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SOME OBSERVATIONS ON LIQUID-LIQUID SETTLING

Berkeley, California

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Wilbur F. Sweeney and Charles R. Wilke

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ERRATUM

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Please make the following change on subject report.

Replace pages 59 and 60.

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SOME OBSERVATIONS ON LIQUID-LIQUID SETTLING

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Berkeley, California

January 20, 1964

ABSTRACT

The separation of water from Aroclor 1248 in a gravity settler was investigated with and without induced coalescence. The extent of mixing of the dispersion entering the settling chamber was determined by a light-intensity measuring device. At low flow rates and extents of mixing these two variables appear to control the water concentration of the effluent Aroclor. However, at higher flow rates the water concentration of the effluent Aroclor was affected by many factors, including the flow rate, the extent of mixing, the dispersion zone present between the Aroclor and water phases, and the short circuiting of the Aroclor layer in the settling chamber. The short-circuiting effect was reduced by increasing the Aroclor layer's flow rate. The short circuiting is well illustrated by drawings of the various Aroclor flow patterns produced by varying the operating conditions.

A 1-inch-thick Fiberglas pad mounted in the settling chamber to induce coalescence effectively separated the two phases within the first foot of the settling chamber for all conditions investigated.

I. INTRODUCTION

The separation of mixtures of two immiscible liquids is accomplished throughout industry by the use of gravity settlers. The most extensive use of gravity settlers is found in the oil and chemical industries. To cite a few examples--the desalting and dewatering of crude oil, the treatment with sulfuric acid, and the recovery and purification of rare metals.

Gravity settling is accomplished in horizontal gravity settlers by introducing a mixture of two immiscible liquids into one end of a chamber of sufficient cross sectional area to allow laminar flow through it and of sufficient length to permit phase separation. This allows the two phases to separate as the liquid flows through the chamber. The lighter liquid is withdrawn from the top at the downstream end of the chamber, and the heavier liquid is withdrawn from the bottom at the downstream end of the chamber.

Many variations of this general design are employed in industrial settlers. These variations include vertical and inclined chambers,[1,2,3] baffles to reduce both turbulence and vertical settling distance,[4,5] and different entrance and exit locations.[2,6]

Although gravity settling is one of the oldest means of separating dispersions of immiscible liquids, there is relatively little published literature on the design of gravity settlers. This work is directed toward a better understanding of the effects on the settling process by variation of entrance conditions of the dispersion, and of the flow rate through the settling chamber. It is hoped that this study will aid the devising of a general scale-up method for gravity settlers.

II. THE CREATION OF DISPERSIONS OF TWO IMMISCIBLE LIQUIDS

The dispersing of one immiscible liquid to another may be accomplished by several different methods. The most common methods are injecting one liquid into a stagnant layer of another at high velocities, mixing the liquids with an agitator, and passing the two immiscible liquids through a pipe with turbulent flow.

Ohnesorge found that stable emulsions can be produced by injecting one liquid into a stagnant layer of another at high velocities.[7] An empirical equation was developed by Richardson[8] relating the injection velocity, mean drop diameter, and liquid properties for velocities that created stable emulsions:

$$d = \frac{4000\,\gamma_D^{1.8}}{U\gamma_c^{0.8}} ,$$

where

d = mean drop diameter (cm),

U = average linear injection velocity (cm/sec),

γ_D = kinematic viscosity of dispersed droplets (cm^2/sec), and

γ_c = kinematic viscosity of continuous phase (cm^2/sec).

Although the velocities used in this work are less than those necessary for the direct application of Richardson's equation, that equation does indicate that increasing the injection velocity decreases the mean drop size, thereby increasing the settling time.

Many investigations have been made of the mixing of two immiscible liquids by agitation. It has been found that increasing the mixing speed decreases drop size,[9-11] and increasing mixing time reduces drop size.[11,12]

An investigation of emulsion formation in turbulent flow through pipes was made by Clay.[13] It was found that the drop size of the dispersion was a function of the Reynolds number. Low Reynolds numbers favor the formation of large drops and increasing the Reynolds number tends to decrease the drop size.

III. FACTORS AFFECTING THE SEPARATION OF TWO IMMISCIBLE LIQUIDS

An excellent description of the primary break of an unstable emulsion in a batch process is given by Meissner and Chertow.[14] Three layers of liquid appear almost immediately after agitation of two immiscible liquids is stopped--a rather clear bottom layer of the heavy liquid, a dispersion of the two liquids coalescing in a middle layer, and another rather clear top layer of the light liquid. When the dispersion disappears and a clearly visible interface between the two layers is present, the primary break is complete. The majority phase, and sometimes the minority phase, usually is still cloudy with tiny droplets of the other phase after the primary break. These tiny droplets will eventually settle out; however, the rate of separation is slower by an order of magnitude than the primary break.

The above description of the primary break is very similar to that of gravity settling. There are the same three layers present at the entrance end of the chamber. If the flow rate through the chamber is slow enough, there is a distinct interface at the exit end of the chamber. For faster flow rates the dispersion may extend the length of the chamber.

The removal of the small droplets still present in two phases after the primary break can often be accomplished by a slight agitation with an excess of the dispersed phase,[14] i.e., a cloud of water droplets in an oil phase may be separated out by a slight agitation in an excess of water.

A. Sedimentation

Many equations have been developed to describe the rate of rise or fall (hereafter referred to as "settling" rate) of isolated drops in a stagnant layer of another immiscible liquid. Although these equations do not apply directly to the gravity settling process because of changes in sphericity and the closeness of drops, they do lend an insight into how the different variables may affect liquid-liquid gravity settling.

Bond and Newton developed a modification of Stokes' law for the terminal settling velocity of a drop in viscous flow,[15]

$$U_{00} = \left(\frac{2 \ \Delta\rho \ gr}{9\mu_c}\right) f \left(\frac{\mu_D}{\mu_c}, \frac{r\sigma_I}{w}\right),$$

where

U_{00} = terminal settling or rising velocity of drop (cm/sec),

μ_c = viscosity of continuous phase (gm/cm sec)

μ_D = viscosity of dispersed phase (gm/cm sec)

r = drop radius (cm),

$\Delta\rho$ = density difference of the two liquids (gm/cm^3),

g = acceleration due to gravity (cm/sec^2),

w = apparent weight of drop (gm), and

σ_I = interfacial tension (dynes/cm).

For small drops, $\frac{r\sigma_I}{w}$ is large and $f\left(\frac{\mu_0}{\mu_c}, \frac{r\sigma_I}{w}\right) \rightarrow 1$,

and Bond and Newton's equation approaches Stokes' law. However, for large drops,

$$f\left(\frac{\mu_D}{\mu_c}, \frac{r\sigma_I}{w}\right) \longrightarrow \left(\frac{1 + \mu_D/\mu_c}{2/3 + \mu_D/\mu_c}\right).$$

These equations show that large density difference, large drop size, and low viscosity of the continuous phase are conditions to seek for efficient separation of the two immiscible liquids.

B. Coalescence

The most important (and in most cases the controlling) factor in the rate of primary break in gravity settlers is the rate of coalescence of the dispersion and not the rate of settling of the dispersed drops to the interface.[16]

Although coalescence of liquids in equilibrium is hindered by increasing the magnitude of several fluid properties (such as viscosity and density)[17,18] coalescence may be promoted by increasing the interfacial tension between the fluids.[5] However, there also are some instances when high interfacial tension may hinder coalescence.[19] When the coalescence is film-thinning-controlled an increase in interfacial tension promotes coalescence, and when the coalescence is film-rupture-controlled an increase in interfacial tension hinders coalescence. High viscosity of the continuous phase reduces the rate of removal of the thin film between drops and hinders coalescence. Emulsifying agents reduce the interfacial tension. Also, the presence of minute dust particles which generally accumulate at the interface of a two-liquid phase system can prevent coalescence. Several investigators have observed that oil and water drops have a finite lifetime at an oil-water interface before they coalesce into the bulk phase.[19-21] A small amount of certain impurities has increased the drop lifetime of a few seconds for pure liquids to minutes or even hours for the contaminated drops.

Coalescence can often be induced by flowing the emulsion through a relatively fine porous, fibrous material. The material should have a large surface-to-volume ratio and be preferentially wetted by the dispersed phase.[22] Although the exact mechanism by which these materials induce coalescence is not clear, it appears to involve the rupture of the film surrounding the dispersed droplets, followed by intimate contact between the dispersed drops.[23]

The flow through the settling chamber is another factor that affects the separation of two immiscible liquids. Hart maintains that, in the design of sedimentation chambers for the separation of oil from refinery waste water, the type of flow through the settling chamber is the most important factor in the separation.[24,25] He found that the flow must be free of turbulence that is powerful enough to overcome the buoyancy of the dispersed drops, and presented an experimental correlation that related the maximum permissible Reynolds number to the density of the oil drops. The allowable Reynolds number varied between 5,000 and 20,000.

IV. DESIGN PROCEDURES

A short period without agitation is usually sufficient for the primary break of an unstable emulsion,[22] and the period can be provided most efficiently by continuous gravity settling. Continuous-flow gravity settling is completed by allowing the dispersion to flow through a vessel of sufficient cross section to allow laminar flow and long enough to yield sufficient residence time to allow separation of the two phases.

There have been three different approaches to the designing of gravity settlers, all of which have been successfully used in scaling up pilot plant models to industrial use.[16,26,27] These three methods are by residence time, by overflow velocity, and by dispersion thickness.

The residence-time method is perhaps the most common. The residence time necessary for separation may be determined from pilot plant equipment or merely from past experience. The petroleum industry generally employs residence times of 30 to 60 minutes.[22] However, different dispersions require different residence times to complete the primary break. Settlers used in solvent extraction of irradiated fuels were satisfactorily scaled up from pilot plant models based on geometrical similarity and residence time.[26]

The design of gravity settlers by the overflow velocity method is based on the assumption that the rate of primary phase separation is controlled by the rate of rising and settling of the dispersed drops to the interface.

The overflow velocity can best be explained by considering the settler shown in Fig. 1. Assume that there are no short circuiting of flow, no eddies and no turbulence.

A dispersion of liquid B in liquid A flows into the settler. Liquid B, being less dense than liquid A, forms the top layer. The drops of liquid B dispersed in liquid A move toward the interface at a velocity equal to the terminal velocity, U_{00}, which can be approximated by Stokes' law. These drops also have a horizontal velocity,

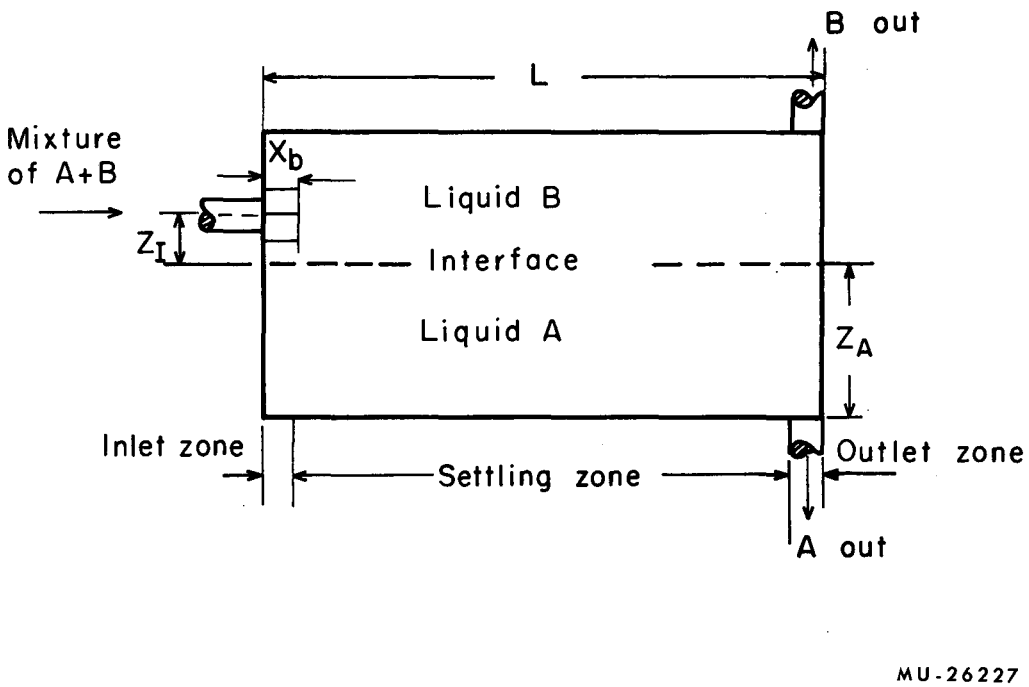

Fig. 1. Ideal settling-rate-controlled gravity settler.

U_A, which can be assumed to be equal to the no-mixing velocity of liquid A. The maximum vertical distance these drops of liquid B must travel to reach the interface is Z_A. On the basis of these assumptions, any drop of liquid B would separate from liquid A if $U_{OO} \geq U_A Z_A/L$; $U_A Z_A/L$ is known as the overflow velocity, V_O. This factor was cited as the chief criterion in the design of settling tanks for separation of suspensions of solid particles from waste water.[28]

The dispersion-thickness method is based on the assumption that the rate of primary phase separations is controlled by the rate of coalescence of the dispersed phase, rather than by the rate of settling, as was assumed in the overflow velocity method.

Figure 2 shows a gravity settler for coalescence-controlled settling.

The design of settlers by this method is based on constant flow rate of the dispersion per unit area of interface between the two layers yielding a constant dispersion-zone thickness. Ryon and fellow workers successfully applied this method of scale-up to an emulsion of aqueous uranium solutions and kerosene-alkyl-phosphate solvents.[16] The dispersion-zone thickness at a given flow rate was independent of settler size. A total liquid depth of 30 inches was selected and the interface was controlled near the midpoint of the settler. A maximum dispersion-zone thickness was arbitrarily selected. The liquid flow rate necessary for this dispersion thickness was determined. The design flow rate was then chosen to be half the maximum flow rate to allow a safety factor of two in the design. A 1000-fold scale-up of throughput capacity was confirmed for this method of design.

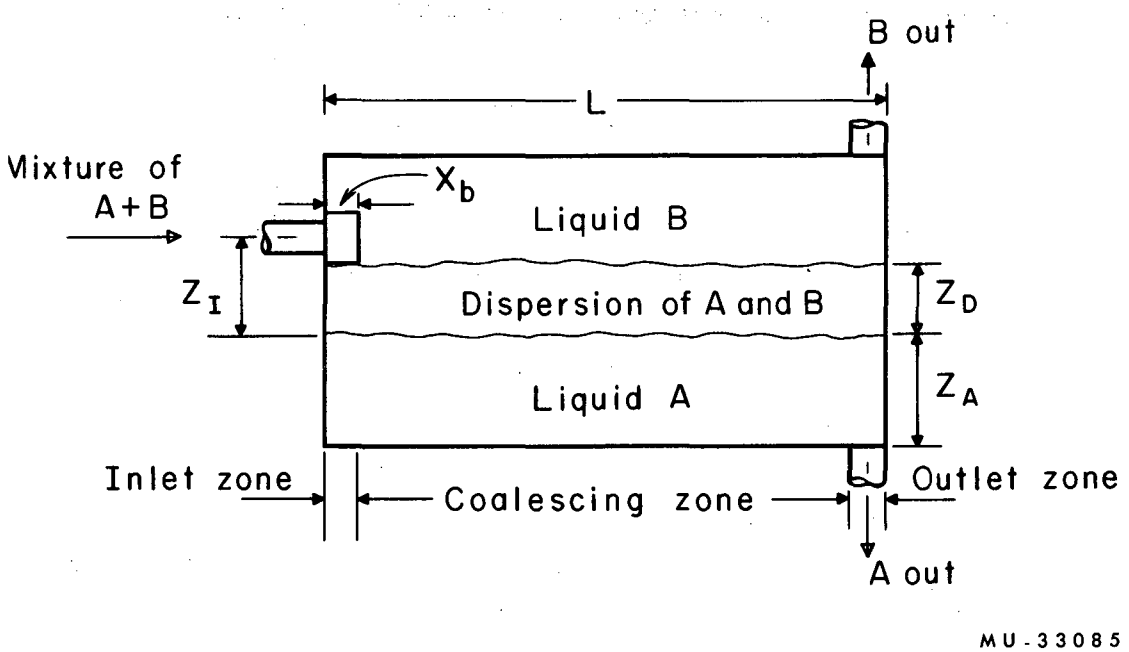

Fig. 2. Ideal coalescence-rate-controlled gravity settler.

