Title

SEPARATION OF IMMISCIBLE LIQUIDS BY GRAVITY SETTLING AND INDUCED COALESCENCE; PART ONE: HORIZONTAL GRAVITY SETTLERS; PART TOO: GRAVITY SETTLING WITH INDUCED COALESCENCE

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Publication Date

1962-02-05

UCRL 10048

University of California

Ernest O. Lawrence Radiation Laboratory

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UCRL-10048
UC-4 Chemistry
TID-4500 (17th Ed.)

UNIVERSITY OF CALIFORNIA
Lawrence Radiation Laboratory
Berkeley, California

Contract No. W-7405-eng-48

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(M. S. Thesis)

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Printed in USA. Price $2.00. Available from the
Office of Technical Services
U. S. Department of Commerce
Washington 25, D.C.

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ABSTRACT

The separation of water from Aroclor 1248 by use of gravity settlers was investigated. The water concentration in the effluent Aroclor was found to depend on several factors, including the inlet and baffle positions, the initial water concentration, and the degree of turbulent mixing upstream from the settler, as well as on the Aroclor flow rate and depth, and the settler length. At high flow rates, an emulsion of water and Aroclor droplets formed at the Aroclor-water interface. This emulsion influenced the water concentration in the effluent Aroclor. Although the general influence of these factors on the separation of water from Aroclor 1248 was established, it was not possible to correlate them in designing full-scale gravity settlers from laboratory-scale settlers.

Fiberglas coalescers mounted in the settlers were effective in coalescing the emulsion and removing Aroclor droplets from the water layer. The coalescers with the most surface area were the most efficient in removing water from the Aroclor. Pressure drops through the coalescers were negligible.

NOMENCLATURE

C_W = insoluble water concentration in Aroclor effluent from settler (weight %)

$C_{W(0)}$ = initial water concentration present in feed to settler (weight %)

D = inlet diameter (in.)

d = mean droplet diameter (cm)

g = gravitational acceleration

L = settler length (ft)

N_{RE} = Reynolds number in the mixing pipe

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

r = dispersed-droplet radius (cm)

U = average linear inlet velocity (ft/sec)

U_∞ = terminal settling or rising velocity of dispersed droplets (cm/sec)

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

U_O = Ohnesorge's critical velocity (ft/sec)

V_o = overflow velocity (ft/sec)

W_A = work of adhesion

w = apparent weight of drop

X_b = distance, baffle to inlet (in.)

X_c = distance, inlet to coalescer (in.)

Z_A = depth of Aroclor layer in settler (ft)

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

$\Delta\rho$ = difference in density between dispersed-liquid and continuous phases

μ = viscosity of injected liquid

μ_c = viscosity of continuous liquid phase

μ_d = viscosity of dispersed liquid phase

θ = angle of contact between solid surface and liquid drop

σ = surface tension

σ_1 = interfacial tension

ν = kinematic viscosity of injected liquid

ν_c = kinematic viscosity of continuous phase

ν_d = kinematic viscosity of dispersed phase

PART I

HORIZONTAL GRAVITY SETTLERS

I. INTRODUCTION

Gravity settlers are used extensively in the oil and chemical industries to separate mixtures of two immiscible liquids. In many liquid-liquid solvent-extraction operations, settlers are used to separate the extract liquid from the raffinate liquid after the two have been contacted in a mixing tank. Refineries use settlers in many operations, for example for dewatering crude oils, desalting crude oils, caustic washing of light distillates, sulfuric acid treatment, and solvent extractions.

The simplest form of gravity settler consists of a long horizontal chamber with a circular or rectangular cross section. A mixture of two immiscible liquids is introduced into one end of the chamber. The liquids then separate into two layers because of a difference in density. The lighter liquid is withdrawn at the top of the opposite end of the chamber, and the heavier liquid is withdrawn from the bottom of the opposite end.

There are many possible variations of the simple horizontal settler, and many patents have been issued on them. The vertical settler is one variation, in which the feed is introduced at the middle of a vertical or inclinded cylinder.[1, 2, 3] The light liquid is withdrawn from the top and the heavy liquid is withdrawn at the bottom.

Baffles are used in many settlers to reduce both turbulence and vertical settling distance.[4, 5] Variations in the inlet position and design are also incorporated to reduce turbulence at the inlet.[2, 6]

In spite of their extensive use, the literature contains little on the design of gravity settlers. The purpose of Part I of this paper is to investigate (a) some of the factors affecting gravity-settler operation, and (b) the feasibility of devising a scale-up method for gravity settler. In Part I, the separation of Aroclor 1248 and water by gravity settling is studied. (See Appendix C for the physical properties of Aroclor.)

II. FACTORS AFFECTING THE SEPARATION OF TWO IMMISCIBLE LIQUIDS

A. Unstable Emulsions

An unstable emulsion described by Chertow and Meissner[7] is reported[5] to be similar to the separation of two immiscible liquids in a gravity settler. In their description, when agitation of two immiscible liquids is stopped, three layers become apparent almost immediately. The upper layer contains the light liquid, the lower contains the heavy liquid, and the central zone contains the unsettled emulsion. The emulsion of the center layer continues to break up by coalescence and sedimentation until upper and lower layers meet at a sharp interface. This period is called the primary break, and takes place within a few seconds in most cases. After the primary break, a cloudy dispersion may be present in either the upper or lower layer. This dispersion eventually settles out, but may take days in some cases.

D. H. Putney describes the separation of paraffinic hydrocarbons from sulfuric acid in alkylation operations.[8] When mixing is stopped, two zones become apparent, an upper zone composed of hydrocarbons, and a lower zone composed of an emulsion of hydrocarbons dispersed in sulfuric acid. Dispersed hydrocarbon droplets in the emulsion layer collect at the interface. The droplets at the interface have the appearance of a foam from which globules of hydrocarbons continually coalesce into the hydrocarbon layer.

B. Settling Velocity of Isolated Drops

Many equations have been proposed to describe the rate of rise or fall of an isolated drop immersed in a stagnant layer of another immiscible liquid.[9-12] These equations cannot be applied directly to gravity-settler design because they do not take into account such factors as coalescence, foaming, and droplet-size distribution. However, these equations do provide some useful information about a liquid-liquid system.

Bond and Newton's modification of Stokes' Law is one such equation:

$$U_\infty = \left(\frac{2\Delta\rho g\ r^2}{g\ \mu_c}\right)\left(f\ \frac{\mu_d}{\mu_c}\ ,\ \frac{r\sigma_I}{w}\right) ,$$

where

U_∞ = terminal settling or rising velocity of dispersed droplets (cm/sec),

μ_C = viscosity of continuous liquid phase,

μ_d = viscosity of dispersed liquid phase,

r = dispersed-droplet radius (cm),

$\Delta\rho$ = density difference between liquid dispersed and continuous phases,

g = acceleration due to gravity,

w = apparent weight of drop,

and

σ_I = interfacial tension.

