Phase Separation and Mass Transfer in a Liquid-liquid Cyclone

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Liquid-liquid phase separation and mass transfer studies were made in a 4-in.-diam. cyclone of conventional construction. The cyclone was tested with oil-water volume phase ratios ranging from values of 1/3 to 9/1 and for total flows up to 24 gal./min., although most variables were studied at a feed rate of 10 gal./min. Kerosene or a white oil (vis. 9 centipoises at 77°F.) was used as the oil phase. Valve or line premixing was used to disperse the feed. Valve pressure drops were in the range of 0.1 to 1.0 lb./sq. in., and inlet drop sizes, where determined, were about 1 mm. The optimum cyclone geometry (volume, diameter of inlet, overflow and underflow lines) and the optimum split (overflow/underflow) were determined in terms of a phase-separation efficiency E_s.

At optimum geometry and split a number of mass transfer runs were made in which monobutylamine solute was transferred from the kerosene to the water phase. These runs indicated that E_s decreased but mass transfer efficiency increased as the feed rate or pressure drop across the mixing valve was increased.

The application of cyclones to liquid-liquid systems has been suggested (*3*, *7*) as a means of attaining efficient contacting and separation of the two phases. The vortex action leads to high centrifugal force, which aids the phase separation. Vortex motion in the column can be produced either by using a tangential inlet to the cyclone or by providing suitable directing vanes within a column. A number of devices, such as the Podbielniak and Luwesta extractors (*1*, *2*, *10*), employ centrifugal force obtained by internal moving parts to accomplish necessary mixing and settling. It was thought that the cyclone principle might lead to a low-cost device in which efficient phase contacting and separation could be achieved with no moving internal parts.

The available literature on liquid-liquid cyclones is sparse. Tepe and Woods (*15*) tried to separate isobutanol and water mixtures in 1- and 2-in. I.D. conventional-type cyclones and reported very poor separation. Dahlstrom (*3*) studied the separation of water-benzene + CCl_4 mixtures using a 3-in. I.D. cyclone. Netherlands Patent 67,244 (*7*) discusses, in a very general manner, the application of cyclones to the separation of immiscible liquids. Investigations made by Van Rossum (*16*) in 3- and 5-in. I.D. cyclones have indicated a fair degree of dewatering for oil-in-water emulsions.

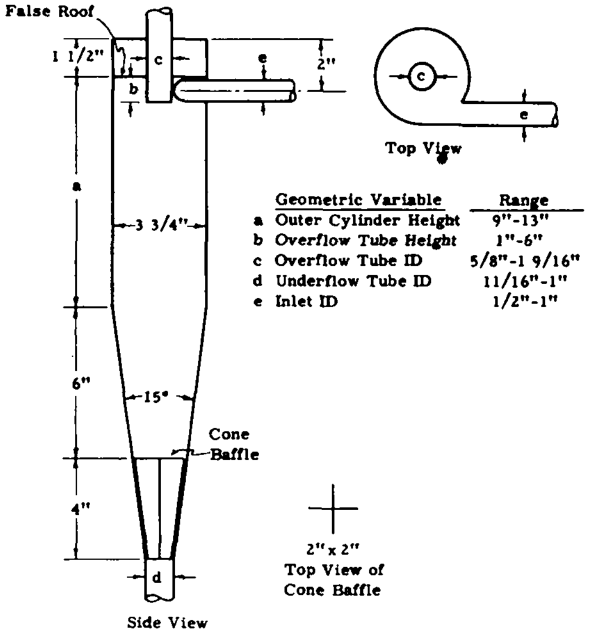

Fig. 1. Details of assembled cyclone.

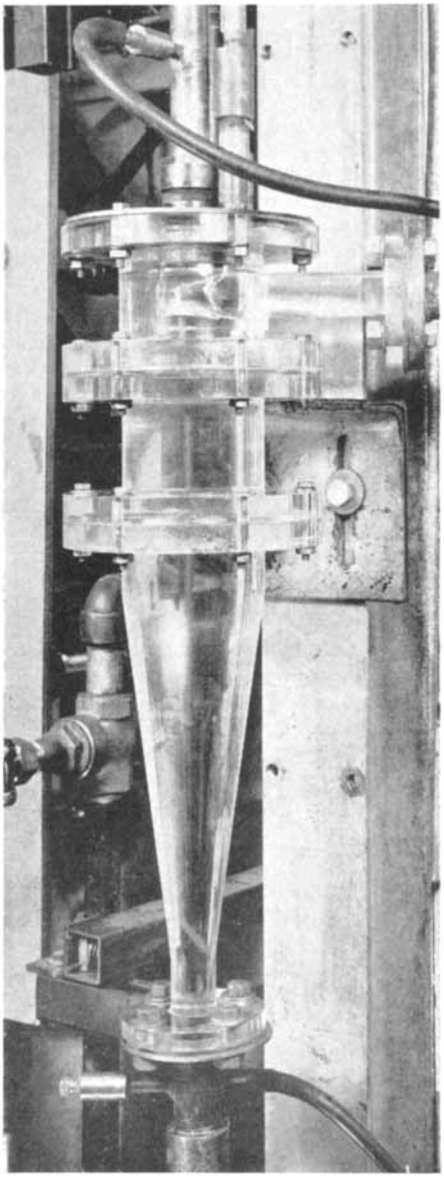

Fig. 1*a*. 3.75-in. I.D. cyclone.

This investigation was undertaken to determine the most favorable conditions for separating liquids *in* cyclone-type devices. Some extraction runs were also made in order to determine the relationship between the mass transfer efficiency and the degree of phase separation.

EQUIPMENT AND OPERATION

The cyclone used was of conventional design and had a 3.75-in. I.D. It contained a round tangential inlet, 15° cone bottom, and axial discharge lines; the volume of the cyclone and the dimensions of the overflow, underflow, and inlet were varied in the study. The cyclone is shown in Figures 1 and 1A.

