Ft./Hr.	V_T , Ft./Hr.
T → W	○	140	12-50
	×	185	
	□	230	
	△	282	
W → T	●	140	12-50
	⊙	180	
	■	230	
	■	280	

shown graphically against $m(V_w/V_s)$ in Figures 11 to 13 were somewhat the highest for kerosine, and decreased in the order Pegasol to toluene as core liquid solvents. The film transfer units obtained by resolving the over-all transfer units by plotting according to Colburn's equations are listed in Table V for acetone-water-solvent systems.

An inspection of the film HTU values shows that the core liquid-film transfer unit is nearly the same for kerosine and Pegasol as solvents, but somewhat less for toluene. How-

Table VI. Film HTU Values for the Wetted-Wall Extraction Tower in Acetone-Water-Solvent Systems

Solvent (Core Liquid)	Direction of Extraction	HTU _w , Ft.	HTU _s , Ft.
Kerosine	K to W	60.0	1.60
Pegasol	P to W	40.0	1.70
Toluene	T to W	18.8	1.35
Toluene	W to T	12.5	1.30

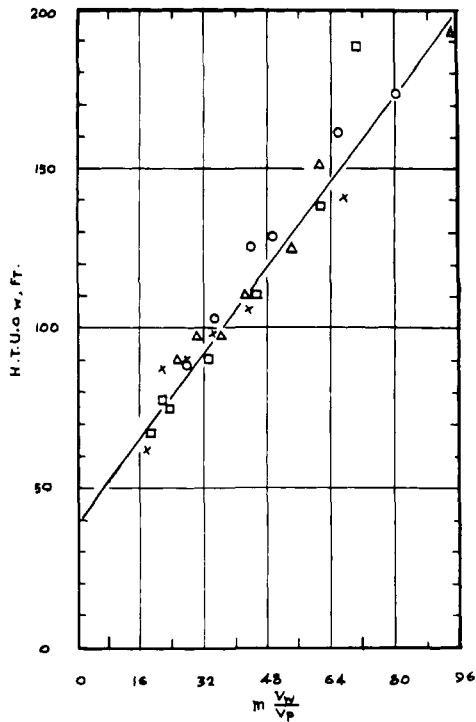


Figure 12. HTU correlation for extraction of acetone from Pegasol to water

	V _w , Ft./Hr.	V _p , Ft./Hr.
X	140	12-50
□	180	
△	230	
○	280	

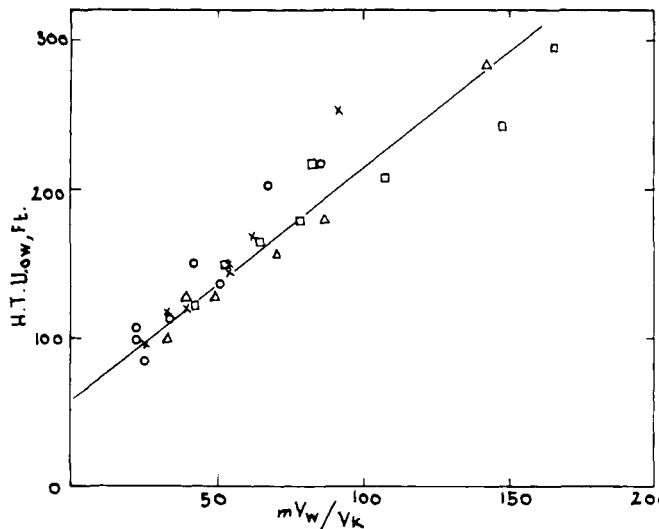


Figure 13. HTU correlations for extraction of acetone from kerosine to water

	V _w , Ft./Hr.	V _k , Ft./Hr.
○	145	12-50
X	185	
△	230	
□	280	

ever, the value of the water-film transfer unit differs largely from one solvent to another, and progressively decreases from kerosine to toluene through Pegasol. Reversing the direction of extraction in the acetone-water-toluene system had no appreciable effect on the core-film transfer unit, whereas it reduced the water-wall film unit from 18.8 to 12.5 feet—i.e., as much as 33%.

Column Height. It is clear from Figure 6 that over-all mass transfer coefficients, $K_w a$, for the extraction of acetone from Pegasol (core) to water (film), were higher for the short tower than for the long tower by about 13%. This might be due to the fact that wave motion in the wall liquid became more and more pronounced and turned more violent with increased tube length, thereby reducing the interfacial transfer area and hence the over-all coefficient.

Direction of Extraction. It is apparent from Figure 4 that the extraction rates expressed as $K_w a$ are increased by about 15% in extracting acetone from water (film) to toluene (core), as compared with those in the reverse direction. The toluene phase had a slightly greater effect on the transfer coefficient in the direction of extraction, water to toluene, than in the other direction, whereas the water rate influenced the coefficient in nearly the same way for the two cases, as is evident from Equations 2 and 3.

$$K_w a = 0.072 V_w^{0.54} V_T^{0.47} \quad T \rightarrow W \quad (2)$$

$$K_w a = 0.060 V_w^{0.56} V_T^{0.56} \quad W \rightarrow T \quad (3)$$

As the distribution of acetone was in favor of the aqueous phase, the principal diffusional resistance could be expected to lie in the toluene phase, and this might explain why the coefficient was affected by the toluene rate to a slightly greater extent in extracting acetone from water to toluene than in the reverse direction.