For small values of $(r\sigma_I)/w$, i.e., large droplets,

$$f \ \frac{\mu_d}{\mu_c}, \ \frac{r\sigma_I}{w} \rightarrow \frac{(1 + \mu_d/\mu_c)}{(2/3 + \mu_d/\mu_c)} \quad .$$

For large values $(r\sigma_I)/w$, i.e., small droplets,

$$f\left(\frac{\mu_d}{\mu_c}, \frac{r\sigma_I}{w}\right) \rightarrow 1.$$

From the equation it can be concluded that high viscosity of the continuous phase, small density difference between the two phases, and small droplet size, are factors that hinder separation.

For small drops Bond and Newton's equation becomes Stokes' law. Stokes' law was therefore applied to the Aroclor 1248-water system, as shown in Table I. Table I shows that the settling time for water droplets dispersed in Aroclor 1248 should be sixteen times as great as for Aroclor 1248 droplets in water. This indicates that the separation of water droplets from Aroclor would be more difficult than the separation of Aroclor droplets from water. Very fine dispersions in either phase have very long settling times and may be equivalent to stable emulsions.

Table I. Comparison of U_∞ for an Aroclor 1248-water system at 200 °C

r (cm)	$r\sigma_I/w$	U_∞ for water drops in Aroclor (cm/sec)	U_∞ for Aroclor drops in water (cm/sec)
0.01	272	0.1795	2.94
0.001	27200	0.001795	0.0294

C. Coalescence

Coalescence is a most important factor in the separation of two immiscible liquids. Several investigations of the coalescence of oil and water drops at an oil-water interface have been performed, but this information cannot be applied directly to gravity-settler design. However, some of the observations are of interest.

Cockbaine and McRoberts,[13] Nielsen, Wall, and Adams,[14] and Lang et al.[15] have observed that oil and water drops have a finite lifetime at an oil-water interface before they coalesce into the bulk phase. This lifetime may be only a few seconds for pure liquids, but even a small amount of certain impurities such as dissolved air can increase the drop lifetime. Small amounts of certain substances have been found to increase the drop lifetime by minutes or even hours.

Lang's data for the coalescence of Aroclor-1248 drops, at an Aroclor-water interface at 25° C, are shown in Fig. 1.[15] The data were obtained by allowing a droplet of Aroclor 1248 to rest at an Aroclor-water interface, and then measuring the time required for the droplet to coalesce. For some of the liquid-liquid systems studied, the coalescence of a droplet occurred in several steps, i.e., in each step, part of the droplet coalesced into the bulk liquid while part also remained at the interface as a smaller droplet. The Aroclor droplets, however, coalesced completely into the Aroclor layer in one step. Droplets of various radii were formed by passing small amounts of Aroclor through various-sized dropping tips.

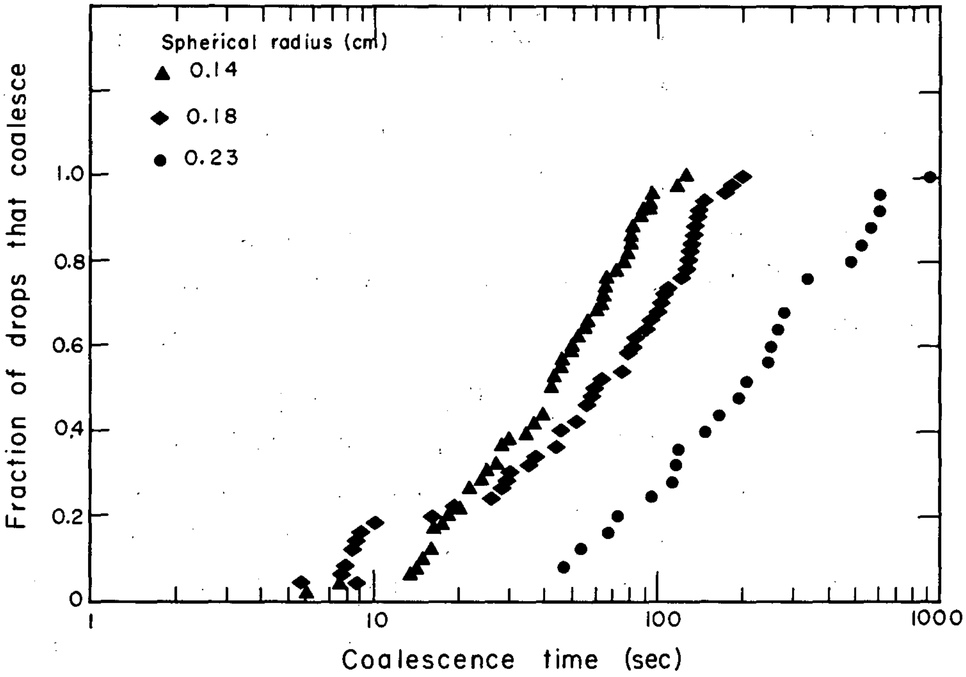

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Fig. 1. Coalescence curves for Aroclor 1248 drops at the Aroclor-water interface (25° C).

Figure 1 shows that the larger droplets were more stable. Droplet lifetimes as high as 900 sec were observed in some cases.

Lang developed a correlation relating liquid properties and temperature to the average droplet lifetime. Increasing the temperature greatly reduces the average droplet lifetime. At the operating temperature used in this work (200° F), Lang's correlation predicts an Aroclor droplet lifetime about 1/20 as long as at 25° C. It also predicts that water droplets at an Aroclor-water interface will have lifetimes five times as great as Aroclor droplets at 200° F.

These observations on droplet lifetime may explain the stability of the emulsion layer described by Chertow and Meissner, and Putney. Coalescence is also important in the settling process, because coalescence of small droplets to form large ones increases the rate of separation.

D. Inlet Velocity

Stable emulsions can be produced by injecting one liquid into a stagnant layer of another at high velocities. Ohnesorge's relation predicts the critical velocity, U_O, required to form a stable emulsion in terms of the inlet diameter and the liquid properties:[16]

$$\mu^{1/2} \left(\frac{\sigma_I D}{\nu} \right)^{-1/2} = 2000 \left(\frac{\nu}{U_O D} \right)^{4/3} ,$$

where

μ = viscosity of injected liquid,

ν = kinematic viscosity of injected liquid,

σ_I = interfacial tension between injected liquid and stagnant liquid,

D = inlet diameter

and

U_O = critical inlet velocity for injected liquid

Richardson developed an empirical equation relating the mean droplet diameter, the inlet velocity, and the liquid properties for velocities above the Ohnesorge critical velocity:[17]

$$d = \frac{4000\,\nu_d^{1.8}}{U\nu_c^{0.8}} ,$$

where

d = mean droplet diameter,

U = average linear inlet velocity,

ν_d = kinematic viscosity of dispered droplets,

and

ν_c = kinematic viscosity of continuous phase.