The cyclone was constructed of flanged sections of Plexiglas for ease of visual observation and photographic recording. The flanged sections were grooved for O rings so that the internal walls of the column would be smooth and continuous at the flanged joints. The entry section consisted of a 3.75-in. I.D. cylinder, 4 in. high with a 1-in. I.D. entry tube, the outside surface of which met the vertical section tangentially midway between the ends of the section. Directly under the entry section there were one or more vertical sections of 3.75-in. I.D. and 4-in. height. The next lower section consisted of a 2-in.-long, 3.75-in. I.D. cylindrical section followed by a 15° cone 10 in. long. In addition to the plastic sections, the roof of the column consisted of a 7¼-in. O.D. stainless steel plate which was provided at its center with a gland fitting through which the overflow tube was first positioned and then fixed. Three overflow tube sizes were used, and therefore three cover plates with gland fittings were required. A "false roof" was connected to the overflow tube by means of a collar as shown in Figure 1. This put the effective overhead closure at the level of the highest point of the entry tube and reduced the strength of the energy-consuming "double eddy" (*17*), which is a toroidal circulation pattern in the section between the entry and column roof.

Kerosene-water and white-oil-water (oil viscosity 9 centipoises) were used for hydraulic and phase-separation tests in the cyclone. The system kerosene–water–*n*-butylamine was used for mass transfer runs since several other extraction devices have been evaluated with this system (*8*, *12*) in this laboratory. The kerosene used had an average density of 0.8 g./cc. and viscosity of 1.4 centipoises at the normal operating temperature (65° to 70°F.), and the white-oil density and viscosity were 0.84 g./cc. and 8.9 centipoises (75°F.). The distribution coefficient for butylamine C, expressed as (g. amine/g. water)/(g. amine/g. kerosene), was about 3.3 at normal operating temperature. The mutual solubility of kerosene-water and white oil–water was negligible at the temperature used.

The flow circuit is illustrated in Figure 2. Before a run began, the overflow and underflow valves were set to give the desired split (overflow/underflow volume ratio). Oil from a 275-gal. feed tank and water from the city main were metered and then brought together in a tee, mixed in a 1-in. entry line and gate valve, and transported

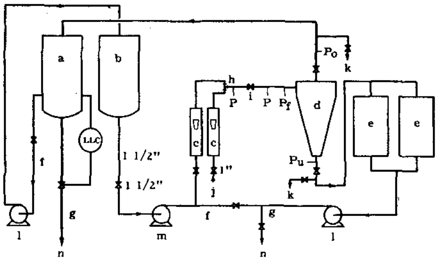

Fig. 2. Flow diagram of liquid-liquid cyclone.

to the column through the tangential entry. In most runs sufficient mixing was obtained in the 1-in. line to give a dispersion which was not separated completely in the cyclone, and the 1-in. gate valve was fully open in these runs. In one series of tests the mixing valve was throttled in order that the effect of degree of feed dispersion on cyclone efficiency might be studied. The pressure drop across the mixing valve was measured in all runs.

After a steady operation was achieved, usually after a period of 3 to 5 min., overflow and underflow samples were taken. Because of the high flow rates involved, it was necessary to take a bleed sample of the high-flow-rate stream, whereas a total sample of the other stream was feasible. Both overflow and underflow streams were gauged during the run. The high-flow-rate stream was metered in the 275-gal. storage tank by means of a float gauge; the low-flow-rate stream was measured by stick gauging in the receiving vessels or by calculating the flow into a graduated 5-gal. receiver. In almost all the runs, mass balances indicated a deviation of less than 3% for the total flow and less than 10% for the individual components.

For the mass transfer runs, *n*-butylamine was added to the kerosene feed before the run to make a 0.35% weight solution. The run was made as indicated above, and after the hydraulic data were recorded bleed samples of effluents were withdrawn from the sampling points shown in Figure 2. The effluent samples were two-phase mixtures, and the desired phase was removed from the mixture as rapidly as coalescence would permit. Samples were taken at 3 and 5 min. aftra the column appeared stable, and they checked well in all cases, an indication that mass transfer steady state was reached rapidly.

EQUATION FOR PHASE-SEPARATION EFFICIENCY

Tepe and Woods (*15*) derived separation efficiency by assuming that each effluent stream consists of one portion having the same composition as the feed and the rest being pure liquid, light phase in the case of the overflow and heavy phase in the case of the underflow. The efficiency is then defined as the sum of the pure-liquid rates expressed as a fraction of the total feed rate. The derivation of the separation efficiency is as follows:

The total rate of flow of light phase leaving the overhead minus the flow rate of light phase required for feed make-up is

$$Q_0 y_0 - Q_0(1 - y_0)\frac{y_f}{1 - y_f} = \frac{Q_0(y_0 - y_f)}{1 - y_f} \quad (1)$$

The total rate of flow of heavy phase leaving the underflow minus the flow rate of heavy phase required for feed make-up is

$$Q_u(1 - y_u) - Q_u y_u\left(\frac{1 - y_f}{y_f}\right) = \frac{Q_u(y_f - y_u)}{y_f} \quad (2)$$

Then by the definition of separation efficiency

$$E_s = \frac{Q_0}{Q_f}\left(\frac{y_0 - y_f}{1 - y_f}\right) + \frac{Q_u}{Q_f}\left(\frac{y_f - y_u}{y_f}\right) \quad (3)$$

By means of the material balance equations

$$Q_f = Q_0 + Q_u$$

$$Q_f y_f = Q_0 y_0 + Q_u y_u$$

Equation (3) can be reduced to

$$E_s = \frac{Q_0}{Q_f}\left[\frac{y_0 - y_f}{y_f(1 - y_f)}\right] \quad (4)$$

Van Rossum (*16*) has defined a separation number E_s', where

$$E_s' = \frac{Q_u}{Q_f}\left[\frac{y_f - y_u}{y_f(1 - y_f)}\right] \quad (4a)$$

By means of the same material-balance equations, it can be shown that E_s and E_s' are identical. It is recognized that E_s does not uniquely describe the separation; however, it was selected as a convenient guide for describing the effect of variables on cyclone performance in the present study.