V. EQUIPMENT

The equipment used in this work is shown in Fig. 3 through 8. The four main sections consisted of the Aroclor system, the water system, the mixing system, and the settling system.

A. Aroclor System

The Aroclor was stored in a 55-gallon stainless steel drum equipped with a sight glass and thermometer. The Aroclor was heated by three 3750-watt immersion heaters controlled by a rheostat, and a 1/4-hp mixer was used to agitate it.

The Aroclor was pumped with a 5-gpm gear pump through a 3/4-inch copper tubing to the mixing section. A needle valve on a bypass line was used to control the Aroclor flow rate. The fine adjustment of the flow rate was accomplished with a needle valve located upstream from a rotameter. Rust and dirt particles were removed from the liquid by a 25-micron Cuno filter located downstream from the pump.

B. Water System

The water was stored in another 55-gallon stainless steel drum equipped with a sight glass and thermometer. It was agitated by a 1/8-hp mixer.

The water was pumped to the mixing section by a 5-gpm centrifugal pump through 3/8-inch copper tubing. Control of the water flow rate was maintained by a needle valve located just upstream from the water rotameter. A 10-micron Cuno filter was located upstream from the rotameter to remove the dirt and rust particles.

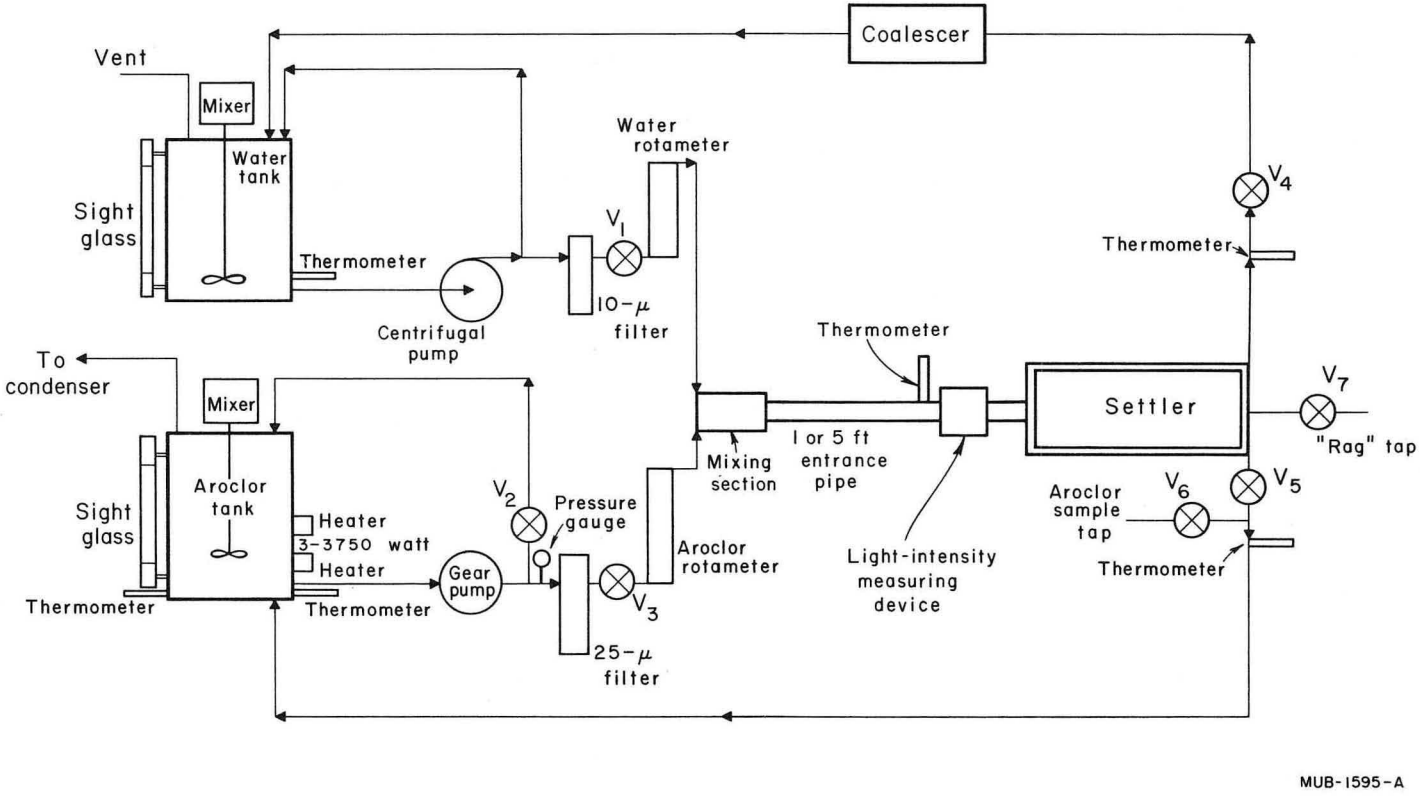

Fig. 3. Schematic diagram of settler apparatus.

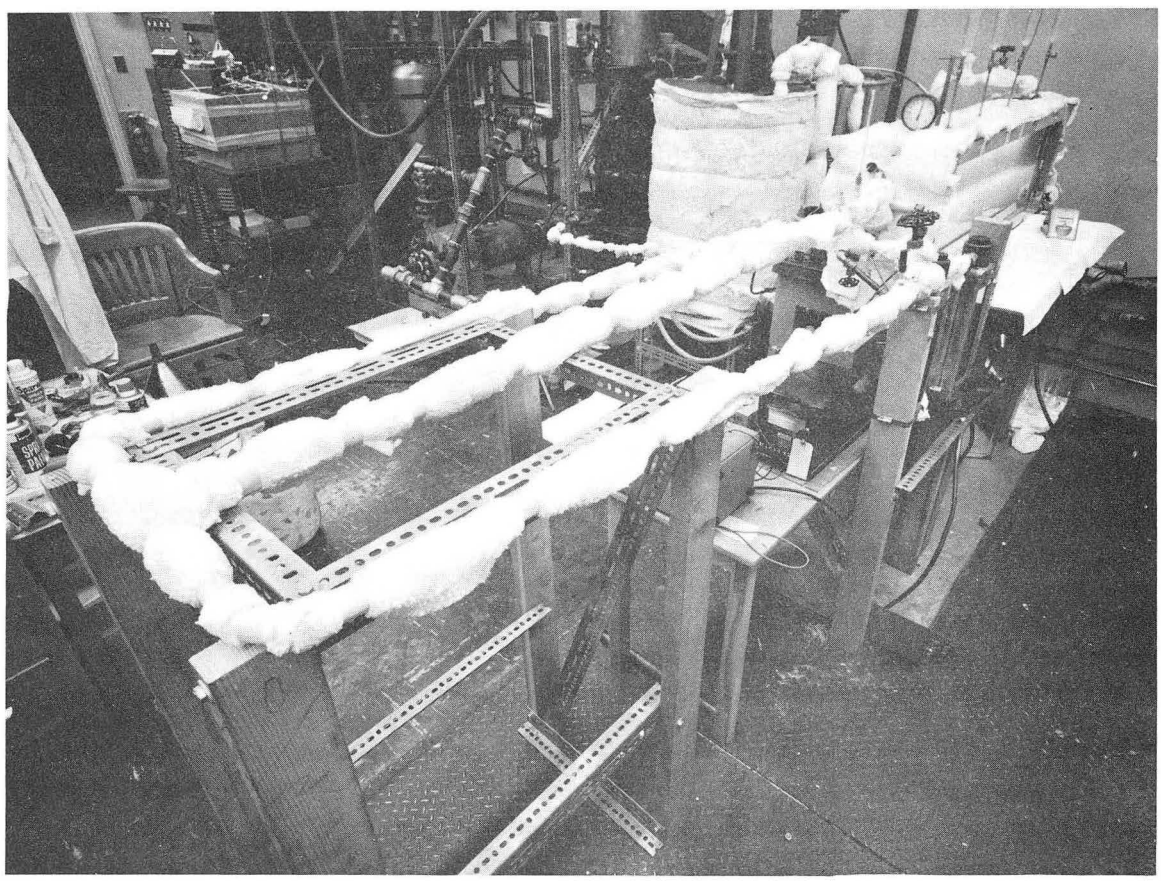

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Fig. 4. Overall view of the experimental apparatus.

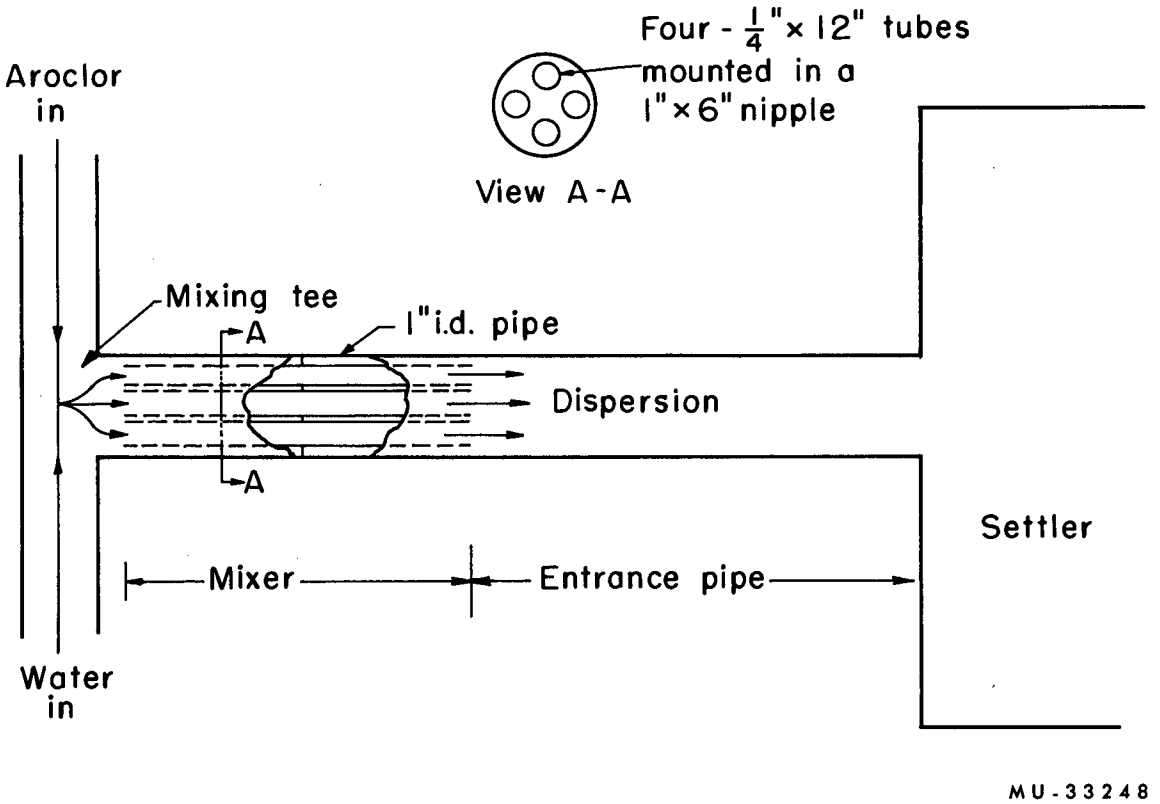


Fig. 5. The mixing section.

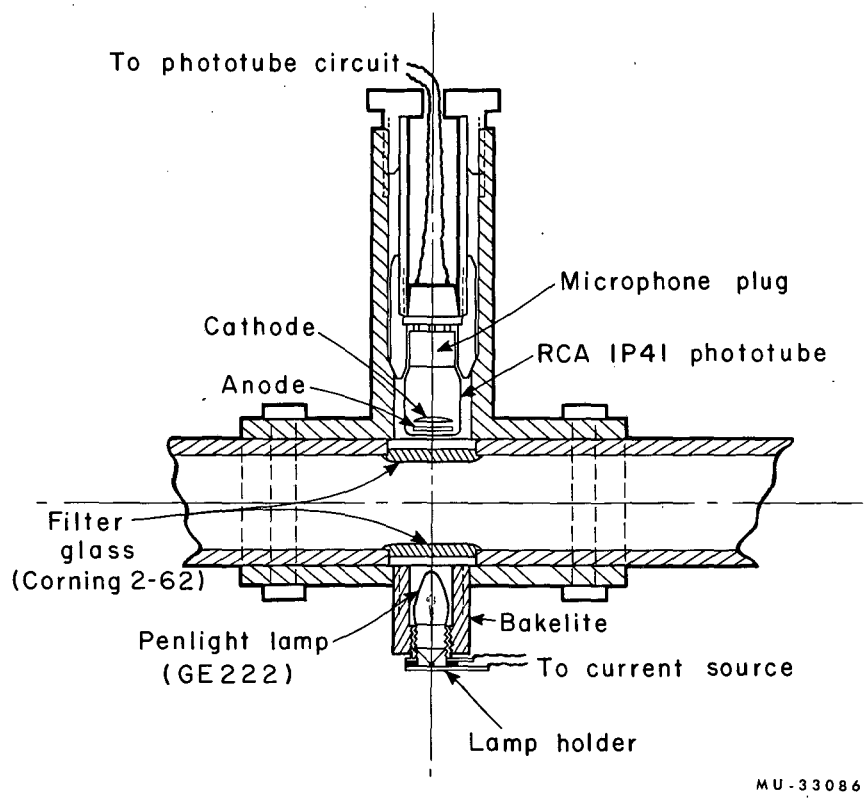


Fig. 6. Device for measuring light intensity.