Since the inlet velocities used in this paper were much less than Ohnesorge's critical velocity, Richardson's formula cannot be applied directly. However, the formula does indicate that increasing the inlet velocity decreases the mean droplet size thereby increasing the settling time.

E. Mixing of Two Immiscible Liquids

The degree of mixing of the two immiscible liquids has a definite effect on the particle size, and thereby the settling time. Many investigators studying emulsions have found that increasing the mixing speed decreases particle size.[18-21, 23] Other investigators have found that increasing the mixing time reduces the particle size.[22, 23]

Clay found that the particle-size distribution of two immiscible liquids passing through a pipe in turbulent flow was affected by the Reynolds number.[24] That is, increasing the Reynolds number decreases the droplet size, while decreasing the Reynolds number favors the formation of large droplets.

F. Type of Flow in Settler

W. B. Hart wrote a series of articles on the design of sedimentation chambers for the separation of oil from refinery waste water.[25, 26] The separation process involves passing the waste water through large open rectangular settling chambers. Oil globules dispersed in the water rise to the surface, where the oil is collected. Hart maintained that the type of flow through the settling chambers is the most important factor in the separation. The flow must be free of turbulence that is powerful enough to overcome the buoyancy of the dispersed oil globules. He used the Reynolds number as a measure of the turbulence in the settler, and presented an experimental correlation that related the density of the dispersed oil to the maximum permissible Reynolds number (see Fig. 2).

In this work, the Reynolds numbers used were much lower than those used by Hart. Hart's correlation allowed Reynolds numbers as high as 20000 for the separation of 45° A. P. I. oil from water. The maximum Reynolds number used in our work was 124. Since laminar flow always occurs for Reynolds numbers less than 144, according to the investigation by Hopf,[27] it is doubtful that turbulence was a significant factor in this work.

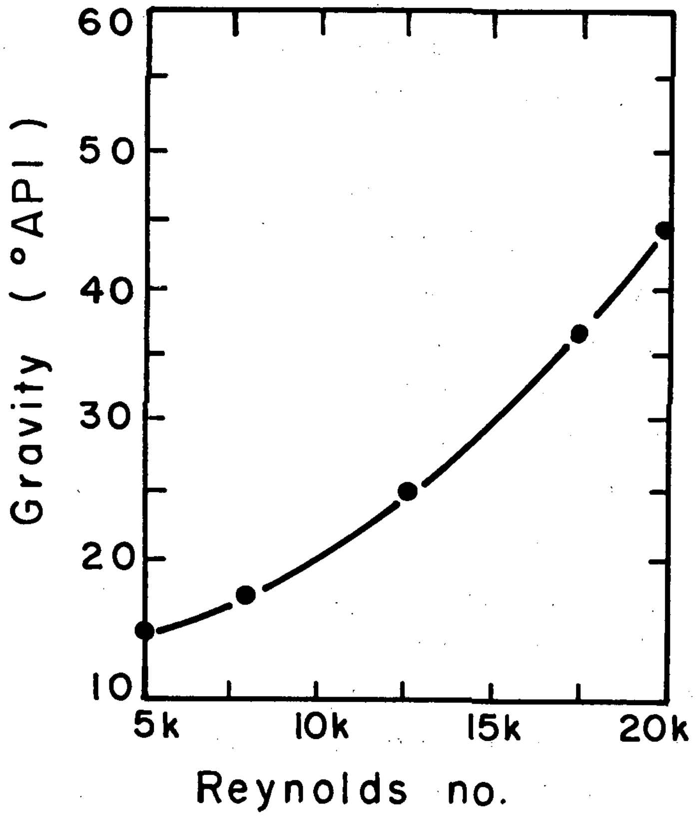

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Fig. 2. Relationship of Reynolds' s number to gravity (°API) of oil to be separated (see reference 25), given here for a settler with cross section 8 ft deep by 20 ft wide.

III. DESIGN PROCEDURES

A. General Design Procedures

Treybal states that the primary break of an unstable emulsion is ordinarily so rapid that a short period without agitation is sufficient to permit separation of two immiscible liquids.[5] In continuous flow, this period without agitation is provided by allowing the mixture to pass through a large vessel where turbulence is small and with a large enough residence time to provide separation. The residence time may be defined as the volume of the settler divided by the volumetric flow rate into the settler.

The residence time is determined from past experience, or by pilot-plant equipment. In the petroleum industry, residence times of 30 to 60 min are used.[5] D. H. Putney advises a residence time of 60 to 70 min for the separation of spent sulfuric acid catalyst from reacted hydrocarbons in alkylation operations.[8] Warner states that settlers used in solvent extraction of irradiated fuels were scaled up satisfactorily from pilot-plant models on the basis of geometrical similarity and residence time.[28]

B. Design Procedures Based on the Overflow Velocity

Consider the ideal settler shown in Fig. 3. Assume that there is no short circuiting of flow, no turbulence, and no eddies.

A mixture of two immiscible liquids, A and B, flows into the settler and liquid B, being less dense than liquid A, forms the upper layer. Consider droplets of B dispersed in liquid A. These droplets move upward toward the interface of A and B with a velocity equal to the terminal velocity U_∞ determined by Stokes' Law. These droplets also have a horizontal velocity U_A, determined by the volumetric flow rate of liquid A divided by the cross-sectional area of liquid A perpendicular to the direction of flow. The maximum vertical distance that a dispersed droplet of B would have to travel to reach the interface would be Z_A. On the basis of these assumptions, then, any droplet of liquid B dispersed in liquid A would separate from liquid A for $U_\infty \geqslant (U_A Z_A)/L$. The term $(U_A Z_A)/L$ is known as the overflow