SCOPE OF INVESTIGATION

Exploratory runs were first made at various total feed rates, oil-water flow ratios and effluent splits (overflow/underflow), to demonstrate the likely operating range of a liquid-liquid cyclone. The overflow/underflow split giving the maximum separation was then determined at selected geometric conditions for a feed rate of 10 gal./min. Geometric variables and feed premixing effects were then investigated in the established range of optimum effluent split, again for a feed rate of 10 gal./min. The effect of the following range of variables on E_s was determined in testing the 3.75-in. I.D. cyclone:

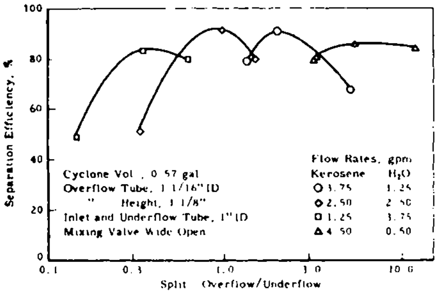

Fig. 3. Effect of split on separation efficiency; kerosene-water; feed rate 5 gal./min.

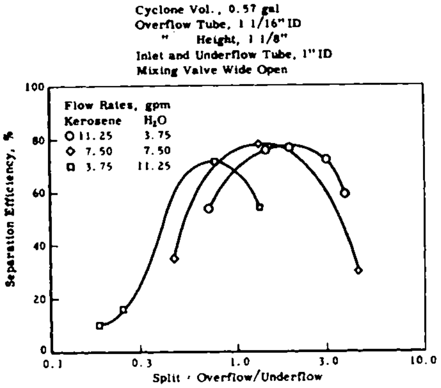

Fig. 4. Effect of split on separation efficiency; kerosene-water; feed rate 15 gal./min.

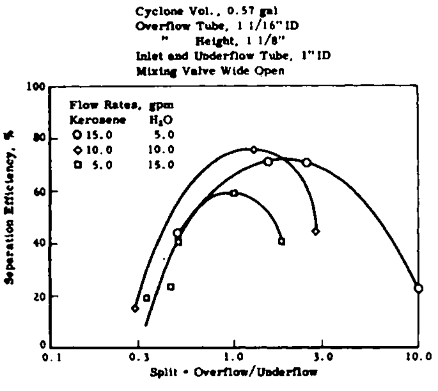

Fig. 5. Effect of split on separation efficiency; kerosene-water; feed rate 20 gal./min.

Geometric variable	Range
Overflow tube height	1–6 in.
Overflow tube I.D.	5/8-1-9/16in.
Cyclone height	19–23 in.
Cyclone volume	0.57–0.76 gal.
Underflow tube I.D.	11/16–1 in.
Inlet diameter	1/2–1 in.
Hydrodynamic variable	
Feed composition	10–90% volume oil
Product split (overflow/underflow)	1/10–12/1
Feed liquids	Kerosene-water White oil–water

Table 2. Optimum Flow Conditions in Cyclone, Low Premixing Intensity, 10 gal./min, Feed Rate

(Figures 7 and 8 based on these data)

Phase ratio, O/W	Optimum split, Q_0/Q_u	Maximum separation effect E_s, %	Volume fraction of water In overflow Q_0	In underflow Q_u	Approximate mixing valve ΔP lb./sq. in.
		Kerosene-water			
1/3	0.61	78	0.364	0.985	0.08
1/1	1.25	73	0.172	0.911	0.08
3/1	2.1	74	0.045	0.680	0.09
9/1	6.0	91	0.0045	0.673	0.06
		White oil–water			
1/3	0.70	77	0.400	0.995	0.06
1/1	1.25	87.5	0.106	0.992	0.05
3/1	2.4	86	0.021	0.797	0.05
9/1	3.4	72	0.016	0.384	0.06

The effect of a vortex breaker, also called the *cone baffle*, in the cyclone cone was studied in one series of runs.

The mass transfer efficiency of the cyclone plus mixing valve was then determined at the split and cyclone geometry corresponding to maximum phase-separation efficiency E_s.

Experimental data are tabulated in Tables 1, 2, and 3; Table 1* gives for the various geometries tested the inlet, overhead, and underflow rates, the split, overhead and underflow water concentrations, separation efficiencies, break times for the overhead and underflow mixtures, and pressure drops across the inlet mixing valve. Table 2 lists the optimum flow conditions for maximum phase separation, and Table 3 shows the mass transfer efficiency data. Pressure drops from cyclone inlet to underflow and from inlet to overflow were measured for all runs but the results have not been correlated with geometric and flow variables. However, the following is an example of the magnitude of the pressure drops. With kerosene-water feeds and a 1-in. I.D. entry and overflow tube, 1-1/16-in. overflow tube, 1-1/8-in. overflow height, and 0.566-gal. cyclone volume, the inlet to overflow and inlet to underflow ΔP's were roughly equal regardless of split. These ΔP's ranged from about 0.1 lb./sq. in. for a total flow of 5 gal./min. up to a maximum of about 4 lb./sq. in. for a total flow of 24 gal./min.