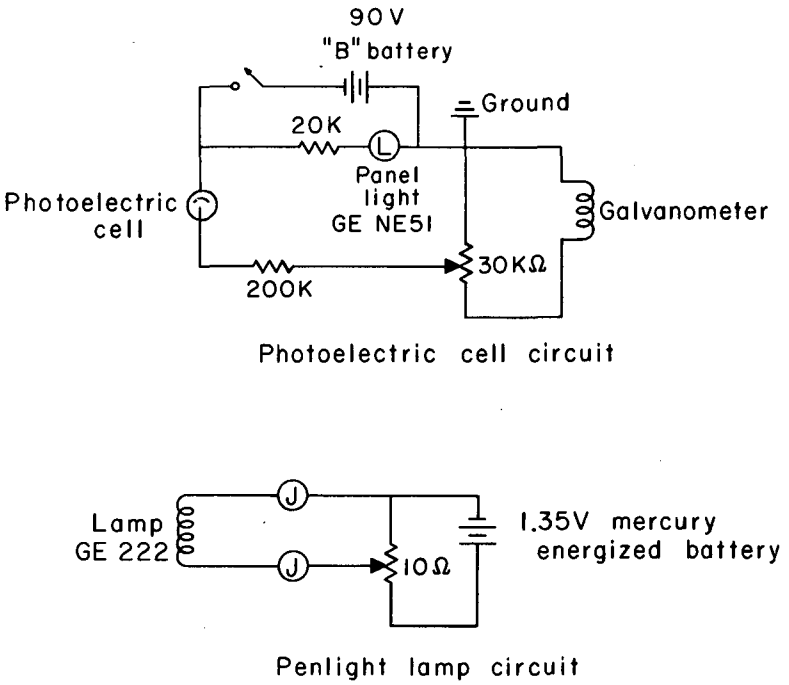


Fig. 7. Electrical circuits for light intensity measuring device.

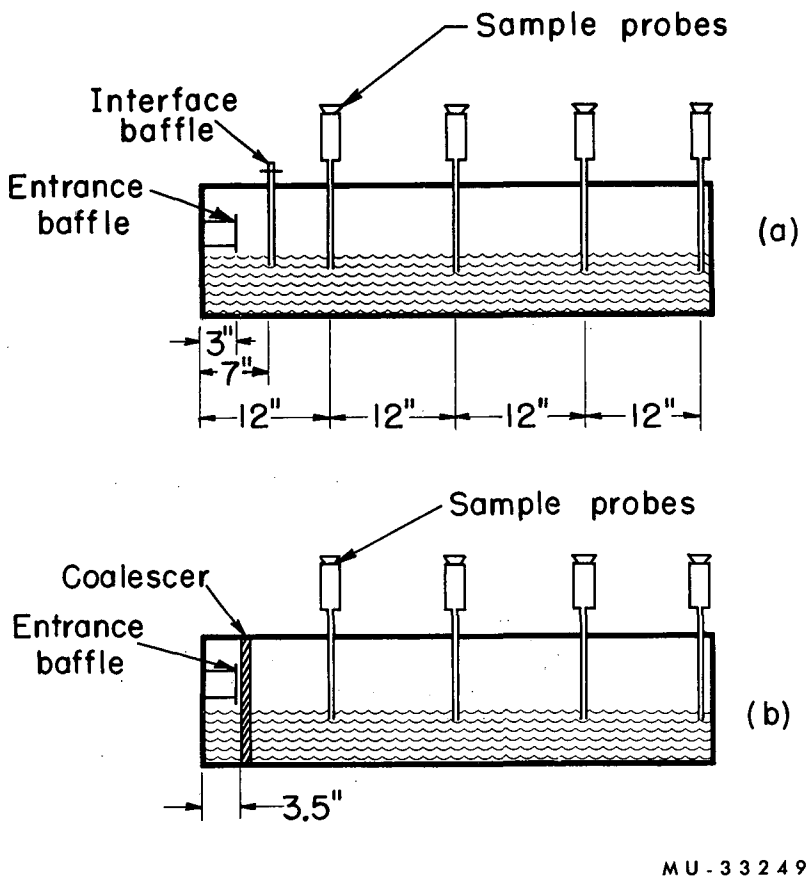


Fig. 8. Settling chamber illustrating baffle and probe locations (top) and coalescer location (bottom).

C. Mixing Section

The Aroclor and water streams met at a 3/4-in. i.d. mixing tee. They passed through a mixer which consisted of four copper tubes, 1/4-in. i.d. by 12-in. long, mounted in a 1 × 6-in. brass nipple. Metal plugs were used to block off the tubes to vary the Reynolds number of the mixing pipe for a given flow rate. The mixing was completed with the injection of the liquid directly into the settler or into a 1-in. i.d. entrance pipe leading into the settler. Two different lengths of pipe were employed to determine the effects of the pipe on drop size.

When the entrance pipe was employed, a 1 × 6-in. nipple with a light intensity measuring device mounted on it was located at the entrance of the settler to measure the relative interfacial area present in the entering dispersion. This measured the extent of mixing for the mixing tee, for the mixing pipe, and for the injection of the liquids into the entrance pipe.

Figures 6 and 7 illustrate the light intensity measuring assembly and electrical circuits. The lower part of the device contained a small penlight flashlight bulb (GE 222) rated at 2 volts, with a built-in lens that gave nearly parallel light. The lamp was operated with an applied potential of 1.00 V. At this potential, the lamp current remained constant and the lamp issued more light in the ultraviolet wavelength range than it would have at 2 volts. The voltage source was a 1.35-V mercury dry cell(E42).

The upper part of the device contained a gas-filled phototube, RCA 1P-41, operated at an applied voltage of 90 V. The voltage source for the phototube was a 90-V type "B" radio battery.

Two filter glasses (Corning 2-62) were mounted in the 1 × 6-in. nipple to pass the light beam through the liquid to the phototube. They also narrowed the wavelength range of the beam to the peak sensitivity of the tube near 8000 Å.

The current output from the phototube was measured with a G. E. galvanometer, Type GR14, with a sensitivity of 0.0017 μa/mm. A Helipot potentiometer with a maximum resistance equal to the critical damping resistance of the galvanometer was placed in the circuit and adjusted to obtain a full deflection of the galvanometer for the measurements. Stray-current problems were avoided by mounting the entire phototube circuit assembly on a metal plate to which the circuit was grounded.

D. Settling System

The settler was a 4-ft-long aluminum chamber with a rectangular cross section, 12 in. high and 7 in. wide. A 0.5 in.-thick Pyrex glass window extended the entire length of one side of the settler to allow observation of the settling process. To aid in the observation of the settling process a 42-in. 30-W fluorescent light, installed inside a 30-mm o.d. Pyrex tube, was mounted across the upper rear of the settling chamber.

A baffle 4 in. high and 2 in. wide was mounted 3 in. in front of the inlet to reduce short circuiting of the flow. The baffle was attached by two 3/8-in. tie rods threaded into the inside of the front plate on either side of the inlet nipple.

During some of the runs another baffle 1 in. high and 7 in. wide was located at the interface 7 in. downstream from the entrance. This baffle was mounted on a 0.5-in. shaft with free vertical motion so that it could be removed from the interface.

Constant temperature in the settler was maintained by four 384-W 4-ft.-long heating tapes wrapped uniformly around it.

Four concentration probes were located at 1-ft. intervals along the settler top. These probes were 3/8 × 16-in. stainless steel tubes with stainless steel hypodermic syringe tips soldered on one end so that samples could be withdrawn with a 50-ml syringe. Each probe had free vertical motion, allowing samples to be withdrawn at any height in its particular cross section.

The water was withdrawn from a 3/4-in. outlet on the top downstream end of the settler. It flowed through a coalescer to remove any small Aroclor droplets and then back into the storage tank.

The coalescing chamber was 1 ft. long, 1 ft. high, and 0.5 ft. wide. The water entered the coalescer through a 0.5-in. tap located on the top upstream end of the chamber. It flowed through a Fiberglass packing and exited through another 0.5-in. tap 1 in. from the top of the downstream end of the chamber. The coalescer was periodically drained to remove the Aroclor that collected in the bottom of the chamber.

Owens-Corning Fiberglas, No. 600 fiber was used as the packing. The Fiberglas comes in 25 × 12 × 0.5-in. mats, with the fibers arranged in a loose jackstraw pattern and held together by a water-soluble binder. The Fiberglas mats were cut with a scissors into pieces 6 × 6 in. which were stacked in the chamber between two 10-mesh stainless steel screens, mounted at distances of 3 and 9 in. down the chamber, until the cross section was filled. The Fiberglas was not compressed, and maintained its original density of 4.70 lb/ft^3. The chamber was flushed several times with hot water to dissolve and remove the soluble binder.

A 1.25-in. pipe was welded across the bottom of the exit plate for the removal of Aroclor from the main settling chamber. This large outlet reduced the disturbance at the Aroclor outlet. The Aroclor was returned to the top of the storage tank to facilitate rapid evaporation of the entrained water.

A tap was located at the downstream end of the settling chamber for periodical removal of "rag," a scum of dust particles and other impurities that collect at the interface. This rag forms even when the entering liquids appear to be clean.

The position of the interface in the settler was controlled by means of needle valves located on the Aroclor and water outlet lines.

Induced coalescence was investigated by inserting in the settling chamber a coalescer made from Owens-Corning Fiberglas basic fiber No. 28C. A weighed quantity of the Fiberglas was compressed to a density of 13.0 lb/ft^3 and a thickness of 1.0 in. between two 10-mesh stainless steel screens. Care was taken to obtain as uniform a thickness and density as possible. The Fiberglas was allowed to overlap the edges of the screen slightly, to insure a tight fit when the coalescer was inserted in the chamber. The coalescer was placed 3.5 in. from the settler entrance and held in place by set screws. Pressure taps were located in both sides of the coalescer. A red fluid manometer (specific gravity = 2.95) was attached to the taps to obtain the pressure drop through the coalescer.

The Aroclor storage tank, mixing section, settling section, and all connecting pipes were wrapped in 0.5-in. Fiberglas insulation.

VI. OPERATING PROCEDURE

The agitator on the Aroclor storage drum was turned on and the Aroclor was heated to about 190° F. The Aroclor pump was started and Aroclor was allowed to flow into the settling chamber. The water and Aroclor outlet valves, V_4 and V_5 were opened. The heaters in the Aroclor holding tank were adjusted so that the Aroclor entering the settler was maintained at exactly 190° F. The light intensity through the pure Aroclor was then measured.

The light source switch was turned on and its voltage was adjusted to 1.00 V by use of a Hewlett-Packard vacuum tube voltmeter. The galvanometer was turned on and adjusted to give a reading of zero current. The phototube switch was then turned on and the potentiometer was adjusted to 100% deflection. This was noted as the output current for the pure continuous phase.

Upon completion of the pure Aroclor light-intensity measurement the phototube switch was turned off and the Aroclor pump was stopped. The remaining Aroclor in the storage tank was heated to 220° F. The Aroclor pump was then restarted.

When the settler was half full the Aroclor pump was stopped and the water pump was started. When the settler was full, the Aroclor pump was restarted. The Aroclor and water flow rates were then adjusted to their desired rates by valves V_1 and V_3, respectively. A 15.0 weight % water mixture was used in this investigation. The outlet valves, V_4 and V_5, were adjusted until the 6-in. interface was maintained.

The water temperature was allowed to rise by direct-contact heat transfer with the Aroclor until the temperature of the incoming stream, water outlet stream, and Aroclor outlet stream were all constant and within the range of 190 ± 3° F. Constant temperatures were maintained by adjusting the heaters in the Aroclor storage tank and by use of the heating tapes on the surface of the settling chamber.

Experimental data were taken approximately 30 minutes after the system reached steady state. Light-intensity measurements of the entering dispersion were made by obtaining a steady reading of the galvanometer's deflection. This was the phototube output current for the dispersion. Care was taken to insure that the inlet temperature was exactly 190° F during this time. The relative interfacial area was determined by the procedure described in Appendix A.

The height of the interface and the depth and length of the dispersion were then recorded. Also the inlet stream, water outlet and Aroclor outlet temperatures were noted.

Samples were then taken from the Aroclor layer at depths of 2, 4, and 5 in. below the interface (in initial runs samples were withdrawn at 6, rather than 5 in. below the interface) with each of the four concentration probes. An exit sample was obtained from valve V_6. These samples were later analyzed for water content by the dead-stop end-point method described in Appendix B.

After the concentration samples were withdrawn, the residence time of the Aroclor layer was measured. A 25-ml pulse of saturated salt solution was injected into the entering water stream with a syringe, and 100-ml samples of the Aroclor outlet stream were withdrawn every 20 seconds from valve V_6. These samples were later washed with 100 ml of distilled water and the extracted salt was determined with an electrical conductivity cell.

This procedure was repeated for various flow rates and mixing-section Reynolds numbers with various entrance pipe lengths, with and without a coalescer in the settling chamber, and with and without the interface baffle in place.

Samples of the water and Aroclor were withdrawn from the system at various times during the experimental work, and the interfacial tension was determined with a Cenco-Du Nouy interfacial tensiometer (No. 70545).

VII. EXPERIMENTAL DATA

The experimental data and derived results are tabulated in Tables I through VI. Tables I and II contain the data and results for the separation of water from Aroclor for a temperature of 190° F. The experimental data for the settling chamber residence times are listed in Table III. The interfacial tension measurements are recorded in Table IV. Induced coalescence results are shown in Tables V and VI. These results are discussed in detail in Section IX.

Table I. Experimental data and derived results for the separation of water from Aroclor 1248 by gravity settling.

Run	(ft^3/min)	N_{Re}	Interfacial area	Average exit C_w	Average settler velocity: Aroclor (ft/min)	Average settler velocity: Water (ft/min)	V_i (ft/min)	Water θ_{nm} (min)	Aroclor θ_{nm} (min)	Aroclor θ_{exp} (min)	Depth of dispersion (in.) Distance down settler (ft.) 0	1	2	3	4
1	0.190	1,546	--	--	0.521	0.130	186	30.0	7.66	--	0.31	0.25	--	--	--
1B	0.190	1,546	--	--	0.521	0.130	186	30.7	7.66	--	0.50	--	--	--	--
2B	0.190	1,546	--	--	0.521	0.130	186	30.7	7.66	--	0.50	--	--	--	--
3B	0.283	2,303	--	--	0.779	0.192	277	20.8	5.13	--	1.50	0.19	--	--	--
4B	0.283	2,303	--	--	0.779	0.192	277	20.8	5.13	--	1.50	0.19	--	--	--
5	0.283	2,303	--	--	0.779	0.192	277	20.8	5.13	--	0.88	1.00	0.63	0.00	--
6	0.283	2,303	--	--	0.779	0.192	277	20.8	5.13	--	0.88	1.00	0.50	0.12	--
7	0.283	2,303	--	--	0.779	0.192	277	20.8	5.13	--	0.88	1.00	0.58	0.12	--
8	0.283	6,910	--	--	0.779	0.192	831	20.8	5.13	--	1.20	1.22	1.38	1.25	1.12
8B	0.283	6,910	--	--	0.779	0.192	831	20.8	5.13	--	2.5	1.00	1.00	1.00	1.00
5-foot entrance section employed															
A11	0.283	6,910	14.15K	0.17	0.779	0.192	51.8	20.8	5.13	1.83	0.25	0.25	0.25	0.12	--
A12	0.283	6,910	14.15K	0.19	0.779	0.192	51.8	20.8	5.13	1.83	0.25	0.25	0.25	0.19	--
A12B	0.283	6,910	14.15K	0.195	0.779	0.192	51.8	20.8	5.13	1.83	0.75	0.12	0.12	0.06	--
B11	0.520	12,696	15.67K	0.44	1.43	0.353	95.1	11.3	2.79	1.83	0.38	0.38	0.38	0.38	0.38
B11B	0.520	12,696	15.67K	0.48	1.43	0.353	95.1	11.3	2.79	1.83	1.00	0.19	0.19	0.19	0.19
C11	0.681	16,627	16.39K	0.58	1.87	0.466	124.6	8.57	2.14	1.50	0.80	0.80	0.88	0.88	0.88
C11B	0.681	16,627	16.39K	0.60	1.87	0.466	124.6	8.57	2.14	1.50	2.50	0.80	0.88	0.88	0.88
C12	0.681	16,627	16.39K	0.58	1.87	0.466	124.6	8.57	2.14	1.50	0.19	0.19	0.25	0.38	0.44
C12B	0.681	16,627	16.39K	0.57	1.87	0.466	124.6	8.57	2.14	1.50	2.50	0.19	0.25	0.38	0.44
D11	0.200	4,883	13.70K	0.07	0.549	0.135	36.6	29.5	7.28	--	0.50	0.50	0.44	--	--
D11B	0.200	4,883	13.70K	0.05	0.549	0.135	36.6	29.5	7.28	--	1.25	0.31	0.31	--	--
D12	0.200	4,883	13.70K	0.05	0.549	0.135	36.6	29.5	7.28	--	0.19	0.25	0.05	--	--
D12B	0.200	4,883	13.70K	0.05	0.549	0.135	36.6	29.5	7.28	--	0.32	0.12	0.05	--	--
A21	0.283	3,455	8.67K	0.04	0.779	0.192	51.8	20.8	5.13	1.83	0.19	0.31	0.25	--	--
B21	0.520	6,348	10.11K	0.20	1.43	0.353	95.1	11.3	2.79	1.83	0.38	0.38	0.31	0.31	0.31
C21	0.681	8,314	12.33K	0.39	1.87	0.466	124.6	8.57	2.14	1.50	0.50	0.50	0.50	0.50	0.50
A31	0.283	2,303	7.97K	0.03	0.779	0.192	51.8	20.8	5.13	--	0.19	0.25	0.00	--	--
B31	0.520	4,232	8.53K	0.15	1.43	0.353	95.1	11.3	2.79	1.17	0.44	0.38	0.31	0.25	0.05
C31	0.681	5,542	9.35K	0.35	1.87	0.466	124.6	8.57	2.14	1.50	0.50	0.50	0.50	0.50	0.44
3.66 wt % sodium chloride solution employed															
A22	0.283	3,455	7.45K'	0.14	0.779	0.192	51.8	20.8	5.13	1.83	0.25	0.25	0.19	--	--
B22	0.520	6,348	9.25K'	0.46	1.43	0.353	95.1	11.3	2.79	1.83	0.38	0.38	0.50	0.38	0.25
1-foot entrance section employed															
A23	0.283	3,455	10.11K	0.08	0.779	0.192	51.8	20.8	5.13	1.83	0.19	0.38	0.25	--	--
B23	0.520	6,348	11.63K	0.31	1.43	0.353	95.1	11.3	2.79	1.83	0.44	0.50	0.50	0.44	0.31
C23	0.681	8,314	12.33K	0.40	1.87	0.466	124.6	8.57	2.14	1.50	0.50	0.50	0.50	0.50	0.50