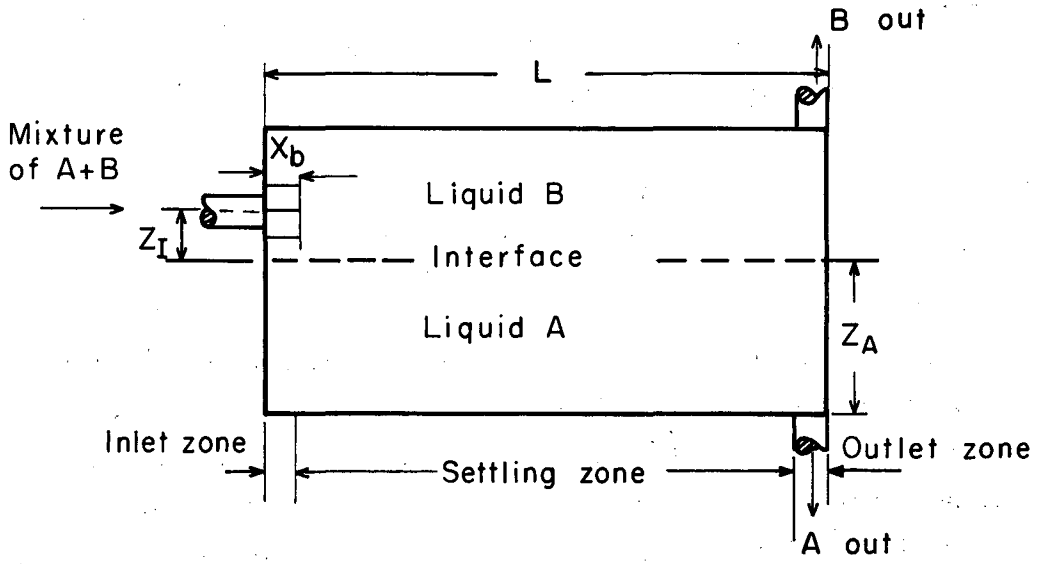

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Fig. 3. Ideal horizontal gravity settler.

velocity V_o. This overflow velocity may also be expressed in terms of the residence time T, where $T = L/U_A$, and

$$V_o = \frac{Z_A}{T} = \frac{U_A Z_A}{L} \quad .$$

Camp states that the overflow velocity is the chief criterion in the design of settling tanks for the separation of suspensions of solid particles from waste water.[29] From a distribution curve of the settling velocities of the particles of the suspension, the theoretical removal of particles can be determined on the basis of the overflow velocity.

Hart applied the overflow velocity to the design of separators for removing oil from refinery waste waters.[25, 26] The length of the settler L was calculated from the relation

$$V_o = \frac{U_A Z_A}{L} \ , \ \text{or} \quad L = \frac{U_A Z_A}{V_o} \quad .$$

The value of U_A was determined from a relationship between the maximum possible Reynolds number and the density of the oil to be separated (see Sections II-E, F). The value of Z_A usually has been taken as 8 ft, since this was a standard depth used in oil-water separators. The overflow velocity V_o was determined by equating V_o to U_∞, calculated from Stokes' law by using a particle diameter of 0.02 cm, the densities of the oil and water, and the viscosity of water.

Hart's articles served as the basis for the A.P.I. design for oil-water settlers.[30] However, several modifications were made. The overflow velocity V_o was calculated by the same method as Hart used, except that the particle diameter was taken to be 0.015 cm. The horizontal velocity was taken as 15 times V_o, but no greater than 3 ft/min. The depth-to-width ratio was taken to be between 0.3 and 0.5. The width was taken to be between 6 and 20 ft. The length was calculated by the relation

$$V_o = F_s F_t \left(\frac{U_A Z_A}{Z} \right) \ , \ \text{or} \quad L = F_s F_T \left(\frac{U_A Z_A}{V_o} \right) \quad .$$

The factor F_s was a correction for short circuiting of flow and was found experimentally to be 1.2. The factor F_t was a correction for turbulence related to the ratio of U_A/V_o.

The relationships used in the A. P. I. design were determined from data taken from separator models and plant operations. The A. P. I. manual states that the design may be applied to any rectangular settling chambers where the flow of waste water is uniform and free from eddy currents, and where the oil particles to be separated are 0.015 cm in diameter. The extent to which such ideal conditions are met are limited. However, data taken from operating plants and experimental studies indicate that oil-water settlers designed by these methods separate a high percentage of the oil in refinery waste water.

Ledesma applied the overflow velocity to the design of gravity settlers for separating water from Aroclor 1248.[31] For the separation of two immiscible liquids, assuming the ideal settler, it was shown that any droplet of a size such that the terminal rising velocity U_∞ was equal to or greater than the overflow velocity V_o would separate. Complete separation of all droplets larger than a given size would yield a definite water concentration C_W in the Aroclor effluent, due to the smaller drops remaining. This would mean

$$C_W = f\left(\frac{U_A Z_A}{L}\right), \quad \text{or } C_W = f(V_o) \quad .$$

IV. EQUIPMENT

A schematic diagram of the equipment used in this experiment is shown in Fig. 4. The three main sections consisted of the Aroclor system, the water system, and the mixing and settling system.

A. Aroclor System

The Aroclor was stored in a 55-gal Bisonited drum equipped with a sight glass and two thermometers. A 1/4-hp mixer was used to agitate it, and four 3750-watt immersion heaters controlled by a rheostat were used to heat it.

A 3/4-in. copper line connected the Aroclor tank with a 5-gpm gear pump. A needle valve on a bypass line was used to control the Aroclor flow rate.

Rust and dirt particles were removed from the liquid by a 25-μ filter downstream from the pump. Final adjustment of the flow rate was accomplished with a needle valve located upstream from a rotameter.

B. Water System

The water was stored in another 55-gal Bisonited tank equipped with a sight glass and thermometer. The water was heated to operating temperature by a 15-psig steam line that injected steam directly into it. A 500-w heater with a thermostat control was used to maintain the water temperature at a constant level, and a 1/8-hp mixer was used to agitate it.

The water was pumped by a 5-gpm centrifugal pump through 3/8-in. copper tubing. Control of the water flow rate was maintained by a needle valve located in front of the water rotameter. A 10-μ Cuno filter was located upstream from the rotameter for removing dirt and rust particles.