Effect of Feed Rate and Phase Ratio on Optimum Split

Total feed rate was varied from 5 to 25 gal./min. with the kerosene-water system and from 10 to 20 gal./min. for white oil–water in order to demonstrate the probable range of operation of a liquid cyclone. In these tests the cyclone volume was 0.57 gal. and the other dimensions were 1-in.-diam. feed inlet and underflow line, 1-1/16-in.-diam. overflow line, and 1-1/8-in. overflow height. At each feed rate the oil-water flow ratio was varied and the separation efficiency E_s was determined at several overflow/underflow splits. These exploratory studies were made with the 1-in. gate valve (mixing valve) wide open and, consequently, gave a low but variable mixing intensity in the inlet feed line, changing as the feed rate (and, accordingly, the line pressure drop) was varied. Since the degree of feed dispersion thus changed with increasing feed rate, one cannot satisfactorily isolate from these data the competing effects of increased centrifugal force and of decreased drop size due to breakup in the inlet line.

Figures 3, 4, and 5 show the effect of split on separation efficiency at different kerosene-water volume flow ratios (O/W) and at 5, 15, and 20 gal./min. total flow rates. It is seen that an optimum split corresponding to maximum separation efficiency is obtained and that the optimum split approaches the phase ratio O/W at low flow rates and approaches unity at high-flow rates. Other kerosene-water data, obtained at 10 and 25 gal./min. flow rates, substantiate these trends. Similar data for white oil–water at 10 and 20 gal./min. total rates, given in Figures 6 and 7, likewise, show the same shift of optimum split. A possible explanation of the observed effect of phase ratio on optimum split is as follows. At low flow rates the separated phases should be drawn off in proportion to their entry rates because the phases are clarifying at about the same rate as they are entering. At high flow rates, however, the remixing of the partially separated liquids in the cyclone becomes intense, and, since the main contribution to a high separation efficiency, E_s, comes from the liquid in greater concentration in the feed, the remixed emulsion should leave the column with the liquid which had a low feed concentration. This phenomenon then tends to make the optimum split equal to unity at high flow rates.

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*Table 1 has been deposited as document 5050 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for $1.25 for photoprints or 35-mm. microfilm.

At the highest line mixing intensity (20 gal./min. total flow) the low-viscosity kerosene system apparently gives an increase in separation efficiency as O/W ratio is increased, whereas the 9-centipoise white oil shows efficiency decreasing under the same conditions. Visually, the kerosene-water dispersion in the entry pipe was seen to change from a coarse dispersion of water drops to a fine dispersion of presumably kerosene droplets as O/W ratio was increased. The poorer separation found for the white-oil system at the higher feed rates and O/W ratios presumably reflects impeded settling due to viscous effects in the intermediate settling range ($500 > Re > 2$) plus the effects of feed rate and viscosity on equilibrium drop size at a given energy input per unit mass per unit time, as developed by Hinze (*4*).

The effect of phase ratio and split on the visual appearance of the cyclone was as follows: at O/W ratios of 1/3 and 1/1, a strong central core was visible, indicating that the oil phase was dispersed; at an O/W ratio of 3/1, the water phase apparently was dispersed; and at constant O/W ratio (O/W = 1/3 or 1/1), decreasing the split (underflow rate increased) caused the central core to

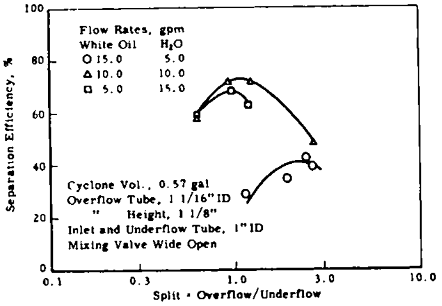


Fig. 7. Effect of split on separation efficiency; white oil-water; feed rate 20 gal./min.

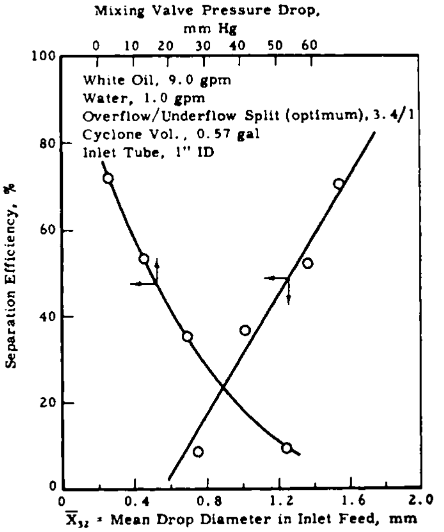


Fig. 9. Effect of feed premixing on phase separation.

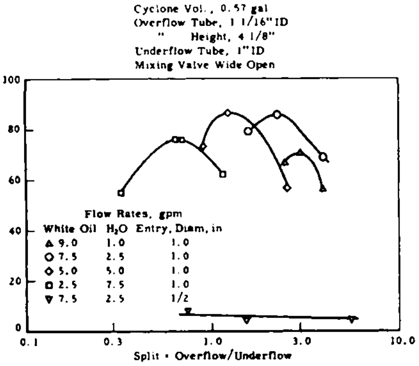


Fig. 6. Effect of split and entry diameter; white oil-water; feed rate 10 gal./min.

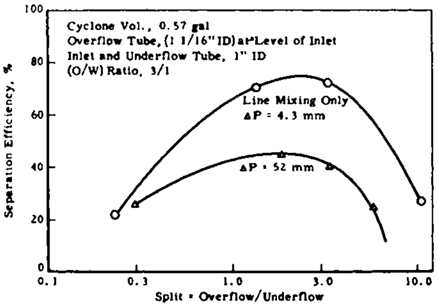


Fig. 8. Effect of mixing-valve pressure drop on separation efficiency; kerosene-water; feed rate 10 gal./min.

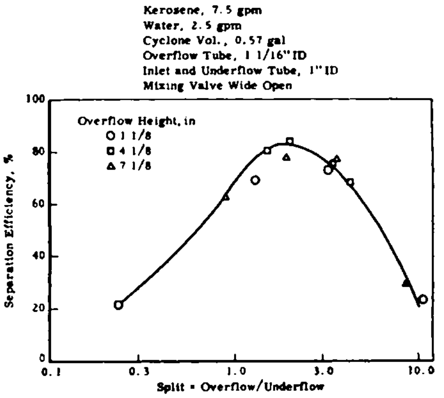


Fig. 10. Effect of overflow position on separation efficiency.