For all runs:

Entering water concentrations, C_o = 15.0 wt %

Aroclor layer depth, Z_A = 6.0 in.

Settler length, L = 4.0 ft.

Temperature = 190° ± 3° F.

Runs ending in "B" were made with the interface baffle in position.

Table II. Water concentration profiles in the Aroclor phase (wt % free water).

Run	Depth below interface (in.)	Length down settler (ft.) 1	2	3	4	Exit	Entrance
1B	2	0.05	0.04	0.05	0.05		
	4	0.04	0.05	0.05	0.05		
	6	0.05	-	0.05	0.05		15.0
2B	2	0.05	0.05	0.05	0.04		
	4	0.05	0.04	0.05	0.04		
	6	0.05	0.05	0.04	0.04		15.0
3B	2	0.06	0.07	0.06	0.07		
	4	-	0.07	0.07	0.07		
	6	0.04	0.07	0.07	0.07		15.0
4B	2	0.06	0.06	0.06	0.05		
	4	0.05	0.05	0.05	0.06		
	6	0.05	0.04	0.05	0.06		15.0
5	2	0.05	0.05	0.05	0.05		
	4	0.04	0.05	0.05	0.05		
	6	0.03	0.04	0.05	0.04		15.0
6	2	0.05	0.06	0.06	0.07		
	4	0.04	0.04	0.06	0.06		
	6	0.04	0.04	0.05	0.05		15.0
7	2	0.06	0.06	0.06	0.05		
	2	0.05	0.06	0.06	0.05		
	4	0.03	0.05	0.05	0.05		
	4	0.03	0.05	-	0.06		
	6	0.04	0.05	0.04	0.05		
	6	0.05	0.04	0.04	0.05		15.0
8	2	0.06	-	0.05	0.05		
	4	0.06	0.07	0.05	0.05		
	6	-	0.05	0.06	0.06		15.0
8B	2	0.04	0.04	0.05	0.04		
	4	0.04	0.05	0.05	0.04		
	6	0.05	0.05	0.04	0.04		15.0

Table II. (Continued)

Run	Depth below interface (in.)	Length down settler (ft.) 1	2	3	4	Exit	Entrance
5-foot entrance section employed							
A11	2	-	1.17	1.27	1.39		
	4	0.13	0.12	0.38	0.56		
	6	0.06	0.04	0.05	0.07	0.17	15.0
A12	2	0.92	1.13	1.28	1.39		
	4	0.12	0.10	0.34	0.61		
	6	0.05	0.06	0.05	0.06	0.19	15.0
A12B	2	0.68	1.00	1.02	1.11	0.20	
	4	0.11	0.12	0.33	0.46	and	
	6	0.05	0.03	0.03	0.06	0.19	15.0
B11	2	2.06	2.47	2.97	3.17		
	4	0.42	0.41	0.48	0.72		
	6	0.05	0.03	0.03	0.04	0.44	15.0
B11B	2	1.94	2.19	2.67	4.03		
	4	0.54	0.49	0.65	0.83		
	6	0.03	0.04	0.03	0.04	0.48	15.0
C11	2	3.80	4.76	4.85	6.20		
	4	0.61	0.72	0.78	1.34		
	5	0.34	0.39	0.42	0.61	0.58	15.0
C11B						0.60	15.0
C12	2	2.03	3.48	3.58	4.20		
	4	0.64	0.74	0.83	1.55		
	5	0.39	0.64	0.46	0.62	0.58	15.0
C12B	2	2.65	3.50	4.39	5.70		
	4	0.63	0.78	0.94	1.51		
	5	0.58	0.69	0.42	0.56	0.57	15.0
D11	2	0.16	0.25	0.20	0.25		
	4	0.03	0.04	0.11	0.21		
	5	0.03	0.03	0.03	0.04	0.07	15.0
D11B						0.05	15.0

Table II. (Continued)

Run	Depth below interface (in.)	Length down settler (ft.) 1	2	3	4	Exit	Entrance
D12	2	0.18	0.25	0.22	0.29		
	4	0.03	0.04	0.16	0.26		
	5	0.03	0.03	0.03	0.07	0.05	15.0
D12B	2	0.19	0.22	0.24	0.24		
	4	0.03	0.03	0.05	0.20		
	5	0.03	0.03	0.03	0.04	0.05	15.0
A21	2	0.07	0.16	0.08	0.04		
	4	0.03	0.04	0.05	0.05		
	5	0.02	0.03	0.05	0.05	0.04	15.0
B21	2	0.96	1.77	1.67	2.20		
	4	0.26	0.05	0.32	0.70		
	5	0.04	0.03	0.03	0.17	0.20	15.0
C21	2	2.48	3.99	4.25	4.64		
	4	0.34	0.49	0.42	0.99		
	5	0.16	0.37	0.17	0.31	0.39	15.0
A31	2	0.03	0.03	0.03	0.03		
	4	0.03	0.03	0.05	0.03		
	5	0.04	0.05	0.03	0.04	0.03	15.0
B31	2	0.56	0.53	0.51	0.58		
	4	0.03	0.03	0.08	0.40		
	5	0.03	0.03	0.03	0.10	0.15	15.0
C31	2	0.44	1.60	2.10	3.07		
	4	0.11	0.21	0.43	1.05		
	5	0.03	0.03	0.03	0.12	0.35	15.0
3.66 wt % salt solution employed							
A22	2	0.30	0.27	0.22	0.25		
	4	0.03	0.10	0.16	0.23		
	5	0.02	0.04	0.08	0.18	0.14	15.0
B22	2	2.33	2.74	2.70	3.07		
	4	0.48	0.52	0.72	1.25		
	5	0.30	0.25	0.32	0.45	0.46	15.0

Table II. (Continued)

Run	Depth below interface (in.)	1	2	3	4	Exit	Entrance
1-ft. entrance section employed							
A23	2	0.13	0.18	0.04	0.21		
	4	0.03	0.03	0.06	0.18		
	5	0.04	0.03	0.03	0.08	0.08	15.0
B23	2	1.35	2.21	2.22	3.44		
	4	0.17	0.39	0.51	0.97		
	5	0.06	0.10	0.12	0.28	0.31	15.0
C23	2	2.53	3.90	4.31	4.75		
	4	0.32	0.45	0.41	1.05		
	5	0.16	0.30	0.15	0.30	0.40	15.0

Table III. Experimental data for residence-time measurements (relative conductivity).

Time (min)	Run number														
	D11	D12	D12B	A11	A11B	B11	B11B	C12	C12B	A21	B21	C21	A31	B31	C31
0.83	3.33	5.78	5.00	4.35	5.00	7.14	12.05	9.43	5.88	3.55	3.71	5.41	5.72	11.90	4.43
1.17	4.35	4.48	4.00	4.17	6.07	9.26	12.35	25.00	10.00	4.17	5.50	20.40	2.94	16.13	13.15
1.50	3.03	10.75	3.78	5.27	6.25	15.6	26.3	28.60	28.60	7.15	5.50	33.33	2.70	11.90	23.25
1.83	4.35	6.10	4.17	10.50	10.00	21.7	24.4	20.85	20.85	8.20	14.50	20.00	5.90	5.27	17.55
2.17	6.25	7.70	4.08	8.00	6.46	20.0	20.0	12.50	19.25	3.85	13.30	12.33	3.23	4.36	9.82
2.50	5.55	6.45	3.23	9.10	3.77	16.65	25.6	11.78	13.33	6.25	12.65	10.30	2.70	8.33	9.10
2.83	2.86	8.33	5.27	8.00	6.67	16.40	18.15	10.55	--	7.45	6.90	9.53	5.26	8.33	8.33
3.17	4.26	3.67	4.77	8.50	8.33	14.70	17.20	9.44	13.33	4.17	8.70	8.33	3.23	4.00	9.10
3.50	2.95	3.13	2.50	5.40	8.57	10.90	13.50	8.00	13.15	3.23	7.95	5.72	4.00	8.33	10.00
3.83	4.77	3.33	5.56	5.00	5.00	11.15	14.90	8.00	10.00	6.67	8.27	5.56	6.07	6.67	7.14
4.17	3.45	8.33	2.63	6.68	2.90	12.35	17.20	7.40	7.70	4.00	7.36	5.72	2.50	4.00	6.67
4.50	6.25	4.76	4.35	3.28	6.67	11.20	11.10	8.00	6.67	2.86	5.00	5.27	2.70	4.00	8.33
4.83	5.00	3.23	2.50	4.00	3.57	6.25	10.60	9.1	5.27	4.55	2.86	5.00	2.45	6.25	5.00
5.13	4.55	3.45	2.78	4.35	4.55	10.75	8.93	10.20	9.52	2.86	2.86	4.55	2.30	3.13	6.43
5.50	5.27	6.25	3.85	5.00	5.42	11.10	7.35	9.30	8.33	2.86	5.88	5.00	2.45	4.17	5.40
5.83	3.85	--	3.21	5.00	5.00	7.35	9.10	8.70	6.26	2.86	3.13	6.27	3.13	4.76	6.67

Table IV. Aroclor-water interfacial tension measurements.

Determination	Run after which measurement was obtained	Phase 1	Phase 2	Interfacial tension (dynes/cm)
101	--	Fresh tap water	Fresh Aroclor 1248	45.3
102	--	Distilled water	Fresh Aroclor 1248	46.6
103	B22	Distilled water	Settler Aroclor 1248	45.6
104	B22	Settler tap water	Fresh Aroclor 1248	39.6
105	B22	Settler tap water	Settler Aroclor 1248	39.0
106	D12B	Settler tap water	Settler Aroclor 1248	38.6
107	D11	Settler tap water with a trace of sodium chloride added	Settler Aroclor 1248	35.9
108	C23	Settler tap water	Settler Aroclor 1248	38.7
109	F21	3.66 wt % sodium chloride settler tap water	Settler Aroclor 1248	32.3

Table V. Experimental data and derived results for the separation of water from Aroclor 1248 by induced coalescence.

Run	Q (ft^3/min)	N_{Re}	Interfacial area	Average exit C_w	Average settler velocity: Aroclor (ft/min)	Average settler velocity: Water (ft/min)	V_i (ft/min)	θ_{nm} (min)	θ_{nm} (min)	V_s (ft/min)	ΔP (in. water)	Depth of dispersion (in.) Distance down settler (ft.): 0	1	2	3	4
C13	0.681	16,627	16.39K	0.023	1.87	0.466	124.6	2.14	1.50	1.59	43.0	--	0.19	0.19	0	--
C24	0.681	8,314	12.33K	0.024	1.87	0.466	124.6	2.14	1.50	1.59	43.0	--	0.12	0.06	0	--
E21	0.221	2,700	8.37K	0.015	0.61	0.155	40.4	26.70	6.57	0.59	15.5	--	0.03	--	--	--
F21	0.442	5,400	9.80K	0.015	1.22	0.310	80.8	13.35	3.29	1.13	33.0	--	0.03	0.03	--	--

For all runs:

Entering water concentrations, C_o = 15.0 wt%

Aroclor layer depth, Z_A = 6.0 in.

Settler length, L = 4.0 ft.

Temperature = $190^\circ \pm 3^\circ$ F

Table VI. Water concentration profiles in the Aroclor phase for separation by induced coalescence (wt % free water).