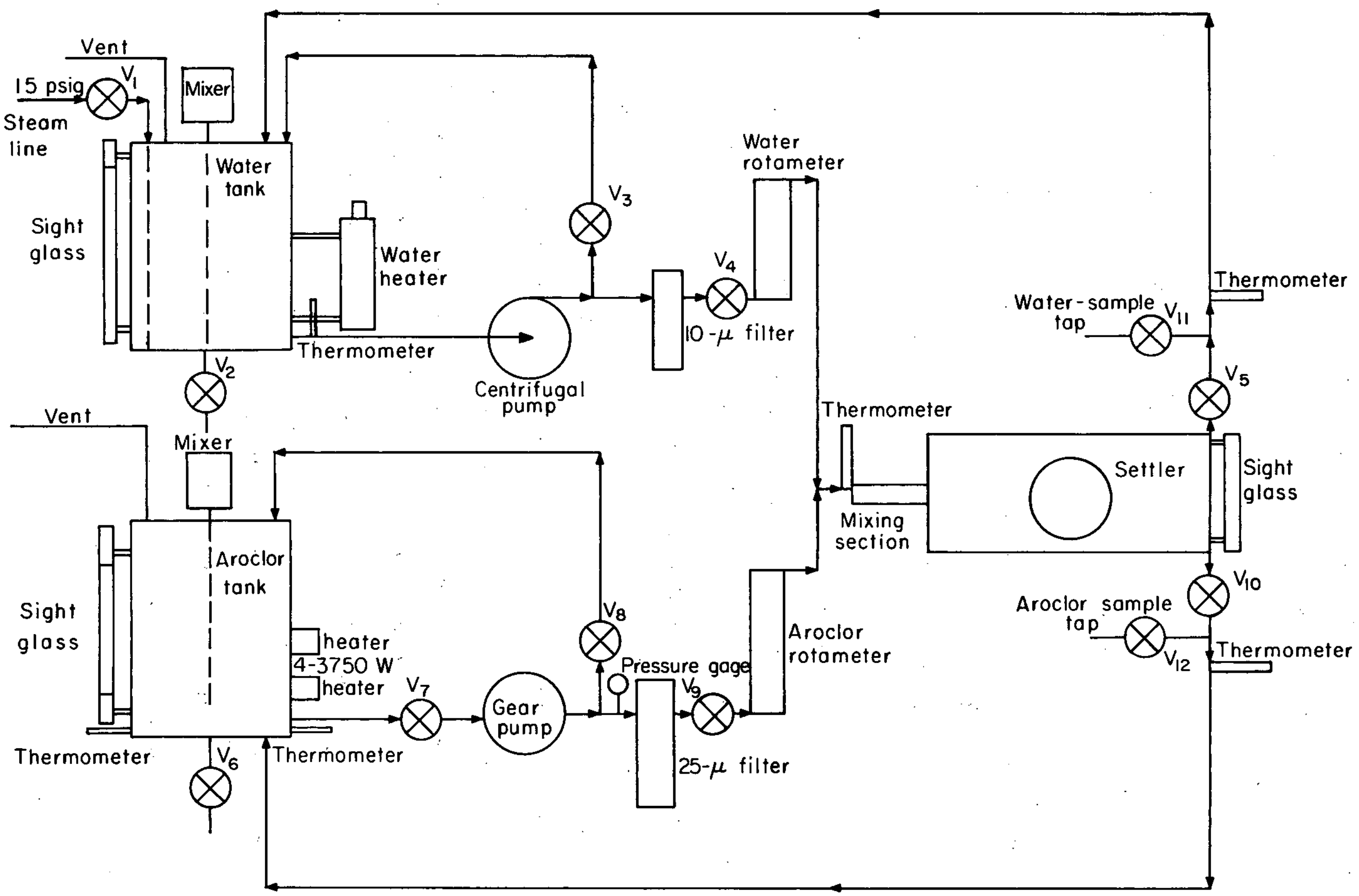

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Fig. 4. Schematic diagram of settler apparatus.

C. Mixing and Settling System

1. Mixing Pipes

The Aroclor and water streams met at a 3/4-in. tee and were conveyed through a short section of 3/4-in. tubing to the mixing pipe, which consisted of four 1/4-in.-i.d. × 12-in.-long copper tubes encased in a 1 × 6-in. brass nipple. The liquid passed through the 1/4-in. tubes and into the settler. Metal plugs were used to block off the tubes as required to give the desired Reynolds number for a given flow rate. The 6-in. mixing pipe was connected to any of the 1 × 5-in. nipples on the settler by means of a coupling.

2. The Two-Foot Settler

The settler was a 2-ft-long aluminum chamber with a rectangular cross section 12-in. high × 6-in. wide. A 9-1/8-in.-diam window was mounted in one side, midway from the ends, for observing the flow.

The inlets to the settler are shown in Fig. 5. Five 1 × 5-in. nipples were mounted in the front plate 2, 4, 6, 8, and 10 in. from the settler bottom. One 5 × 1-in. nipple was mounted in the side at the 10-in. level. The mixing pipe is shown attached to the inlet at the 10-in. level on the front plate.

A baffle 2 in. high and 4 in. wide was mounted in front of the inlet to break up the incoming jet of Aroclor-water mixture. The baffle was mounted on two 6-in. tie rods threaded into the front plate on either side of the inlet nipple. The baffle could be mounted before any of the front inlets and could be placed at any distance from the inlet on the tie rods. No baffle was used on the side entrance.

Four 1-1/4-in. pipes were welded to the back of the settler as Aroclor outlets. However, only the bottom one was used. The purpose of this large outlet was to reduce turbulence at the Aroclor outlet. The water was withdrawn from a 3/4-in. outlet on top of the end of the settler.