TABLE 3. MASS TRANSFER IN CONVENTIONAL CYCLONE

(Runs at approximately optimum split)

1-in. I.D. entry; 4-1/8-in. overflow height; 1-1/16-in. I.D. overflow tube; 1-in. I.D. underflow; 0.566-gal. cyclone volume; *n*-butylamine-kerosene-water

Run	Flow rates, gal./min. Kerosene	Water	Q_o, gal./min.	Q_u, gal./min.	$1 - y_o$ % water in Q_o	$1 - y_u$ % water in Q_u	Separation efficiency E_s, %	ΔP gate valve, lb./sq. in.	Amine concentration, g./g. Extract	Feed (kerosene)	Raffinate	Theoretical stages, %
140	5.0	5.0	5.05	5.13	7.53	87.7	81.6	0.0592	0.0008716	0.003404	0.001717	35.0
149	7.5	7.5	8.22	7.04	17.65	86.2	69.4	0.114	0.001065	0.003322	0.001444	45.8
147	10.0	10.0	8.47	11.78	22.0	6.93	46.5	0.204	0.001626	0.003322	0.001248	63.3
142	7.5	2.5	6.71	3.25	3.01	72.9	79.9	0.00	0.002546	0.003524	0.002309	45.4
151	11.25	3.75	10.38	4.78	5.05	70.6	74.6	0.0527	0.002431	0.003303	0.002094	49.2
145	15.0	5.0	13.27	6.96	10.82	53.3	51.1	0.204	0.003220	0.003190	0.001700	76.7
141	5.0	5.0	5.51	4.63	43.3	59.6	16.3	1.02	0.001884	0.003359	0.0007738	95.5
150	7.5	7.5	8.66	5.72	34.9	72.9	36.6	1.00	0.001752	0.003519	0.001052	76.4
148	10.0	10.0	8.92	11.20	32.6	63.6	30.7	1.02	0.001728	0.003470	0.0009829	79.2
143	7.5	2.5	6.77	3.35	16.00	46.4	34.0	0.925	0.003441	0.003568	0.001720	80.5
152	11.25	3.75	10.23	5.06	13.7	49.3	41.7	(1.0)	0.003591	0.003735	0.002054	73.6
146	15.0	5.0	13.84	6.27	18.3	43.6	26.3	1.09	0.003733	0.003362	0.001750	82.9
154	7.5	2.5	7.24	2.92	4.05	74.1	80.6	*	0.002198	0.003509	0.002497	36.7
155	15.0	5.0	13.78	6.09	13.78	51.9	42.5	*	0.003062	0.003391	0.001917	63.7

*Split tangential entry, no external mixing of feed streams.

lengthen and increase in diameter; the split at which this core penetrated the underflow was roughly equal to the optimum split for phase-separation efficiency.

Stroboscopic observations showed, under conditions where the core was visible, photographs was possible. The accompanying table shows the condition studied and the corresponding Sauter mean diameters (*13*), $\bar{X}_{32}$ of the droplet phase in the inlet feed. The values of $\bar{X}_{32}$, ΔP, and E_s given in the table are shown in Figure 9. The points for E_s and $\bar{X}_{32}$ fall on a straight line over the range studied.

Dispersion and Separation Characteristics White Oil–water

Feed = 10 gal./min., 90% volume oil in feed, split = 3.4/1

Mixing valve ΔP, mm. Hg	$\bar{X}_{32}$, mm.	%v Oil in overflow	%v Oil in underflow	% Separation efficiency, E_s
52.3	0.73	91.1	86.3	9.5
26.2	1.01	94.0	76.3	34.7
12.4	1.38	96.3	68.6	54.0
3.3	1.56	98.3	61.9	71.0

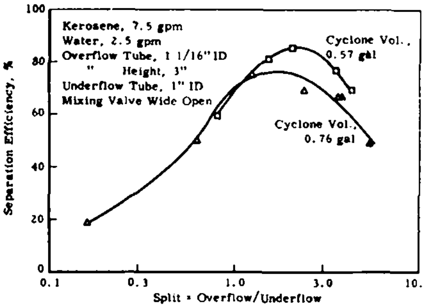
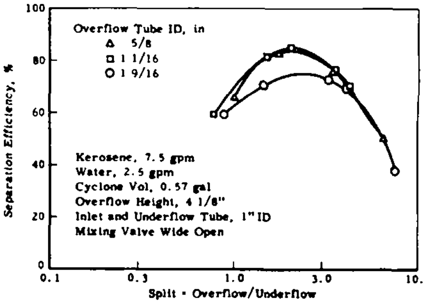


Fig. 11. ↑ Effect of cyclone volume on separation efficiency.

Fig. 12. ↓ Effect of overflow diameter on separation efficiency.



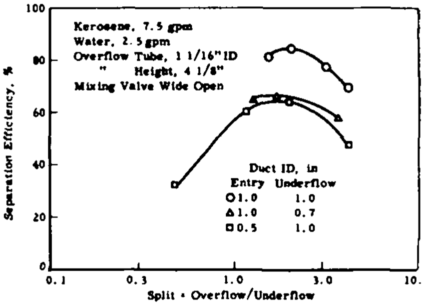
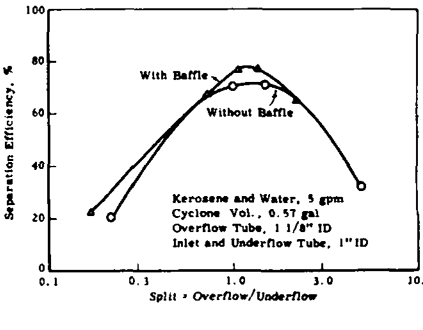


Fig. 13. ↑ Effect of underflow and entry diameter on separation efficiency.