Run	Depth below Interface (in.)	Length down settler (ft.) 1	2	3	4	Exit	Entrance
C13	2	0.019	0.022	0.018	0.020	0.023	
	4	0.020	--	--	0.017	0.019	
	5	0.029	--	--	0.015	0.023	15.0
C24	2	0.019	0.020	0.018	0.019	0.025	
	4	0.019	--	--	0.013	0.021	
	5	0.022	--	--	0.024	0.021	15.0
E21	2	0.018	0.016	0.014	0.018	0.016	
	4	--	--	--	--	0.013	
	5	--	--	--	--	0.015	15.0
F21	2	0.014	0.015	0.014	0.012	0.018	
	4	0.017	0.014	0.017	0.012	0.012	
	5	0.017	0.013	0.010	0.010	0.015	15.0

VIII. OBSERVATIONS

A. Dispersion Zone

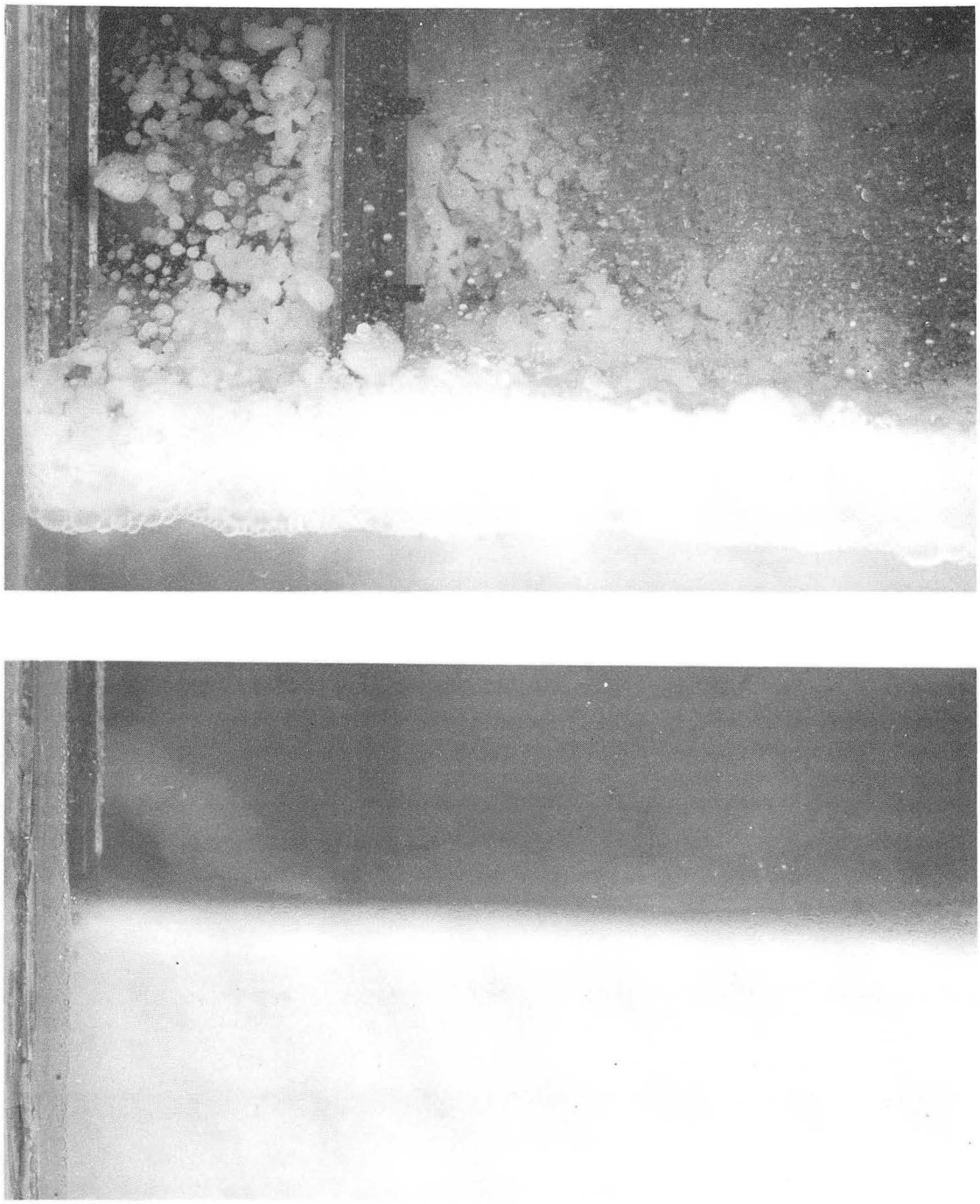
The dispersion zone of primary break described by Meissner and Chertow was present under all operating conditions. However, the behavior of the dispersion was not the same for all operating conditions.

The dispersion that was formed with the mixing section discharging directly into the settling chamber was composed of a complicated mixture of Aroclor and water drops, with a film of water around them. The last drops predominated in the mixture. These Aroclor drops with a water film around them apparently were formed in the inlet zone (Fig. 1). The Aroclor enters the settler in the water layer and impinges against the entrance baffle plate, which breaks the Aroclor into drops and the water film then forms around them.

As shown in the results, (Table I, Runs 1 through 8B) when the interface baffle was inserted, the length of the dispersion zone was shortened.

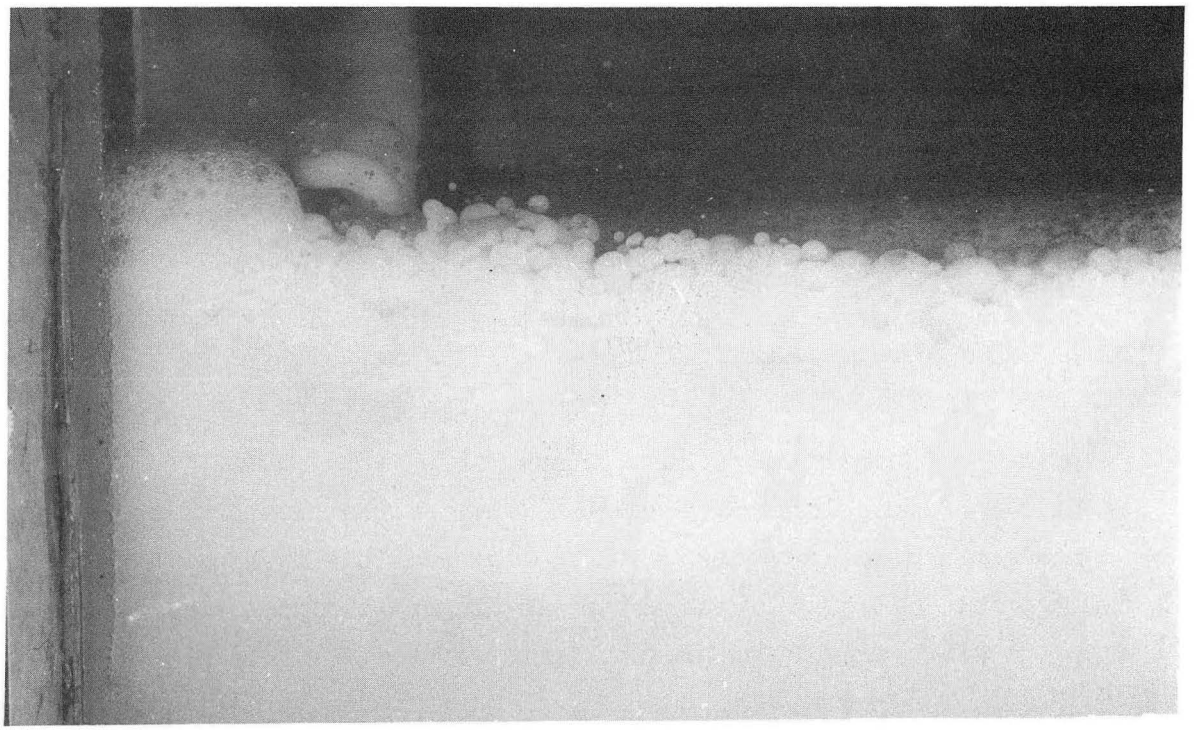
The addition of the entrance pipe to the system caused considerable change in the appearance and behavior of the dispersion (Fig. 9).

At low entrance flow rates the entering stream did not contact the baffle and only a few Aroclor drops with a water film around them were noted. These were perhaps formed when the incoming stream struck the baffle plate tie rod as the stream dropped toward the Aroclor layer. The majority of this dispersion consisted of small water drops that rose out of the Aroclor phase and existed at the interface for a finite lifetime. The appearance of the dispersion did not change as the flow rates and extent of mixing were increased, except for increase in volume, until the entrance velocity exceeded about 100 ft/min. At this speed the entering stream was again impinging against the baffle plate and a complicated mixture was again noted (Fig. 10). This mixture contained the same types of drops mentioned in the first case, except that the Aroclor drops and the Aroclor drops surrounded by a water film contained several small drops of water in them. These small water drops rose to the top



ZN-4192

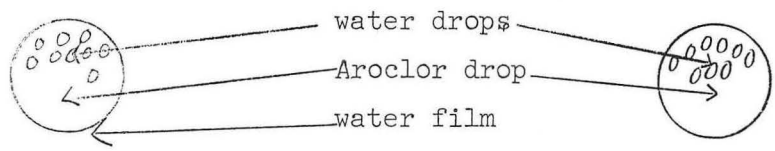
Fig. 9. Dispersion entering the settling chamber by direct injection from the mixing pipe (top) and through the entrance pipe (bottom), $N_{Re} = 4470$.



ZN-4194

Fig. 10. Dispersion entering the settling chamber through the entrance pipe at high flow rate, $Q = 0.681\ ft^3/min$, and high extent of mixing, $A = 16.39$ K.

of the Aroclor drop as illustrated below.



These small water drops did not coalesce into one large drop during the lifetime of the Aroclor drop. When the Aroclor drop collapsed the water drops settled into the rest of the dispersion as isolated drops. The Aroclor drops coalesced completely within the first foot of the settling chamber. These drops and the water drops with a film of Aroclor around them were in a minority. About 95 volume % of the dispersion consisted of water in the form of small drops, which rose out of the Aroclor phase. That the water drops have longer lifetimes the Aroclor drops before coalescence occurs agrees with the observations of Lang.[19] However, the interface lifetime of the drops was longer than predicted by Lang, owing probably to impurities present in the system.

The movement of the dispersion zone became very complicated at the highest flow rate and extent of mixing. A positive (downstream) velocity was noted for the first 3 feet of the settling chamber and a negative (upstream) velocity was noted for the last foot of the chamber.

The dispersion-zone contour varied considerably over the range of conditions investigated. For low flow rates and very low extents of mixing, there was a maximum for the dispersion-zone depth. For low flow rates and slightly higher extents of mixing, the dispersion-zone depth decreased down the settling chamber. At intermediate flow rates and extents of mixing, the dispersion zone was of approximately constant depth the entire length of the settling chamber. Finally, the dispersion thickness increased down the settling chamber for the highest flow rate and extent of mixing investigated. The depth of the dispersion zone at 1-ft intervals down the settling chamber may be found in Table I. Also, the above-mentioned observed dispersion-zone contours are illustrated in Fig. 11.

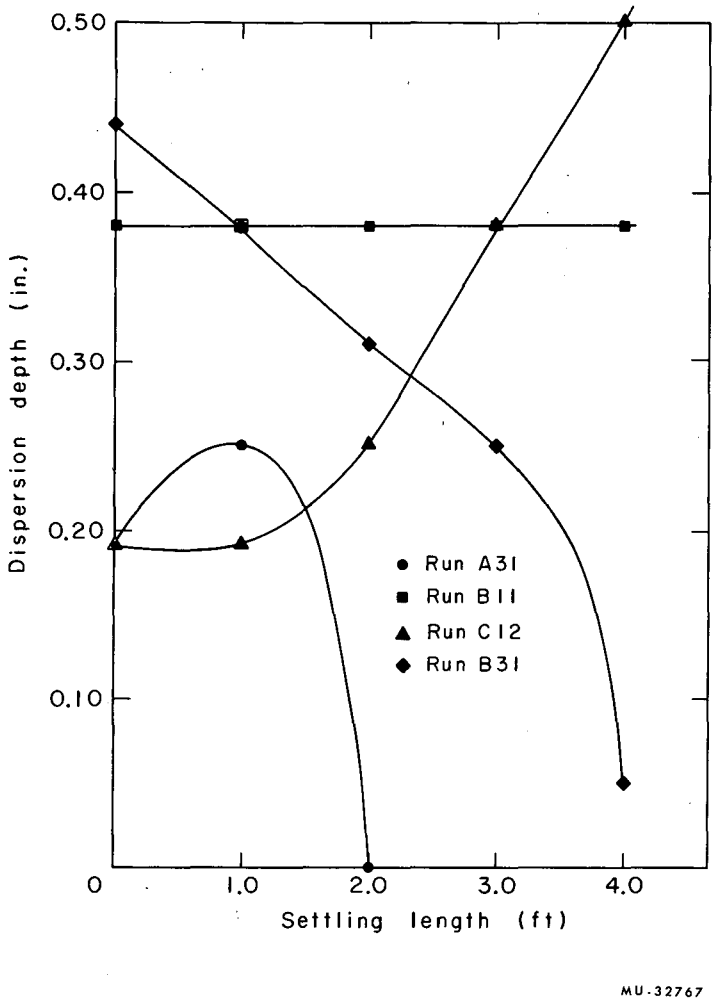


Fig. 11. Various dispersion-zone contours.

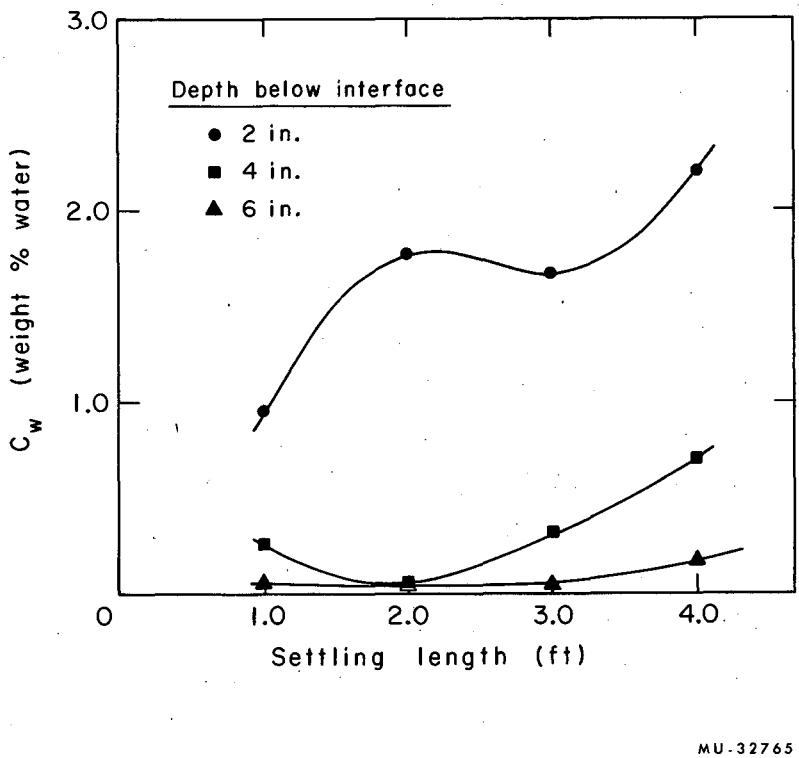
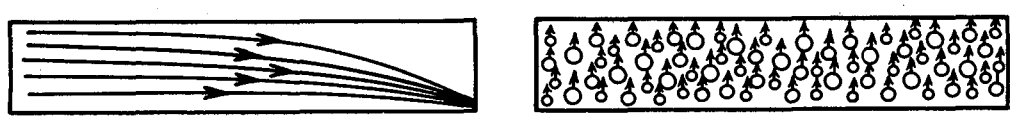


Fig. 12. Concentration profiles at various depths below interface, Run B21.

To explain why short circuiting occurred, consider two separate types of idealized flow in the settling chamber--first, the laminar flow of Aroclor with no water in it through the settler (case 1); and second, the rising of water drops out of the Aroclor layer with a dispersion zone at the top of the layer, with no Aroclor flow through the settler (case 2).

In Case 1, the Aroclor stream lines are all directed toward the Aroclor exit. Therefore the dynamic force of the flow is in that direction, which is for the main part at an angle slightly below the horizontal.



(1) (2)

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In case 2, the motion of the water drops is directed toward the dispersion zone and the dynamic force is directed vertically. The more concentrated the water is and the greater the drop velocities, the greater the vertical dynamic force. Assuming that the water drops enter the settling chamber evenly distributed, the water concentration increases near the interface, as the larger drops move more rapidly than the small drops. The dispersion zone consists mainly of water drops that have "settled" out but have not yet coalesced. These drops act as a barrier to the settling out of other water drops, causing hindered settling to occur. The settling velocity is reduced, but the water concentration is increased by this action.