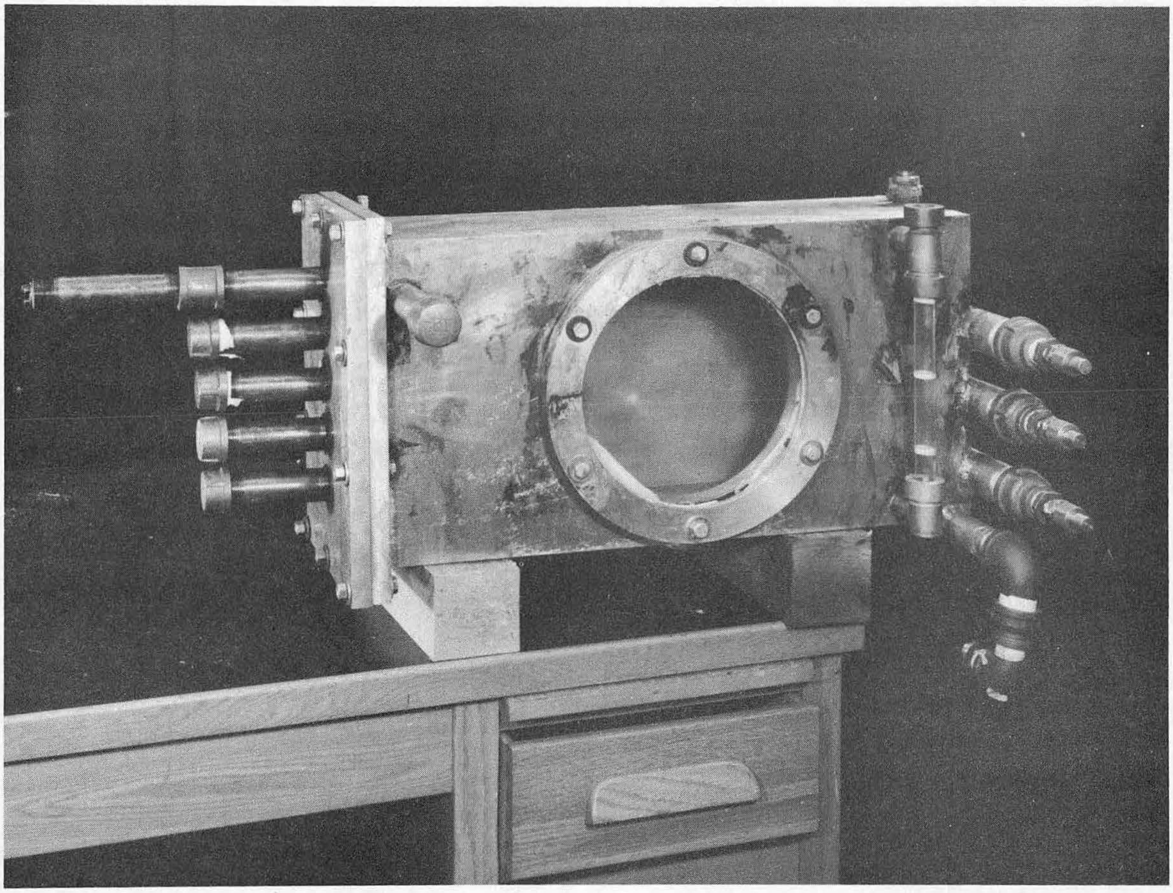

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Fig. 5. Two-foot-long gravity settler. The mixing pipe is shown attached to the inlet at the 10-in. level.

The position of the interface, observed through a sight glass mounted at the end of the settler, was controlled by means of needle valves on the Aroclor- and water-outlet lines.

Two 576-w 6-ft-long heating tapes, controlled by rheostats, were used to keep the settler at constant temperature. The tapes were wrapped at 4-in. intervals around the outside of the settler, which was also wrapped in 1/2-in. -thick Fiberglas insulation.

3. The Four-Foot Settler

The 4-ft settler consisted of the 2-ft settler with the front plate removed, attached to another 2-ft section as shown in Fig. 6. Inlets and baffle were similar to the ones on the 2-ft settler, except that five side entrances were also provided. Insulation, heating tapes, and other parts were installed in the same manner as for the 2-ft settler.

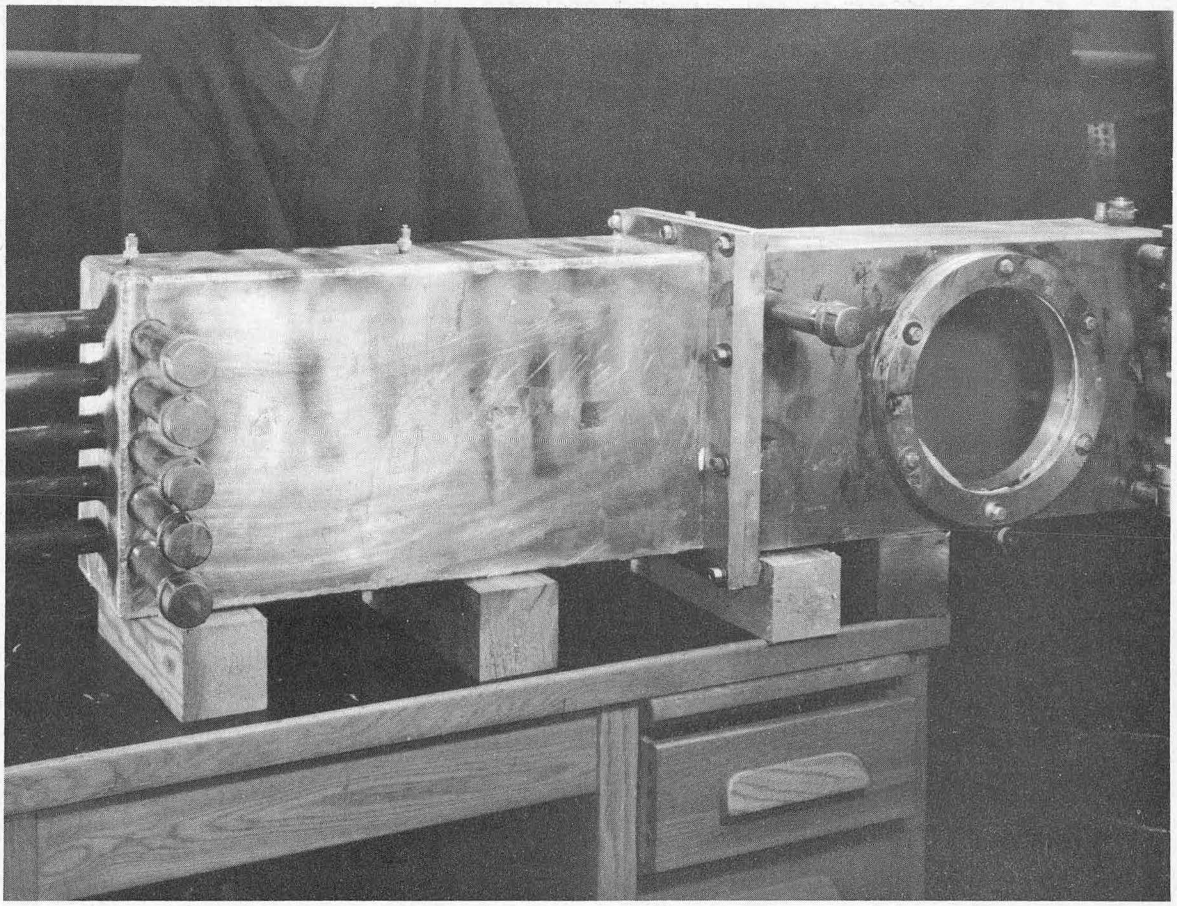

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Fig. 6. Four-foot-long gravity settler.

V. OPERATING PROCEDURE

The baffle was placed at the desired position and the front plate, or the front 2-ft section, was bolted to the end of the 2-ft settler section. The mixing pipe was then connected to the proper inlet nipple.

The Aroclor was heated to 220 to 240° F, and the water was heated to the required temperature by opening the steam valve V_1. The water pump was started and the settler was filled with water. The Aroclor outlet valve V_{10} was closed while the water valve V_5 remained open.

When the settler was filled, the water was shut off by closing valve V_4. Valves V_7 and V_8 were opened and the Aroclor pump was started. Valve V_9 was opened and the Aroclor flow rate was adjusted by valves V_8 and V_9. When the Aroclor in the settler reached a depth of 6 in. Aroclor outlet valve V_{10} was opened and the water-outlet valve V_5 was closed.

When the temperature of the effluent Aroclor from the settler reached 200° F, valves V_4 and V_5 were opened, and the water was introduced at the proper flow rate. The flow rates and interface positions were then adjusted to the desired values. The inlet temperature was adjusted to 200° F by adjusting the temperature of the Aroclor tank with the Aroclor heaters. The heating tapes were turned on to keep the settler at 200° F. Thirty minutes after the inlet streams reached thermal equilibrium at $200 \pm 2^\circ$ F, a sample was taken at valves $V_{12}$and V_{11}.