Fig. 14. ↓ Effect of cone baffle on separation efficiency.



that the core was predominantly light phase and that droplets near the periphery of the core were circulating rapidly. Droplets near the center were rapidly streaming upward without circulation. Kelsall (*6*) and others (*14*) have also observed this streaming in the central region.

The balance of the phase-separation studies were made at a total feed rate of 10 gal./min. This was done to control, as closely as possible, the degree of feed dispersion at the cyclone inlet. Of course, some change in feed-drop-size distribution, at a given feed rate, is to be expected when the phase ratio is changed, but this change was presumed to be of a lower order than that caused by a change in feed rate.

EFFECT OF FEED PREMIXING ON PHASE SEPARATION

The effect of feed premixing on separation efficiency, at 10 gal./min. feed rate, was determined in terms of pressure drop across the mixing valve and of average drop diameter determined from photographs of the feed emulsion in the plastic tangential entry following the mixing valve.

The valve pressure-drop runs were made with kerosene-water at a total flow of 10 gal./min. and an *O/W* ratio of 3/1, and at two valve settings: (*a*) fully open, ΔP = 4.3 mm. Hg, and (*b*) ΔP = 53.3 mm. Hg. The results, as given in Figure 8, show that the maximum separation efficiency dropped from 75 to 45% as a result of the increased pressure drop, whereas the optimum split remained constant. This drop in efficiency corresponds to a decrease in oil recovery in the overflow from 87 to 80% and a decrease in oil purity from 95 to 87%.

The runs in which droplet diameters were measured were made with white oil–water at a flow rate of 10 gal./min., *O/W* ratio of 9/1, and optimum split of 3.4/1. These conditions were chosen to give a sufficiently low concentration of water droplets so that counting from

Neither the valve pressure-drop method nor the drop-diameter method can be expected to yield a quantitative picture from the small amount of data obtained here. Both sets of measurements, however, show the significant effect of pressure drop and, correspondingly, of emulsion droplet size on cyclone separation efficiency. It was this rapid decrease in separation efficiency with decreasing drop size, in fact, which discouraged further investigation extending into the 100-μ size region.

CYCLONE GEOMETRIC VARIABLES

The work described in previous sections was obtained with 0.566-gal. cyclone volume, 1-in.-diam. feed inlet and overflow line, 1-1/16 in.-diam. underflow line, and with the overflow line extending 1-1/8 in. or 4-1/8 in. below the false roof of the cyclone (*See* Figure 1.) These cyclone dimensions were then varied in turn to determine their effect on optimum phase separation. The diameter of the cyclone (3.75 in. I.D.) and the cone angle (15°) were not varied in this program. All geometric variables were tested at a feed rate of 10 gal./min.

Overflow height is defined as the distance from the false roof to the lower end of the overflow tube. This variable had very little effect on maximum separation efficiency or on position of optimum split, as shown in Figure 10. An overflow height of 4-1/8 in. was used in further work since it gave the maximum efficiency by a small margin.

It was observed in these studies of overflow height that a considerable swarm of water droplets traveled across the roof of the cyclone and down the outside of the overflow tube and were sucked into the tube rapidly when they reached its lower end. This bypassing phenomenon was also observed by Kelsall (*6*) and Pollack and Work (*11*) and undoubtedly contributes a major share to the water entrainment. Hughes et al. (*5*) have pointed out that this secondary flow is due to the fact that the laminar film on the roof has a pressure gradient superimposed on it, whereas the tangential velocity is slowed down by the skin friction. The net effect is an inward and then downward flow of liquid.

The *cyclone volume* was varied by changing the length of the cylindrical section; the cone section was not altered. The volume of the original installation (0.566 gal.) was increased 34% to 0.758 gal. by the addition of 4-in. cylindrical section. Figure 11 shows that the additional volume reduced the maximum separation efficiency from 85 to 76% but did not shift the optimum split position. These results support the idea that drop breakup, due to turbulence within the cyclone, rather than drop trajectory controlled the separation since providing added residence time for drop collection did not improve the separation efficiency.

The *internal diameter of the overflow tube* was studied with three tube sizes: 5/8, 1-1/16, and 1-9/16 in. I.D., each with 1/32-in. wall thickness. The inlet diameter was 1 in. I.D. Figure 12 indicates that about the same maximum separation efficiency, 85%, was obtained with 5/8- and 1-1/16-in.-diam. overflow but that maximum efficiency dropped considerably when the 1-9/16-in. overflow was used. Again, the optimum split did not shift noticeably as tube size was changed.

The parameter $d_{in}/(r_0 - r_i)$ may be thought of as an impingement ratio which influences phase separation in that ratios greater than unity cause excessive drop impingement on the outer wall of the overflow tube. Figure 12 shows that best separation was obtained for parameter values of 0.67 to 0.77 but that separation dropped as the parameter approached 0.95.

Two sizes of *underflow diameter* were studied, 1 in. and 11/16 in. I.D. Test conditions were 10 gal./min. feed rate. O/W ratio of 3/1, and 1-in. inlet diameter. Figure 13 shows that decreasing the underflow area 50% with the 11/16-in. tube decreased the maximum efficiency from 85 to 68%. The time for the underflow streams to completely clarify outside the cyclone was increased up to sixfold when tube size was reduced from 1 to 11/16 in.

The *diameter of the tangential entry* was reduced from 1 to 1/2 in. line, resulting in a severalfold higher line pressure drop and consequently the experiments presumably reflect the effect of mixing intensity as well as inlet size. Data obtained with the two entry sizes at a flow rate of 10 gal./min. and O/W ratio of 3/1 are shown in Figure 13 for kerosene-water and in Figure 6 for white oil–water. The smaller entry tube reduced maximum separation efficiency from 85 to 66% with kerosene-water and from 86 to 10% with white oil–water.