The addition of flow to the Aroclor layer (combining case 1 and case 2) causes the water drops to be moved down the settler. Since the drops are hindered from settling out by the dispersion zone, the concentration increases down the settler at a given depth.

The velocity of the Aroclor through the settling chamber in this study was very slow (U_A = 0.55 to 1.87 ft/min and N_{Re} = 135 to 463). Therefore, the vertical component of the Aroclor force vector for case I was very small and, as the water concentration down the settling chamber increased, the vertical force vector of case 2 became larger and the resulting vertical force vector was toward the interface, rather than toward the bottom of the settling chamber. This caused the Aroclor flow to move toward the interface, as was observed and is shown on Page 40.

Increasing the Aroclor flow rate increases the force vector magnitude for case I, necessitating a larger force vector from case 2 before the flow is turned toward the interface. This explains why the point of upturn moves toward the exit of the settling chamber as the Aroclor flow rate is increased as was noted in the observations.

The circular motion at the downstream end of the settling chamber was caused as follows: Water concentration profiles at this cross section show that considerable water was entrained by the Aroclor as it flowed down the end of the chamber. The fast motion of the dispersion zone noted and discussed by Epstein[29] apparently caused a greater mass flow rate of Aroclor down the settling chamber along the interface than the actual flow rate through the chamber. This induced a circular motion at the downstream end of the chamber as the excess Aroclor tended to move back upstream along the bottom of the chamber. As the entrained water drops settled out their upward motion reinforced the circular motion. This circulation also explains the high concentrations often obtained at the 3-ft settling length of the chamber.

The effect of the drop size on the Aroclor phase flow pattern is best illustrated by the different flow patterns obtained at a constant Aroclor layer flow rate of 1.870 ft/min in Runs C12, C21, and C31. Run C31 was made at relatively low entering interfacial area (large drops), and the point of upturn of the Aroclor flow was between 2 and 3 ft down the settler. However, with high entering interfacial area (small drops) in Runs C12 and C21 the point of upturn was near the end of the settling chamber and the direction of circulation at the end of the chamber was reversed.

C. Residence Time

The residence times of the Aroclor layer were obtained by noting the maximum relative conductivity of the extracted salt samples. These measures further illustrated the effects of short circuiting on the behavior of the Aroclor flow through the settling chamber.

At low flow rates and low extents of mixing, residence times could not be obtained because some of the entering fluid moved quickly along the Aroclor-water interface and down the end of the chamber. This movement carried some of the salt pulse very quickly through the settling chamber and some salt was circulated at the downstream end. The remainder of the salt pulse was dispersed in the Aroclor that penetrated deep into the Aroclor layer in the entrance zone and moved more slowly through the chamber. The result was a randomly fluctuating conductivity curve, as is illustrated in Table III and Fig. 13.

The actual residence times obtained were found to be, in most cases, between 1.50 and 1.83 minutes, independent of theoretical holding times and the interface baffle plate. However, the conductivity curve of the individual runs did vary considerably in its fluctuations. Figure 14 compares the curves of Runs C11 and C21 and Run C31. Runs C11 and C21 exhibited smooth curves of a definite bulk residence time with a small peak due to the counterclockwise circulation effect noted. Run C31 exhibited a similar bulk residence curve, but the remainder of the curve shows a very strong fluctuation from the clockwise circulation noted. This curve indicates that, although the observations suggested that a clockwise circulation was occurring, the majority of the exiting Aroclor was flowing below this circulation and the fluctuations were obtained from what Aroclor was actually exiting from the circulation down at the end of the chamber.

Figure 15 illustrates the results obtained for the runs between the two extremes mentioned above. There are considerable random fluctuations from the circulation at the downstream end of the chamber; however, a point of maximum conductivity is obtainable from the curve.

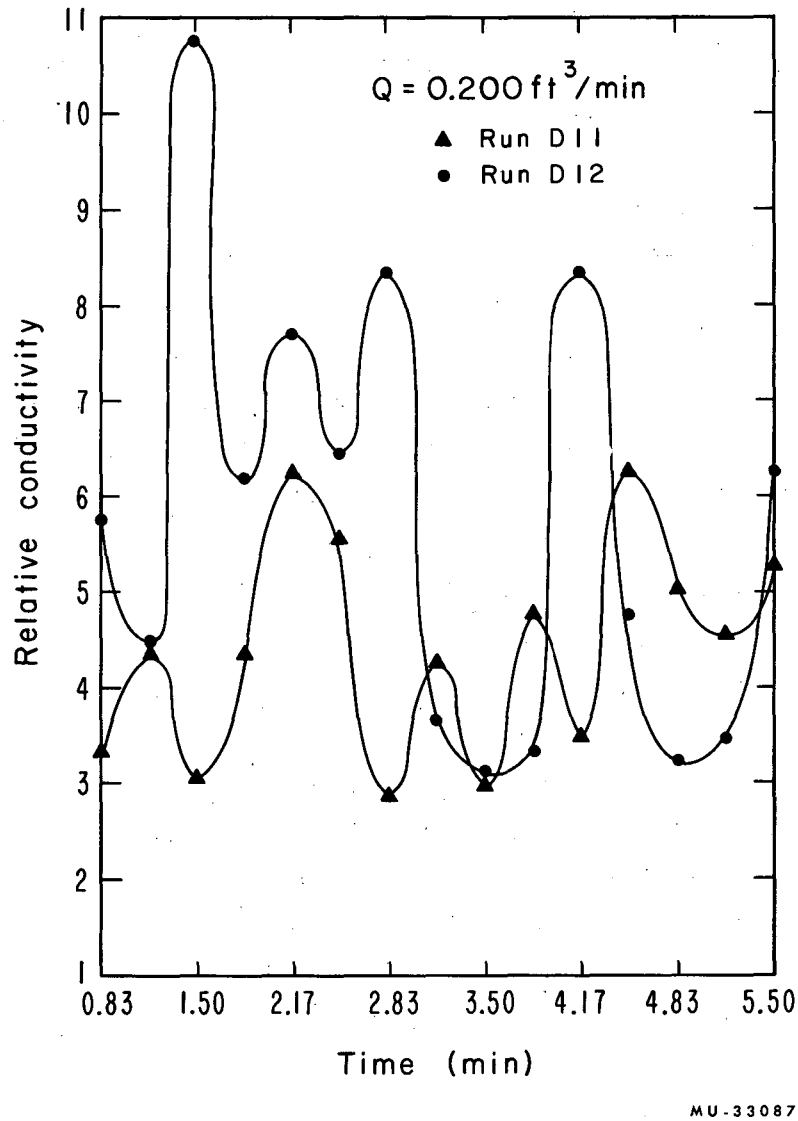


Fig. 13. Conductivity response of injected salt pulse in the Aroclor phase at low flow rate.

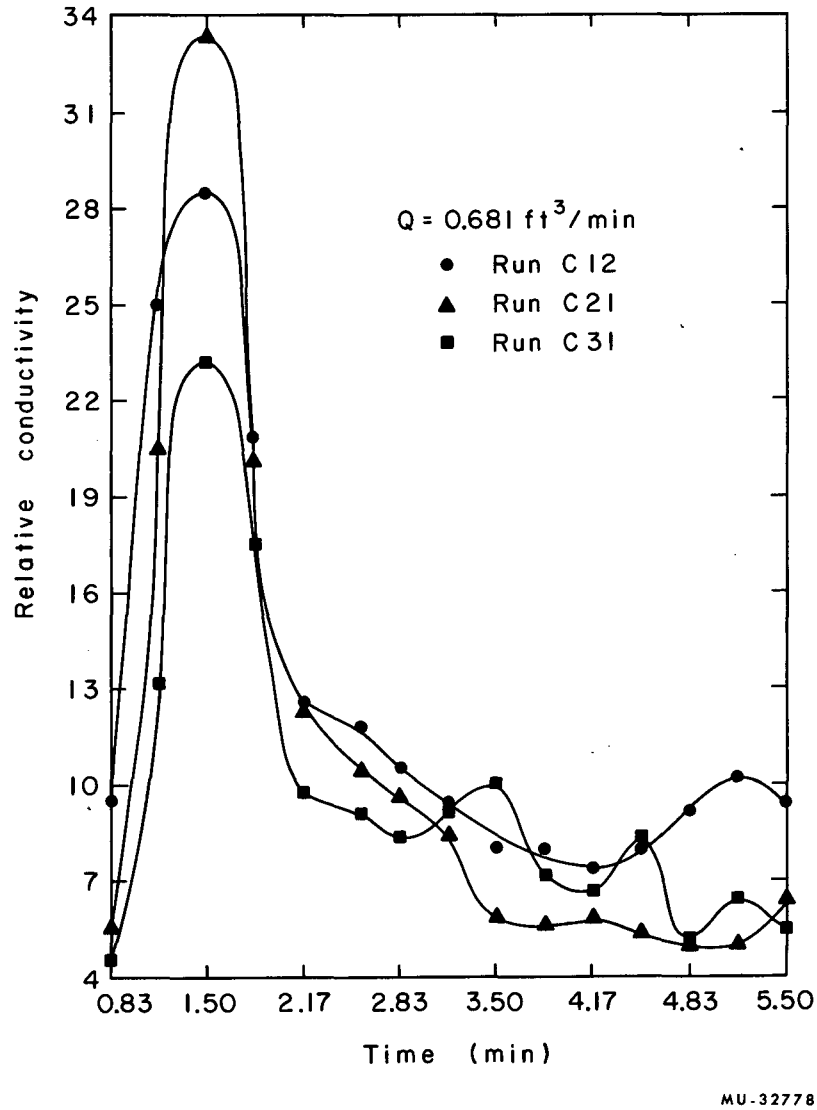


Fig. 14. Conductivity response of injected salt pulse in the Aroclor phase for high flow rate.

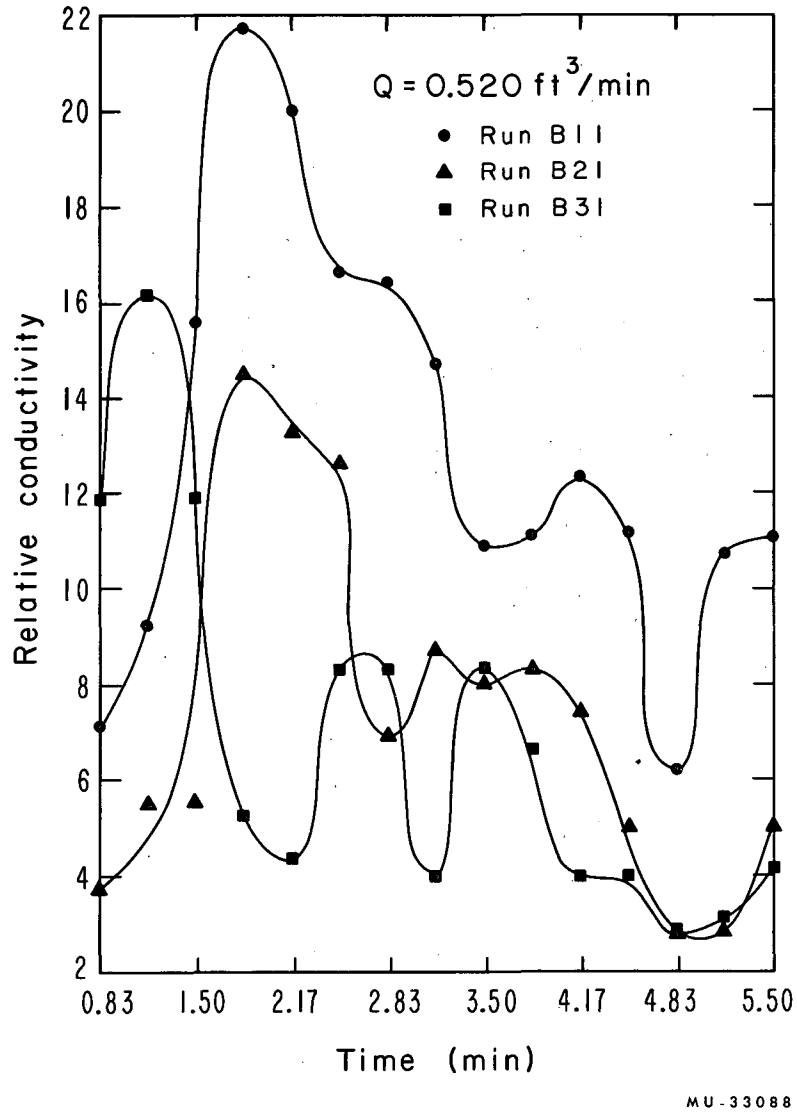


Fig. 15. Conductivity response of injected salt pulse in the Aroclor phase for intermediate flow rate.

D. Flow Patterns

Combining the information obtained by visual observations and from the data, four different types of flow patterns occurring in this study can be sketched.

Figure 16 illustrates the four types of flow patterns. The flow pattern for Run A21 represents the case where primary settling is rapidly completed and no water is entrained when the Aroclor is pulled down the end of the chamber. Run B21 illustrates a situation in which water is entrained in the Aroclor flowing down the back of the settler and circulation is induced. There is considerable clockwise circulation occurring in Run C31, but most of the flow moves below the circulation directly to the exit. Run C12 illustrates the flow pattern where the point of upturn is near the end of the settling chamber and the direction of circulation at the end of the chamber is reversed.

E. Dispersion Zone

There are several other factors affecting the concentration profiles of Table II other than the short circuiting of the flow. Two of the most important factors are the rate of settling and the rate of coalescence.

The concentration profile at a given cross section was always constant or increasing toward the interface. This is because the large drops rise faster than the small ones and approach the interface more rapidly than the small ones. Very high rates of change in concentration were due to the dispersion's preventing the drops from rising and to short circuiting. The constant concentrations obtained were in most cases due to the short-circuiting effect. They also could be caused by the fact that the primary settling was complete in that section of the settler and only very slow secondary settling was occurring. The vertical concentration profiles at distances of 1, 2, and 3 ft down the settler for Run C12 were plotted on Fig. 17 to illustrate the effect of the dispersion on these profiles. The profile at the end of the chamber was not plotted, as it was affected by the short circuiting of the flow.

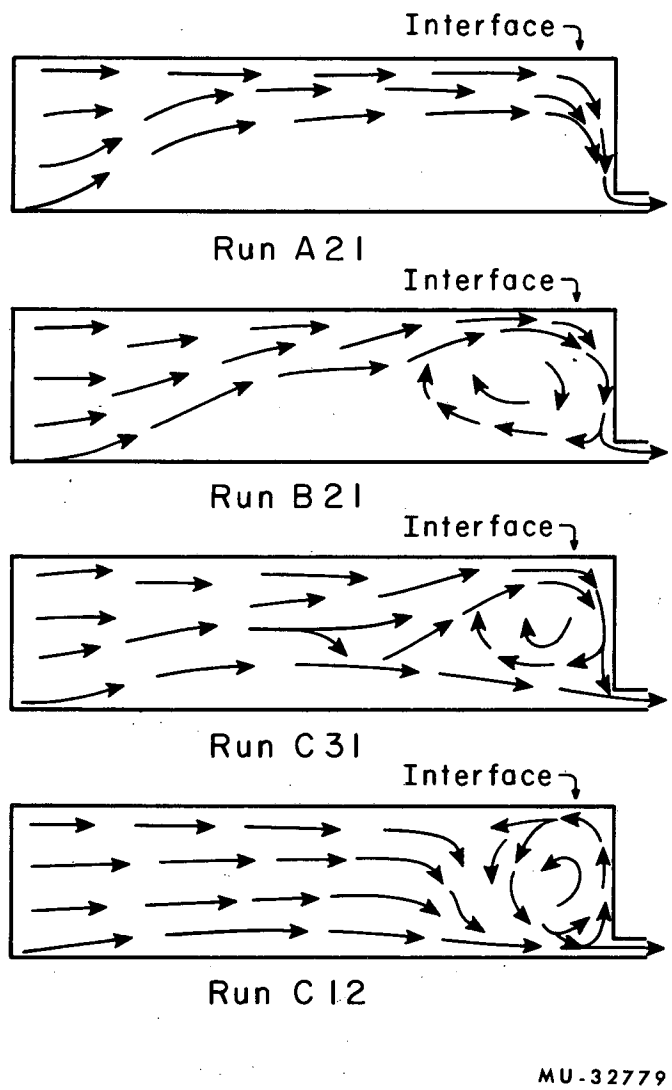


Fig. 16. Typical flow patterns in the settler.