Samples were taken at various flow rates, initial water concentrations, Reynolds numbers in the mixing sections, inlet heights, baffle positions, and Aroclor depths, and analyzed as described in Appendices A and B.

The Aroclor tank was kept above 220° F, so that nearly all the insoluble water carried over in the effluent Aroclor was evaporated. This prevented water from accumulating in the Aroclor tank. Except when the N_{RE} in the mixing section was 5320 or 5900, no Aroclor was carried over into the water tank. Even when the N_{RE} was 5320 or 5900, the Aroclor carry-over was less than 0.1%, and the settler was operated at these conditions for less than 45 min. The loss of Aroclor to the water tank was rendered negligible by this procedure.

The experimental data and derived results for the separation of water from Aroclor are tabulated in Table II, for a temperature of 200° F. The experimental data for the separation of Aroclor from water are listed in Table III. The elements of these tables provide the basis for the detailed discussion in Section VII below.

Table II. Experimental data and derived results for the separation of water from Aroclor 1248 (temperature, 200° F for all data).

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec$\times 10^3$)	Z_A (ft)	L (ft)	V_o (ft/sec$\times 10^3$)	Z_I (in.)	X_b (in.)	N_{RE}	C_W (weight %)
A-1(a)	0.291	15.2	12.9	0.75	2	4.85	1	3.5	2950	0.283
A-1(b)	0.291	15.2	12.9	0.75	2	4.85	1	3.5	2950	0.287
A-2(a)	0.291	15.2	19.4	0.50	2	4.85	4	3.5	2950	0.290
A-2(b)	0.291	15.2	19.4	0.50	2	4.85	4	3.5	2950	0.296
A-3(a)	0.291	15.2	19.4	0.50	2	4.85	2	3.5	2950	0.304
A-3(b)	0.291	15.2	19.4	0.50	2	4.85	2	3.5	2950	0.308
A-3(c)	0.291	15.2	19.4	0.50	2	4.85	2	3.5	2950	0.303
A-4(a)	0.291	15.2	38.8	0.25	2	4.85	5	3.5	2950	0.302
A-4(b)	0.291	15.2	38.8	0.25	2	4.85	5	3.5	2950	0.346
A-4(c)	0.291	15.2	38.8	0.25	2	4.85	5	3.5	2950	0.387
A-4(d)	0.291	15.2	38.8	0.25	2	4.85	5	3.5	2950	0.338
A-5(a)	0.291	15.2	38.8	0.25	2	4.85	3	3.5	2950	0.467
A-5(b)	0.291	15.2	38.8	0.25	2	4.85	3	3.5	2950	0.437
A-6	0.291	15.2	38.8	0.25	2	4.85	1	3.5	2950	0.897

Table II. (continued)

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A ($ft/sec \times 10^3$)	Z_A (ft)	L (ft)	V_o ($ft/sec \times 10^3$)	Z_I (in.)	X_b (in.)	N_R	C_W (weight %)
B-1	0.194	15.2	8.64	0.75	2	3.24	1	3.5	2950	0.103
B-2	0.194	15.2	12.9	0.50	2	3.24	4	3.5	2950	0.113
B-3(a)	0.194	15.2	12.9	0.50	2	3.24	2	3.5	2950	0.108
B-3(b)	0.194	15.2	12.9	0.50	2	3.24	2	3.5	2950	0.113
B-4(a)	0.194	15.2	25.9	0.25	2	3.24	5	3.5	2950	0.115
B-4(6)	0.194	15.2	25.9	0.25	2	3.24	5	3.5	2950	0.113
B-5(a)	0.194	15.2	25.9	0.25	2	3.24	3	3.5	2950	0.137
B-5(b)	0.194	15.2	25.9	0.25	2	3.24	3	3.5	2950	0.132
B-6	0.194	15.2	25.9	0.25	2	3.24	1	3.5	2950	0.169
C-1	0.097	15.2	4.31	0.75	2	1.62	1	3.5	2950	0.070
C-2(a)	0.097	15.2	6.46	0.50	2	1.62	2	3.5	2950	0.054
C-2(b)	0.097	15.2	6.46	0.50	2	1.62	2	3.5	2950	0.060
C-3	0.097	15.2	12.9	0.25	2	1.62	5	3.5	2950	0.059

Table II. (continued)

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec×10^3)	Z_A (ft)	L (ft)	V_o (ft/sec×10^3)	Z_I (in.)	X_b (in.)	N_R	C_W (weight %)
D-1	0.291	5.4	12.9	0.75	2	4.85	1	3.5	2660	0.165
D-2	0.291	5.4	19.4	0.50	2	4.85	4	3.5	2660	0.181
D-3(a)	0.291	5.4	19.4	0.50	2	4.85	2	3.5	2660	0.152
D-3(b)	0.291	5.4	19.4	0.50	2	4.85	2	3.5	2660	0.169
D-3(c)	0.291	5.4	19.4	0.50	2	4.85	2	3.5	2660	0.164
D-3(d)	0.291	5.4	19.4	0.50	2	4.85	2	3.5	2660	0.157
D-4(a)	0.291	5.4	38.8	0.25	2	4.85	5	3.5	2660	0.149
D-4(b)	0.291	5.4	38.8	0.25	2	4.85	5	3.5	2660	0.145
D-5(a)	0.291	5.4	38.8	0.25	2	4.85	3	3.5	2660	0.267
D-5(b)	0.291	5.4	38.8	0.25	2	4.85	3	3.5	2660	0.297
D-6	0.291	5.4	38.8	0.25	2	4.85	1	3.5	2660	0.369
E-1(a)	0.194	5.4	8.64	0.75	2	3.24	1	3.5	2660	0.088
E-1(b)	0.194	5.4	8.64	0.75	2	3.24	1	3.5	2660	0.080
E-2	0.194	5.4	12.9	0.50	2	3.24	4	3.5	2660	0.077
E-3(a)	0.194	5.4	12.9	0.50	2	3.24	2	3.5	2660	0.070
E-3(b)	0.194	5.4	12.9	0.50	2	3.24	2	3.5	2660	0.078
E-4(a)	0.194	5.4	25.9	0.25	2	3.24	5	3.5	2660	0.078
E-4(b)	0.194	5.4	25.9	0.25	2	3.24	5	3.5	2660	0.075
E-5	0.194	5.4	25.9	0.25	2	3.24	3	3.5	2660	0.075
E-6	0.194	5.4	25.9	0.25	2	3.24	1	3.5	2660	0.108

Table II. (continued)