It was thought that improved separation efficiency might be obtained by installing a *baffle in the apex of the cone* (vortex breaker), thus breaking up the lower portion of the fluid core (light phase) and preventing its penetration into the underflow. A bafflle consisting of two vertically crossed sheets of metal, having the same cone angle as the cyclone and extending 4 in. above the opening of the 1-in. underflow tube, was installed. (*See* Figure 1.) The lower portion of the central core was broken up but quickly reformed into four smaller eddies inside the baffled quadrants. Figure 14 shows that the maximum separation efficiency increased only from 73 to 77% owing to the presence of the baffle.

From the limited tests described in this section, it is concluded that the *optimum geometry* of the 3.75-in. cyclone for phase separation is as follows:

Variable	Optimum dimension	Optimum dimension / cyclone diameter
Cyclone volume	0.566 gal. or less	
Overflow height	1-1/8-7-1/7 in.	0.3–1.9
Overflow diameter	1-1/16 in.	0.28
Underflow diameter	About 1 in.	~0.27
Entry diameter	About 1 in.	~0.27

None of these variables had any significant effect on optimum split. Most of the phase-separation studies listed in Table 1 were made at the optimum dimensions, as were all the mass transfer runs described in the next section.

The optimum-flow conditions for kerosene-water and white oil–water, at 10 gal./min.-feed-rate low mixing intensities, corresponding to the optimum structural dimensions given above, are listed in Table 2. The maximum separation efficiency E_s is given in terms of feed rate, O/W ratio, and optimum split, (Q_0/Q_u). Also shown are the calculated water concentrations of the underflow and overflow streams from which the purities and recoveries of the individual streams, rather than the combined-stream efficiency, E_s, may be estimated if desired. For example, for kerosene-water, O/W ratio = 3/1 and feed rate = 10 gal./min., the efficiency E_s = 74% when the split Q_0/Q_u = 2.1. The corresponding purities and recoveries are

Volume fraction oil in overflow = $y_0 = 0.955$

Volume fraction water in underflow = $1 - y_u = 0.680$

Recovery of oil in overflow

$$= \left(\frac{y_0}{y_f}\right)\left(\frac{Q_0}{Q_f}\right)$$

$$= \left(\frac{0.955}{0.75}\right)\left(\frac{2.1}{1 + 2.1}\right)$$

$$= 86.3\%$$

Recovery of water in underflow

$$= \left(\frac{1 - y_u}{1 - y_f}\right)\left(\frac{Q_u}{Q_f}\right)$$

$$= \left(\frac{0.680}{0.25}\right)\left(\frac{1}{1 + 2.1}\right)$$

$$= 87.7\%$$

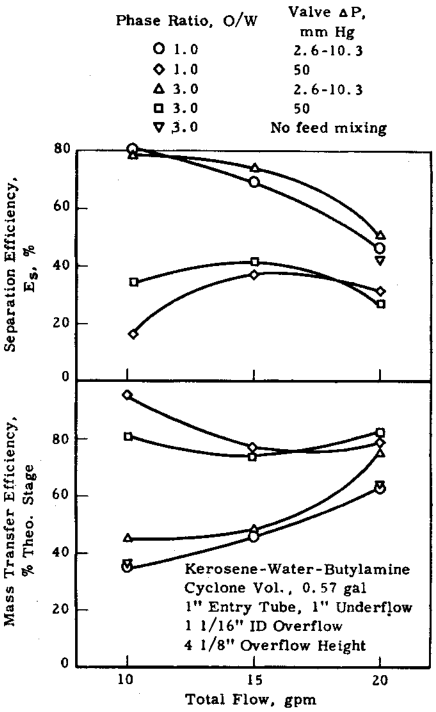


Fig. 15. Mass transfer in cyclone.

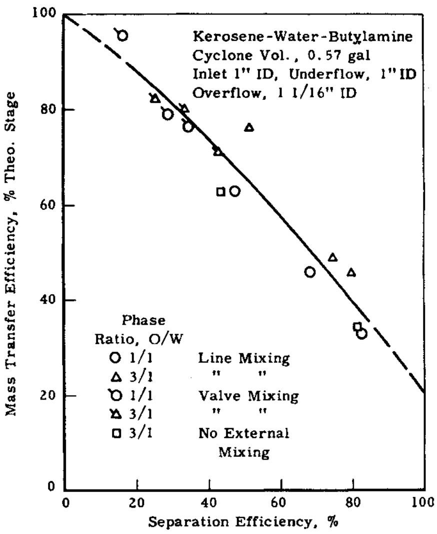


Fig. 16. Phase separation-mass transfer efficiency.

Thus oil of about 96% purity can be obtained at 86% recovery from a feed containing 75% oil in one cyclone when the residence time based on feed = (0.566 gal./10 gal./min.) = 3.4 sec., provided that the feed mixing intensity is low ($\Delta P \simeq 5$ mm. Hg), corresponding to a coarse dispersion.

MASS TRANSFER IN CYCLONE

Transfer efficiency was determined at the optimum phase-separation conditions described in the previous section (1-in. inlet and underflow, 1-1/16-in. overflow, 4-1/8-in. overflow height, 0.566-gal. cyclone volume, and optimum split). The fluid system used was kerosene-water with solute (*n*-butylamine) being transferred from the kerosene phase (raffinate) to the water phase (extract). This same system has been used in a number of other equipment studies in this laboratory (*8*, *12*) and the analytical problems involved in analyzing the phases for *n*-butylamine were discussed in one of these studies (*8*). Transfer efficiency was calculated as the fraction of one ideal stage, according to the method outlined by Perry (*9*) and given by the equation

$$N = \frac{\log \dfrac{Cy_2 - x_2}{Cy_1}}{\log \dfrac{C(y_2 - y_1)}{x_2}} \qquad (5)$$

where x_2 is the concentration of amine in the exit water (concentration in inlet water $x_1 \approx 0$), and y_1 and y_2 are the amine concentrations in the inlet and exit kerosene, respectively. The effect on efficiency of flow rate, phase ratio, and mixing-valve pressure drop was determined; data are tabulated in Table 3.