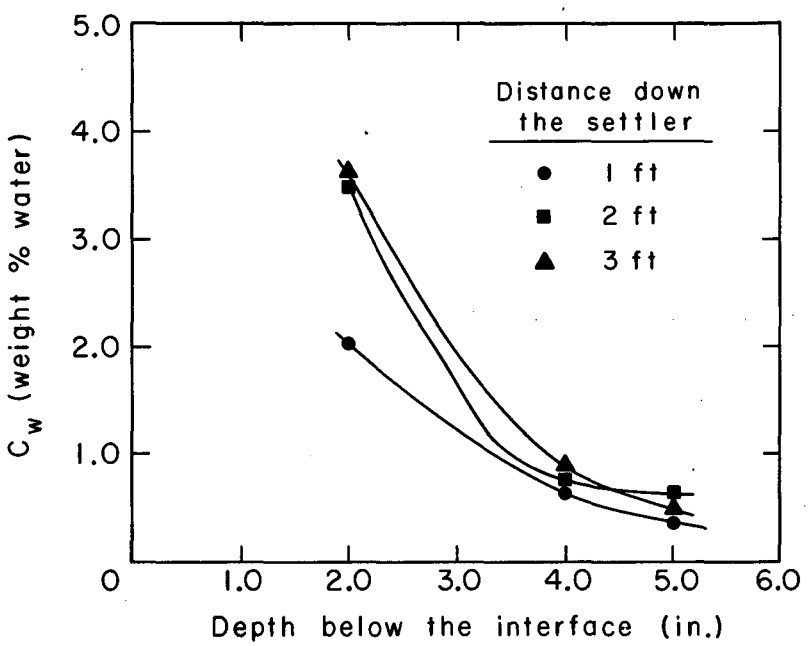


Fig. 17. Concentration profiles at different settling lengths, Run C12.

The profiles for low flow rates and low extents of mixing either increased to a maximum and then decreased until primary settling was complete, or--if primary settling was not completed--decreased until the effects of entrainment from short circuiting were noted. At higher flow rates and higher extents of mixing, the concentration profiles increased down the chamber. These profiles are illustrated in Fig. 18.

The curve on Fig. 18 for Run A21 illustrates the case when the primary settling was complete and no entrainment occurred. Run B21 shows the effect of entrainment on a decreasing concentration profile. In the aforementioned runs, the dispersion zone was either decreasing in depth down the chamber or did not extend the length of the chamber. When the dispersion zone went through a maximum, or increased in depth down the settling chamber, the water concentration profiles increased down the chamber as the curve for Run C21 illustrates.

When the dispersion depths and concentration profiles decreased down the settling chamber there was sufficient time for the dispersion to coalesce or nearly coalesce before it reached the end of the chamber. Therefore the dispersion did not have any effect on the rate of settling or the exit-water concentration, and the rate of settling was considered the controlling factor in the separation process. When the dispersion depth was constant or increasing down the chamber, sufficient time was not available for the dispersion's drops to coalesce. Therefore the dispersion zone was affecting the rate of settling and was an important factor in the separation process. This indicates that the settling process went through a transition stage from a separation controlled by settling rate to a coalescence and settling-rate-controlled separation for the system and conditions studied.

F. Exit-Water Concentrations

The effect of the extent of mixing on the exit-water concentration in the Aroclor phase is shown on Fig. 19. An increase in exit-water concentration with increased entering interfacial area was noted. This may

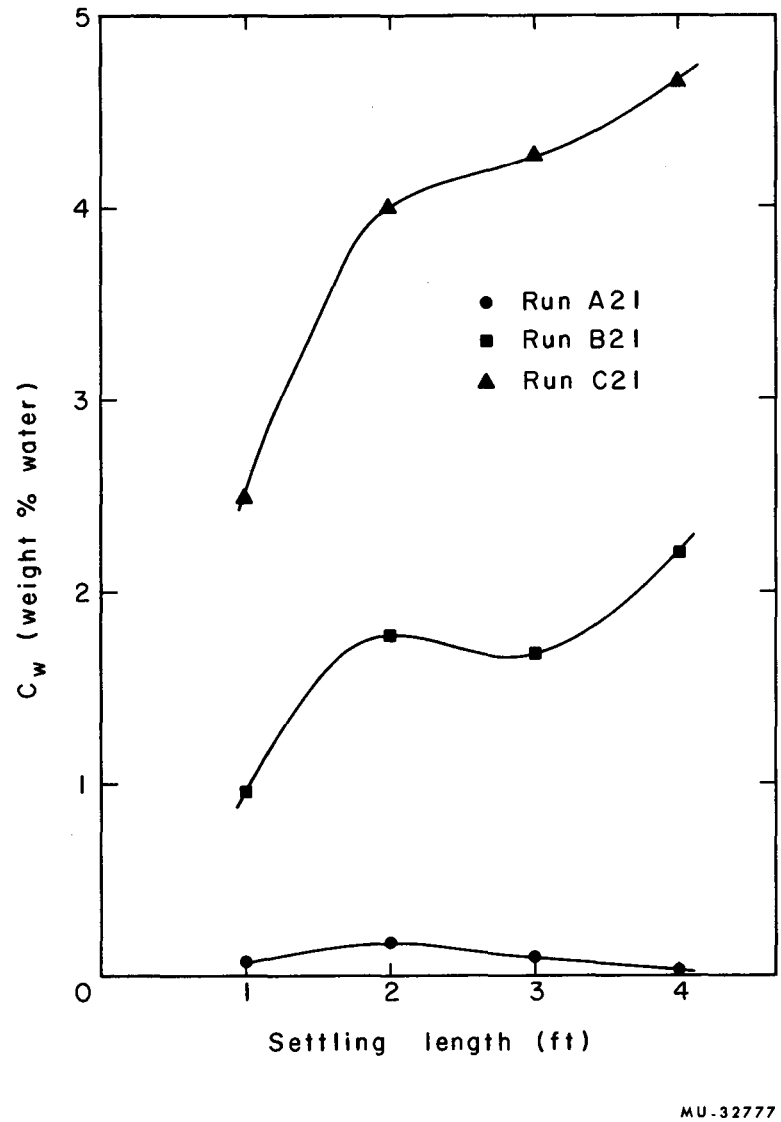


Fig. 18. Various types of concentration profiles obtained 2 inches below the interface.

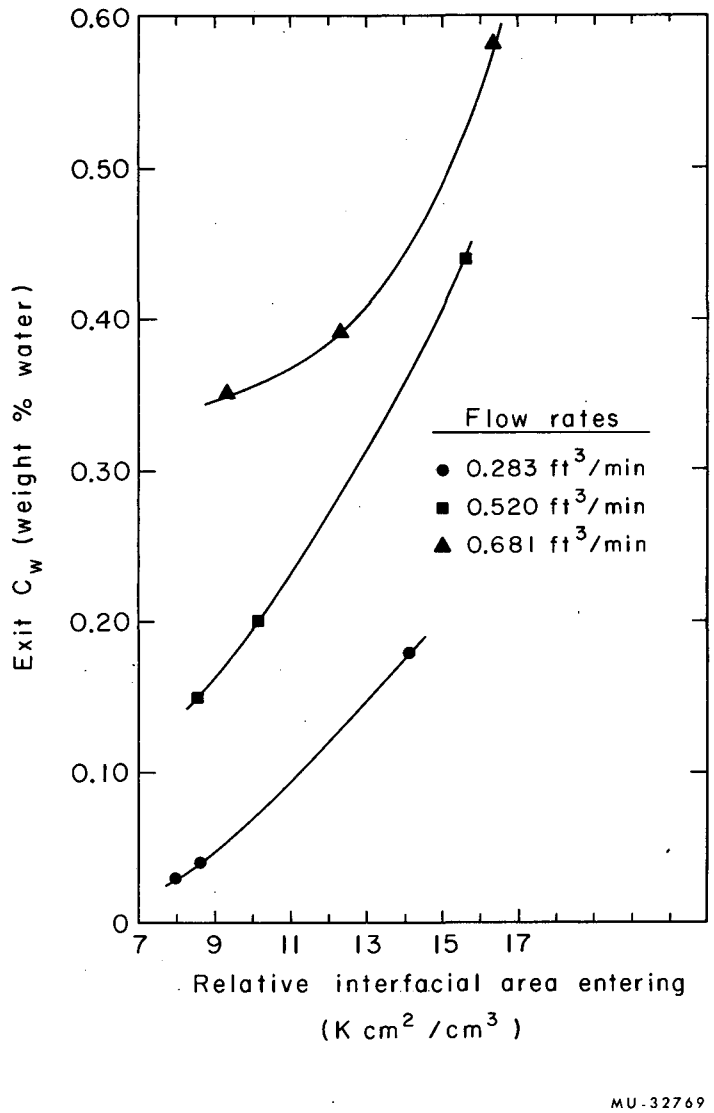


Fig. 19. Effect of extent of mixing on exit-water concentration at constant flow rates.

be accounted for by three factors: (a) the slower rate of "settling" of the smaller water drops; (b) the hindering of the rate of settling by the dispersion; and (c) the effect of short circuiting in increasing entrainment of water drops. At the Aroclor layer flow rate of 0.78 ft/min ($Q = 0.283\ ft^3/min$) the effect of hindered settling was negligible whereas it was very evident at the flow rate of 1.87 ft/min ($Q = 0.681\ ft^3/min$). The exit-water concentration profile for the lower flow rate is nearly linear, and the profile for the higher flow rate is considerably curved. The curvature of this profile is perhaps due to the effects of hindered settling.

G. Interface Baffle

Inserting the interface baffle had no effect on the overall settling process. The depth of the dispersion was reduced by the use of the baffle at the interface for all cases investigated, except for Run C12B. The water concentration profiles near the interface were reduced for those runs in which the dispersion depth was decreased, illustrating the effect that the coalescence rate has on the rate of settling. However, the exit concentration was not affected by the use of the baffle. This tends to substantiate the statement previously made, that when the dispersion depth decreased down the settler, the separation process was said to be settling-rate controlled.

H. Interfacial Tension

The interfacial tension measurements are shown on Table IV. The interfacial tension of Aroclor and water samples withdrawn from the system, although constant, was considerably lower than that of samples of fresh Aroclor and water. Comparing Determinations 103 and 104 illustrates that the reduction of the interfacial tension was caused by changes in the systems water. This is perhaps due to collection in the water of minute traces of contaminating materials from the system. The addition of sodium chloride to the water further reduced the interfacial tension.

Several investigators have found that lowering the interfacial tension lowers the rate of coalescence. This explains the increase in the dispersion zone thickness that occurred when a trace of sodium chloride was added to the water for Runs C11 and D11, for a considerable decrease in the interfacial tension was noted (Determination 107).

I. Induced Coalescence

Induced coalescence was investigated by placing a Fiberglas coalescer in the settling chamber. The results are shown in Tables V and VI.

The water concentrations obtained for induced coalescence show that the coalescer greatly enhances the separation of the two phases with only a small increase in the pressure drop through the system. The superficial velocity through the coalescer was varied from 0.56 to 1.59 ft/min and the entering relative interfacial area was varied from 8.37 K to 16.9 K cm^2/cm^3. The primary separation was found to be complete within the first foot of the settling chamber for the entire range of conditions studied, and the pressure drops through the coalescer were relatively small (15.5 to 43.0 in. water).

This work substantiates the findings of Graham that for induced coalescence with adequate Fiberglas pads, increasing the settling chamber length did not improve the separation of water from Aroclor.

X. SUMMARY AND CONCLUSIONS

The mixing-pipe Reynolds number, N_{Re}, was previously used by Graham and Epstein[23,29] as a measure of the extent of mixing of the entering dispersion. However, the addition of an entrance pipe to the system produced a much higher extent of mixing for the same mixing-pipe Reynolds number. To obtain a true measure of extent of mixing a device for measuring light intensity was mounted on the entrance pipe near the settling-chamber inlet. The relative interfacial area present in the dispersion was determined from the light intensity measurements. This device characterized the various types of mixing that occurred and any coalescing that might have occurred prior to the settling chamber inlet, giving a true extent of mixing parameter.

The concentration profiles and visual observations revealed that considerable short circuiting of flow was occurring in the settling chamber resulting in considerable dead space in the settling chamber and water entrainment in the exiting Aroclor stream. The large variation in the extent of short circuiting that occurred during the investigation produced several flow patterns in the settler's Aroclor layer. These flow patterns are illustrated in Figure 14.

The short circuiting was reduced by increasing the Aroclor velocity through the chamber, while not exceeding laminar flow. The ratio of experimental holding times to theoretical no-mixing holding times increased from 0.35 for $Q = 0.283\ ft^3/min$ to 0.70 for $Q = 0.681\ ft^3/min$. The short-circuiting effect would also be reduced by decreasing the depth of the Aroclor layer. It probably would also be reduced if the Aroclor exit were located on the bottom of the settler a short distance from the end of the chamber. Perhaps this would help to reduce the entrainment down the end of the chamber that occurred in this work.

In most cases, the water concentration profiles increased down the settling chamber. This was caused by the short circuiting previously mentioned and by the dispersion zone. The dispersion zone acts as a barrier to the settling out of the water drops, causing them to be swept further down the chamber and increasing the water concentration downstream.

The water concentration in the exiting Aroclor stream was also increased. The strong effect of the dispersion zone on the rate of separation indicates that coalescence is a rate-determining mechanism. This limits the usefulness of the overflow velocity, V_0 , as a design criterion because V_0 assumes the process to be settling-rate controlled.

Placing a Fiberglas coalescer in the settling chamber effectively separated the two phases within the first foot of the settling chamber with relatively little pressure drop through the coalescer for all conditions investigated. The effectiveness of Fiberglas in separating the two phases indicates that induced coalescence should be considered when the design of a liquid-liquid settler is being considered.

The superficial velocity, V_s , appears to be a useful design criterion in induced coalescence settlers. However, further research is necessary to substantiate this possibility, especially at higher superficial velocities than were investigated in this work.

ACKNOWLEDGMENT

This work was performed under the auspices of the U. S. Atomic Energy Commission.

APPENDICES

A. Determination of Interfacial Area in Dispersions Entering Settling Chamber by Light Transmission

Hulst has theoretically shown that plane-parallel light impinging upon a single drop is deflected into a nonparallel pattern which is determined by the ratio of refractive indices of the continuous and dispersed phases.[30] The amount of scattering is proportional to the frontal area of the drop, and hence to its total area.