Diffusion Accompanied by Chemical Reaction. The distribution of benzoic acid was in favor of the kerosine phase [$(m = \frac{dC_K}{dC_W} = 6.4)$, in the range of concentrations used in the present work], and the major resistance to diffusion was expected to be in the water phase. With a view to reducing the water-film resistance and thereby improving the over-all transfer coefficient, extraction of benzoic acid has been carried out using 0.1N sodium hydroxide solution in place of water as the wall liquid. The results, plotted in Figure 8, indicate that the over-all transfer coefficients were increased 1½ times, and the effect of kerosine flow rate was much increased. The capacity coefficients were correlated as functions of flow rates by Equations 4 and 5.

$$K_w a = 0.120 V_w^{0.65} V_K^{0.33} \quad (\text{Wall liquid, water}) \quad (4)$$

$$K_w a = 0.035 V_w^{0.65} V_K^{0.90} \quad (\text{Wall liquid, 0.1N NaOH}) \quad (5)$$

CONCLUSIONS

The mass transfer rates for the extraction of acetone from Pegasol (core) to water (film) were about 13% higher for the short tower (22.5-inch effective length) than for the long tower (42 inches long), which indicates that end effects

might also prevail in wetted-wall towers as in other extraction equipment, which seem to be significant in short towers and become less marked with increased tower height.

The capacity coefficients were increased by about 15% in extracting acetone from water (film) to toluene (core), compared with those in the reverse direction; while the water-film rate influenced the coefficients in nearly the same way, the toluene rate affected them slightly more in the direction of transfer, water to toluene. This is probably because the principle diffusional resistance lies in the toluene phase, in view of the distribution of acetone in favor of the aqueous phase.

The rates of mass transfer were increased by about 50% when benzoic acid was extracted from kerosine core with 0.1*N* sodium hydroxide solution as the wall liquid, instead of pure water. This was obviously due to the chemical reaction of benzoic acid (solute) with the aqueous sodium hydroxide solution at the interface, and the subsequent reduction in the aqueous film diffusional resistance.

A preliminary empirical correlation of over-all transfer coefficients with flow rates as $Ka = \alpha V_W^m V_S^n$ revealed that with the acetone-water-solvent systems investigated, m ranged from 0.45 to 0.65 and n from 0.35 to 0.65.

The mass transfer data were also expressed in terms of HTU's, which are less sensitive to flow rates than Ka 's. An examination of the film HTU values, obtained by resolving the over-all HTU's by Colburn's method, showed that HTU_S are nearly the same for Pegasol and kerosine cores (1.6 to 1.7 feet), but somewhat less for toluene core (1.35 feet). However, the value of HTU_W decreased 60, 40, and 18.8 feet, respectively, for kerosine, Pegasol, and toluene. While HTU_S was nearly constant at 1.30 to 1.35 feet, HTU_W decreased from 18.8 to 12.5 feet on reversing the direction of extraction of acetone from toluene to water to water to toluene, showing the increased transfer rates in the latter direction.

In view of the nearly equal distribution of acetone between toluene and water ($m = dc_W/dc_T = 1.364$), both film resistances could be expected to be equally significant in controlling the diffusional resistance; hence, to analyze better the effect of phase flow velocities on the film resistances, the acetone-water-toluene system was chosen, and the extraction data of this system was analyzed by the graphical treatment of Brinsmade and Bliss (2). The equation finally obtained was:

$$1/K_S a = (0.710/L_S^{0.64}) + (7.7/L_W)$$

Each film resistance was not appreciably affected by the flow rate of the other phase for the system and type of apparatus used in the present work.

The performance of the wetted-wall extraction tower could not be directly compared with other extraction equipment like packed and spray columns in view of nonwetting and wave motion in the wall liquid, and the narrow range of flow rates obtainable in this type of tower. The necessity

of keeping the glass surface of the tube wall perfectly clean, for maintaining a continuous film of wall liquid, cannot be overemphasized.

NOMENCLATURE

- a = interfacial contact area per unit tower volume
 C = concentration, lb. moles per cu. ft.
 ΔC = over-all concentration difference, lb. moles/cu. ft.
 HTU_{OX} = height of over-all transfer unit based on X phase, feet
 HTU_X = height of individual transfer unit of X phase, feet
 ka = individual volumetric mass transfer coefficient, lb. moles/(hr.)(cu. ft.)(ΔC)
 Ka = over-all volumetric mass transfer coefficient, lb. moles/(hr.)(cu. ft.)(ΔC)
 m = slope of equilibrium line, dc_W/dc_S
 N/θ = rate of solute transfer, lb. moles per hr.
 L or V = superficial velocity, ft./hr., flow rate of phase, cu. ft./hr.)(sq. ft.)
 v = volume of tower, cu. ft.

Subscripts

- S = core liquid (solvent)
 W = wall liquid (water)
 K,P,T = kerosine, Pegasol, and toluene
 Av = average

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