Figure 15 indicates that, with mixing valve fully open (inlet-line mixing only, $\Delta P = 2.6 - 10.3$ mm. Hg), mass transfer efficiencies ranged from 35 to 77%, increasing with feed rate and O/W ratio; over the same range of conditions, phase-separation efficiencies E_s decreased from 81 to 46%. With valve premixing ($\Delta P \sim 50$ mm. Hg), transfer efficiencies varied from 79 to 96%, with lowest values at flows of 15 gal./min.; separation efficiencies were only 16 to 41%, with highest values at 15 gal./min.

Two mass transfer runs were made in which the kerosene and water phases were introduced into the cyclone without external mixing; a split tangential inlet was used, the heavier water phase entering through the entry duct closer to the cyclone wall. The results, compared with line mixing alone and with valve mixing, in Figure 15 show that the cyclone body contributed significantly to total transfer. The ratio of transfer efficiencies is indicated as follows:

Transfer Efficiency Ratio, $(O/W) = 3/1$

Feed rate, gal./min.	No external mixing/line mixing	No external mixing/valve mixing, $\Delta P = 50$ mm. Hg
10	0.810	0.455
20	0.830	0.768

A plot of all the mass transfer efficiencies, as a function of separation efficiency, is given in Figure 16. Although there is some scatter present, transfer efficiency is approximately inversely proportional to separation efficiency. These data show clearly that high extraction efficiencies and a good clarification of both effluent streams cannot be obtained in one stage involving a valve mixer and one cyclone.

CONCLUSIONS

A liquid-liquid cyclone of conventional design gives very high capacities for the separation of coarse two-phase dispersions (drop diameter > 1 mm.). Phase-separation efficiency decreases rapidly as drop size is decreased, however, and poor separation of the dispersions formed at moderate mixing intensity (valve $\Delta P \sim 1$ lb./sq. in. = 50 mm. Hg) is obtained. Mass transfer efficiency and optimum phase-separation efficiency are roughly inversely proportional over the range of mixing intensities studied. Therefore, high extraction efficiency and good clarification of both effluent streams cannot be obtained in one stage involving a valve mixer and one cyclone.

Phase separation is not strongly influenced by cyclone geometry (overflow height and diameter, cyclone volume, underflow diameter, and, perhaps, entry diameter) but is principally a function of underflow/overflow split, feed rate, and mixing intensity for a given feed composition and for liquid viscosities of less than 9 centipoises.

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NOTATION

- C = distribution coefficient, (g. amine/g. water)/(g. amine/g. kerosene)
- d_{in} = equivalent diameter of cyclone inlet
- E_s = phase separation efficiency, defined by Equation (3)
- N = fraction of a theoretical stage
- (O/W) = oil-water volume phase ratio in feed
- ΔP = pressure drop across mixing device (line or valve)
- Q_f = volume flow rate of feed
- Q_0 = volume flow rate of overflow stream
- Q_u = volume flow rate of underflow stream
- (Q_0/Q_u) = effluent split
- r_i = outer radius of inner barrel of cyclone
- r_0 = inner radius of outer barrel of cyclone
- t_0 = time for overflow stream to clarify completely, min.
- t_u = time for underflow stream to clarify completely, min.
- x_2 = concentration of amine in exit water, g./g.
- $\bar{X}_{32}$ = Sauter mean diameter of disperse phase in inlet
- y_f = volume fraction of light phase in feed
- y_0 = volume fraction of light phase in overflow
- y_1 = concentration of amine in inlet kerosene, g./g.
- y_2 = concentration of amine in exit kerosene, g./g.
- y_u = volume fraction of light phase in underflow

LITERATURE CITED

1. Anon., *Chem. Eng.*, p. 186 (November, 1951).
2. ———, *Chem. Processing*, **15**, No. 6, 116 (June, 1952).
3. Dahlstrom, D. A., Northwestern Univ., personal communication (September, 1951).
4. Hinze, J. O., *A. I. Ch. E. Journal*, **1**, No. 3, 289, (1955).
5. Hughes, R. R., C. V. Sternling, and H. D. Evans, *Chem. Eng. Progr.*, **49**, 78, (1953).
6. Kelsall, D. F., *Trans. Inst. Chem. Engrs.*, (*London*) **30**, 87, (1952).
7. Netherlands patent 67,244 (Jan. 15, 1951).
8. Overcashier, R. H., H. A. Kingsley, and R. B. Olney, *A.I.Ch.E. Journal*, **2**, No. 4, 529 (1956).
9. Perry, J. H., "Chemical Engineer's Handbook," 3 ed., p. 554, McGraw-Hill Book Company, Inc., New York (1954).
10. Podbielniak, Walter, U. S. patent 1,936,532 and others.
11. Pollack, Arthur, and L. T. Work, *Trans. Am. Soc. Mech. Engrs.*, **64**, 31, (1942).
12. Reman, G. F., and R. B. Olney, *Chem. Eng. Progr.*, **51**, 141, (1955).
13. Sauter, J., *Z. Ver. deut. Ing.*, **70** (1926); trans. in *Nat'l. Advisory Comm. Aeronaut. Tech. Mem.* 390 (1926).
14. Shepherd, C. B., and C. E. Lapple, *Ind. Eng. Chem.*, **32**, 1246, (1940).
15. Tepe, J. B., and W. K. Woods, *Atomic Energy Rept.* AECD 2864 (Jan. 18, 1943).
16. Van Rossum, J. J., paper presented to the Cyclone Symposium of the Chemical Engineering Section of the Royal Netherlands Institution of Engineers (June 3, 1953).
17. Van Tongeren, Herman, *Mech. Eng.*, **57**, 753 (1935).

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