A study of the amount of light scattering by several dispersions was made by Langlois, Gullberg, and Vermeulen. They found that a linear relation existed between the specific surface area, A, and the extinction ratio, I_0/I, where I_0 is the light intensity through the pure continuous phase and I is the light intensity through the dispersion.[31] The equation obtained was

$$I_0/I = 1 + A/K.$$

To measure the specific area, the light intensity measuring device must be calibrated by photographing dispersions and simultaneously measuring the light intensity. The drops on the picture must then be counted and measured before the constant can be determined. However, the relative interfacial area for different operating conditions is directly obtainable from the light intensity measurements, since $A = (I_0/I - 1)\ K$.

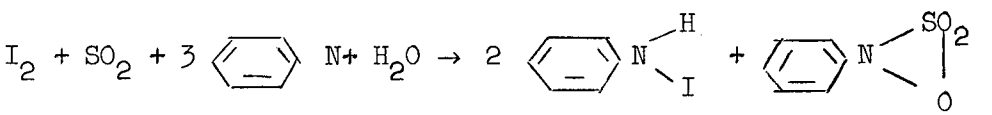
The output current was proportional to the intensity of light reaching the phototube for the operating conditions selected. Therefore, these values were used to determine the extinction ratio, I_0/I.

B. Analytical Procedure for the Determination of Water in Aroclor by use of the Dead-Stop End-Point Method

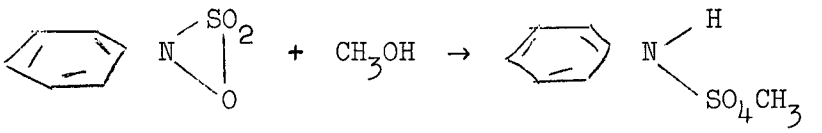
The dead-stop end-point method of water determination involved titrating the sample beyond its end point with Karl Fischer reagent and then back-titrating to an electrolytically determined end point with a standard methyl alcohol-water solution (hereafter called methyl alcohol solution).[32,33]

1. Nature of Karl Fischer Reagent

Karl Fischer reagent is a solution of iodine and sulfur dioxide in pyridine and methanol. The main reactions of Karl Fischer reagent with water appear to be the following:



(1)



(2)

Equations (1) and (2) indicate that the maximum adsorption of water would be 1 mole per mole of iodine. This theoretical strength of the Karl Fischer reagent cannot be obtained, however, because of parasitic side reactions. The exact chemistry of the side reactions is unknown, but they probably included the reaction of iodine and pyridine to form iodopyridine, the reduction of iodine to the iodide ion, and the formation of quaternary methyl-pyridinium salts. The strength of the Karl Fischer reagent decreases rapidly during the first 24 hours after preparation, but within 48 hours the change in strength is less than 0.01 mg/ml/hr.

Stabilized Karl Fischer reagent, which had a water equivalence of about 6 mg per ml solution, was obtained commercially for this work.

2. Preparation of Standard Methyl Alcohol Solution

The standard methyl alcohol solution was prepared by mixing 20 ml of distilled water with 3.8 liters of reagent grade methyl alcohol. This mixture had a water concentration of about 6 mg per ml solution.

3. Apparatus

The equipment used in the determination of water in Aroclor consisted of the dead-stop apparatus, two automatic burets, a magnetic stirrer, and sample bottles.

The dead-stop apparatus consisted of two platinum electrodes connected to a galvanometer and current source as shown in Fig. 20. The electrodes were made by sealing platinum wire in piecesof 4-mm glass tubing.

The two standard solutions were dispensed from 50-ml Pyrex automatic burets, protected from atmospheric moisture by $CaCl_2$ tubes and connected to a vacuum line for filling. Stainless steel hypodermic needles were used as buret tips.

The two platinum electrodes were inserted in a 6 hard rubber stopper. The standard methyl alcohol solution's buret needle was also inserted in the rubber stopper. The Karl Fischer reagent's buret needle was inserted in another hard rubber stopper.

Wide-mouthed 100-ml sample bottles which could be stoppered with 6 rubber stoppers were employed to protect the samples from atmospheric moisture during the titration. Also, magnetic stirrers were used to stir the solutions.

4. Standardization of Reagents

a. Experimental procedure

A 0.25- to 0.35-gram weighed sample of hydrated sodium acetate ($Na_2C_2H_3O_2 \cdot 3H_2O$) was added to a 100-ml sample bottle containing 6 to 8 ml of methyl alcohol solution and a magnetic stirring bar. (The sample bottle and stirring bar were dried at 120° C for several hours and stored in a desiccator.) The sample was then titrated beyond its end point with

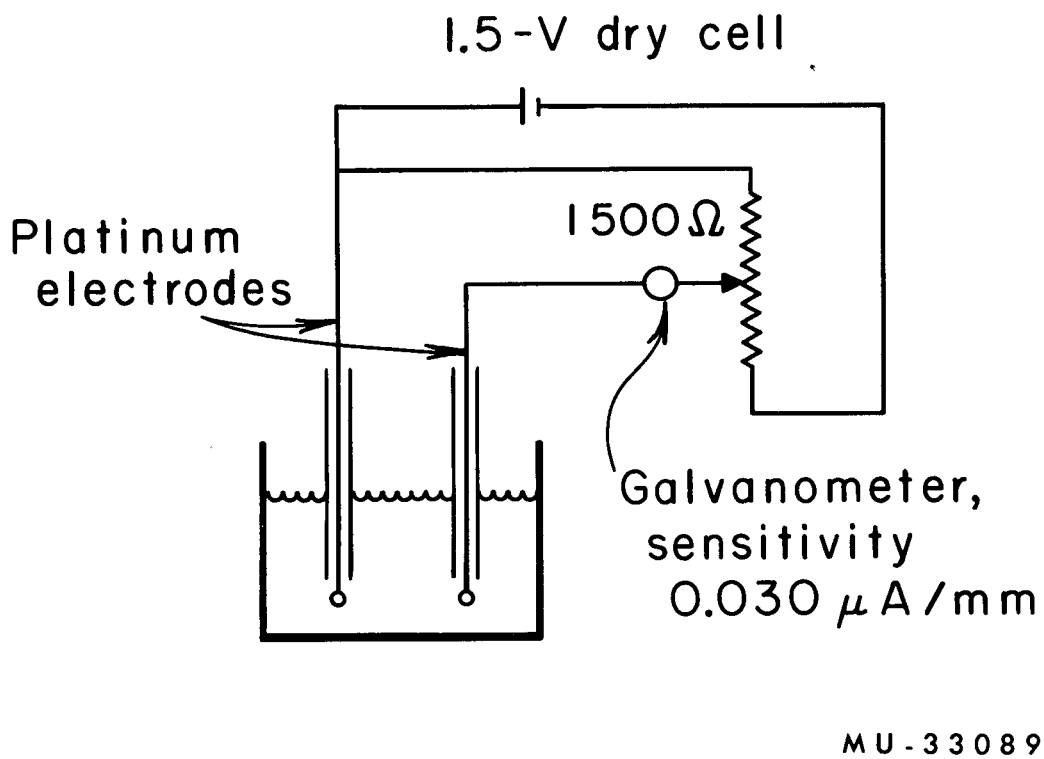


Fig. 20. Dead-stop apparatus wiring diagram.

Karl Fischer reagent. (The Karl Fischer reagent was thoroughly mixed before use to insure a homogeneous solution.) A color change from yellow to the brown color of iodine was noted.

The sample was back-titrated with the methyl alcohol solution to the dead-stop end point. The dead-stop end point was determined as follows:

> The sample was placed on the buret with the electrodes completely immersed in the solution and, with the sample being stirred, the potentiometer was adjusted so that the galvanometer indicated a small current was flowing through the electrodes. The methyl alcohol solution was slowly added. The current flow remained constant until the end point was approached. Then the galvanometer began to show deflections as each drop of solution was added. The end point was reached when the galvanometer suddenly gave a steady reading of nearly no current flow.

A 25-ml aliquot of Karl Fischer reagent was also titrated to the dead-stop end point as described above.

b. Calculation of Reagent Strengths

i. The nomenclature for the equation is:

F = ml of Karl Fischer reagent (KFR),

f = water equivalent of KFR (mg/ml) ,

M = ml of methyl alcohol solution,

m = water concentration in methyl alcohol solution (Mg/ml),

N = water in hydrated sodium acetate (mg),

R = M/F.

ii. The water balance on the first titration yields

$$fF_1 = mM_1 + N .$$

iii. The water balance on the second titration yields

$$fF_2 = mM_2 .$$

iv. Solving these two simultaneous equations gave the water equivalence of the Karl Fischer reagent and the water concentration of the methyl alcohol solution,

$$f = \frac{N/M_1}{1/R_1 - 1/R_2} \quad \text{and} \quad m = f/R_2, \text{ respectively.}$$

A check of the reliability of this method of standardization of solutions was made and the results are shown in Table VII.

Table VII. Standardization of reagents.

Sample	f	m
1	5.910	6.006
2	5.899	5.971
3	5.889	5.997

5. Determination of Water in Aroclor

Samples of Aroclor weighing 30 to 60 grams were collected in 100-ml sample bottles. The adsorbed water in the flasks had been made negligible by drying the flasks at 120° C for several hours and storing them in a desiccator. A dry Teflon-coated stirring bar was placed in the sample. The water content of the sample was then determined by titrating with Karl Fischer reagent until the brown color of iodine appears and then back-titrating with methyl alcohol solution to the dead-stop end point as described in the standardization procedure. The weight percent soluble water (0.081) was subtracted from the total water concentration to show the free water not separated.

The accuracy of this method of water determination is illustrated on Table II, Runs 5, 6, 7, A11, and A12.

C. Physical Properties of Aroclor 1248

Aroclor 1248 is a fire-resistant heat-transfer medium manufactured by the Monsanto Chemical Company.[34] It is chlorinated polyphenyl compounds, essentially tetrachlorodiphenyl. Its physical properties are as follows:

(a) Density[34]

Temperature ($^{\circ}$ C)	Density (g/cm^3)
30	1.44
60	1.41
88 (190°F)	1.382
100	1.37

(b) Viscosity[34]

Temperature ($^{\circ}$ C)	Viscosity (cp)
30	112
60	17.5
88	6.0
100	4.2

(c) Solubility

Graham determined the solubility of water in Aroclor 1248 by titrating with Karl Fischer reagent. He presents a plot of concentration of water (mole %) as a function of temperature. From this plot one finds the solubility of water at 190° F is 1.3 mole %; 1.3 mole % is equal to 0.081 weight %. Aroclor 1248 is insoluble in water.

D. Calculations

1. Mixing-Section Reynolds Number

The mixing-section Reynolds number was calculated by using the average density of the Aroclor-water mixture. Since the viscosity of the

Aroclor-water mixture could not be determined, the viscosity of the continuous phase (Aroclor) was used in the calculation.

For run B11 the conditions were:

$Q = 0.520\ ft^3/min,$

Number of 0.25-in. tubes open = 1

$A_t = 3.4071 \times 10^{-4}\ ft^2,$

$D_t = 0.0208\ ft,$

$\rho_A = 85.5\ lb/ft^3,$

$\rho_W = 61.2\ lb/ft^3,$

$\mu_A = 3.36 \times 10^{-3}\ lb/ft\ sec,$

$C_W = 0.15$ weight fraction,

$C_A = 0.85$ weight fraction.

The average density is

$$\rho_{av} = \frac{1}{\frac{C_W}{\rho_W} + \frac{C_A}{\rho_A}} = \frac{1}{\frac{0.15}{61.2} + \frac{0.85}{85.5}} = 80.7\ lb/ft^3$$

The average velocity is

$$U_{av} \simeq \frac{Q}{A} = 0.5198\ ft^3/min \quad \frac{min}{60\ sec} \quad \frac{1}{3.407 \times 10^{-4} ft^2}$$

$$U_{av} = 25.43\ ft/sec,$$

therefore

$$\mu_{Re} = \frac{DU\rho}{\mu} = \frac{0.0208\ ft \times 2543\ ft/sec \times 80.7\ lb/ft^3}{3.36 \times 10^{-3}\ lb/ft\ sec}$$

2. <u>Standardization of Water Determination Reagents</u>

For Run B11 the data were:

$NaAc \cdot 3H_2O$ wt = 0.23629 gm,

$F_1 = 26.2$ ml,

$M_1 = 10.75$ ml ,

$F_2 = 25.0$ ml,

$M_2 = 25.05$ ml,

$$n = \frac{1000\ (NaAc \cdot 3H_2O\ wt)\ MW_{3H_2O}}{MW_{NaAc \cdot 3H_2O}},$$

$$= \frac{(1000)\ (0.2363)\ (54.05)}{(136.09)} = 93.846 \text{ mg};$$

$$1/R_1 = F_1/M_1 = 26.2/10.75 = 2.437,$$

$$1/R_2 = F_2/M_2 = 25.0/25.05 - 0.998,$$

$$f = \frac{N/M_1}{1/R_1 - 1/R_2} = \frac{93.8458}{(10.75)\ (1.4392)} = 6.066 \text{ mg/ml},$$

$$m = f/R_2 = (6.066)\ (0.998) = 6.054\ .$$

E. Nomenclature

A = surface area in dispersion (cm^2/cm^3)

C_0 = initial water concentration present in feed to the settler (wt %)

C_w = water concentration in the Aroclor phase (wt %)

d = mean drop diameter (cm)

f = water equivalent of Karl Fischer reagent (mg/ml)

f = volume of Karl Fischer reagent (ml)

g = acceleration due to gravity (cm/sec^2)

I = light transmission through dispersion, measured as photocell output (microamperes)

I_0 = light transmission through the continuous phase alone, measured as photocell output (microamperes)

k = experimentally determined constant

L = settling chamber length (ft)

M = volume of methyl alcohol solution (ml)

m = water concentration of methyl alcohol solution (mg/ml)

N = water in hydrated sodium acetate (mg)

N_{Re} = mixing pipe Reynolds number

Q = $Q_A + Q_w$, dispersion flow rate (ft^3/min)

Q_A = Aroclor flow rate (ft^3/min)

Q_w = water flow rate (ft^3/min)

R = M/K, reagent strength ratio

r = drop radius (cm)

U = average linear injection velocity (ft/min)

U_A = average linear velocity of Aroclor layer (ft/min)

U_w = average linear velocity of water layer (ft/min)

U_∞ = terminal settling velocity (cm/sec)

V_i = settling chamber entrance velocity (ft/min)

V_0 = overflow velocity (ft/sec)

V_s = superficial velocity through coalescer (ft/min)

w = apparent drop weight (gm)

X_b = distance, baffle to inlet (in.)

Z_A = depth of Aroclor layer in settling chamber (in.)

Z_D = depth of dispersion zone (in)

Z_I = height of inlet above Aroclor-water interface (in.)

ΔP = pressure drop across coalescer (in. water)

$\Delta\rho$ = difference in density between dispersed-liquid and continuous phases (gm/cm^3)

μ_c = viscosity of continuous phase (gm/cm sec)

μ_D = viscosity of dispersed phase (gm/cm sec)

θ_{exp} = time for bulk of salt pulse to reach settling chamber exit (min)

θ_{nm} = no-mixing residence time (volume of phase)/(volumetric flow rate) (min)

σ_I = interfacial tension (dynes/cm)

γ_c = kinematic viscosity of continuous phase (cm^2/sec)

γ_D = kinematic viscosity of dispersed drops (cm^2/sec)

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