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec$\times 10^3$)	Z_A (ft)	L (ft)	V_o (ft/sec$\times 10^3$)	Z_I (in.)	X_b (in.)	N_R	C_W (weight %)
F-1	0.097	5.4	4.31	0.75	2	1.62	1	3.5	2660	0.040
F-2(a)	0.097	5.4	6.46	0.50	2	1.62	2	3.5	2660	0.043
F-2(b)	0.097	5.4	6.46	0.50	2	1.62	2	3.5	2660	0.043
F-3	0.097	5.4	12.9	0.25	2	1.62	5	3.5	2660	0.043
G-1	0.291	15.2	12.9	0.75	4	2.43	1	3.5	2950	0.180
G-2	0.291	15.2	19.4	0.50	4	2.43	4	3.5	2950	0.199
G-3	0.291	15.2	38.8	0.25	4	2.43	3	3.5	2950	0.257
H-1	0.194	15.2	8.64	0.75	4	1.62	1	3.5	2950	0.080
H-2	0.194	15.2	12.9	0.50	4	1.62	4	3.5	2950	0.077
H-3	0.194	15.2	25.9	0.25	4	1.62	3	3.5	2950	0.084
I-1	0.097	15.2	4.31	0.75	4	0.81	1	3.5	2950	0.054
I-2	0.097	15.2	6.46	0.50	4	0.81	4	3.5	2950	0.051
J-1	0.291	5.4	12.9	0.75	4	2.43	1	3.5	2660	0.113
J-2	0.291	5.4	19.4	0.50	4	2.43	4	3.5	2660	0.121
J-3	0.291	5.4	38.8	0.25	4	2.43	3	3.5	2660	0.127

Table II. (continued)

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec×10^3)	Z_A (ft)	L (ft)	V_o (ft/sec×10^3)	Z_I (in.)	X_b (in.)	N_R	C_W (weight %)
K-1	0.194	5.4	8.64	0.75	4	1.62	1	3.5	2660	0.060
K-2	0.194	5.4	12.9	0.50	4	1.62	4	3.5	2660	0.060
K-3	0.194	5.4	25.9	0.25	4	1.62	3	3.5	2660	0.058
L-1	0.097	5.4	4.31	0.75	4	0.81	1	3.5	2660	0.036
L-2	0.097	5.4	6.46	0.50	4	0.81	4	3.5	2660	0.037
M-1	0.194	5.4	25.9	0.25	2	3.24	-1	3.5	5320	1.12
M-2	0.194	15.2	25.9	0.25	2	3.24	-1	3.5	5900	1.72
N-1	0.194	5.4	25.9	0.25	2	3.24	5	3.5	5320	0.166
N-2	0.194	15.2	25.9	0.25	2	3.24	5	3.5	5900	0.359
N-3	0.194	15.2	12.9	0.50	2	3.24	4	3.5	5900	0.337
N-4	0.194	15.2	12.9	0.50	4	1.62	4	3.5	5900	0.191
N-5	0.194	5.4	12.9	0.50	2	3.24	4	3.5	5320	0.173
N-6	0.194	5.4	12.9	0.50	4	1.62	4	3.5	5320	0.089

Table II. (continued)

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec$\times 10^3$)	Z_A (ft)	L (ft)	V_o (ft/sec$\times 10^3$)	Z_I (in.)	X_b (in.)	N_R	C_W (weight %)
O-1	0.194	5.4	12.9	0.50	2	3.24	4	1	2660	0.135
O-2	0.194	15.2	12.9	0.50	2	3.24	4	1	2950	0.196
O-3	0.291	5.4	19.4	0.50	2	4.85	4	1	2660	0.275
O-4	0.291	15.2	19.4	0.50	2	4.85	4	1	2950	0.416
P-1	0.194	5.4	12.9	0.50	2	3.24	4	no baffle	2660	0.084
P-2	0.194	15.2	12.9	0.50	2	3.24	4	"	2950	0.133
P-3	0.291	5.4	19.4	0.50	2	4.85	4	"	2660	0.169
P-4	0.291	15.2	19.4	0.50	2	4.85	4	"	2950	0.319

Table III. Experimental data for the separation of Aroclor 1248 from water.

Run No.	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	Z_A (ft)	L (ft)	Z_I (in.)	X_b (in.)	N_{RE}	Aroclor concentration in water effluent (weight %)
Q-1	0.194	5.4	0.50	2	4	3.5	2660	0.00
Q-2	0.194	15.2	0.50	2	4	3.5	2950	0.00
Q-3	0.194	5.4	0.50	2	4	3.5	5320	0.042
Q-4	0.194	15.2	0.50	2	4	3.5	5900	0.098

VI. OBSERVATIONS

As the water and Aroclor flowed through the entrance pipe to the settler, turbulent mixing occurred. Further mixing took place at the entrance of the settler when the incoming jet of Aroclor and water passed through the water layer and struck the baffle plate. The mixing produced an emulsion layer of water and Aroclor droplets.

The emulsion layer was also partically formed by droplets of water trapped in the Aroclor layer by mixing. These droplets moved upward through the Aroclor and collected at the interface. Aroclor droplets trapped in the water layer behaved in a similar manner. These droplets did not coalesce immediately, but remained at the interface for a finite time. This behavior was consistent with the observations by Lang et al., as described in Section II-C. The emulsion layer could be compared to the mixture layer of Meissner and Chertow, or the foam described by Putney, referred in Section II-A.

The emulsion layer appeared to consist of clusters of Aroclor droplets surrounded by a thin film of water, and clusters of water droplets surrounded by thin films of Aroclor. Being less dense than the Aroclor layer, but denser than the water layer, the emulsion layer collected at the Aroclor-water interface.

The emulsion layer droplets moved along the interface at velocities of up to several inches per second. As they moved along the interface, droplets of water and Aroclor combined to form bigger droplets, or else collapsed into their respective layers. This caused the droplet concentration of the emulsion layer to decrease toward the far end of the settler: dropping from a high concentration at the entrance to a low concentration at the far end of the settler, producing the high emulsion droplet velocities observed.

The emulsion layer was observed through the circular window located 12 in. from the back end of both the 2-ft and 4-ft settlers. The nature of this layer varied considerably with flow rate, the Reynolds number in the entrance pipe, initial water concentration, the length of settler, and to some extent with the distance of the inlet above the interface.

The photograph shows a thick emulsion layer, in Fig. 7. This layer was composed of clusters of small water bubbles and clusters of large Aroclor bubbles. The emulsion droplets moved along the interface at velocities of several in. per sec at the center, and somewhat lower near the sides.

The Aroclor layer had a milky white appearance because of the small water droplets dispersed in it. The water layer was clear except for a few small Aroclor droplets that settled rapidly down to the interface.

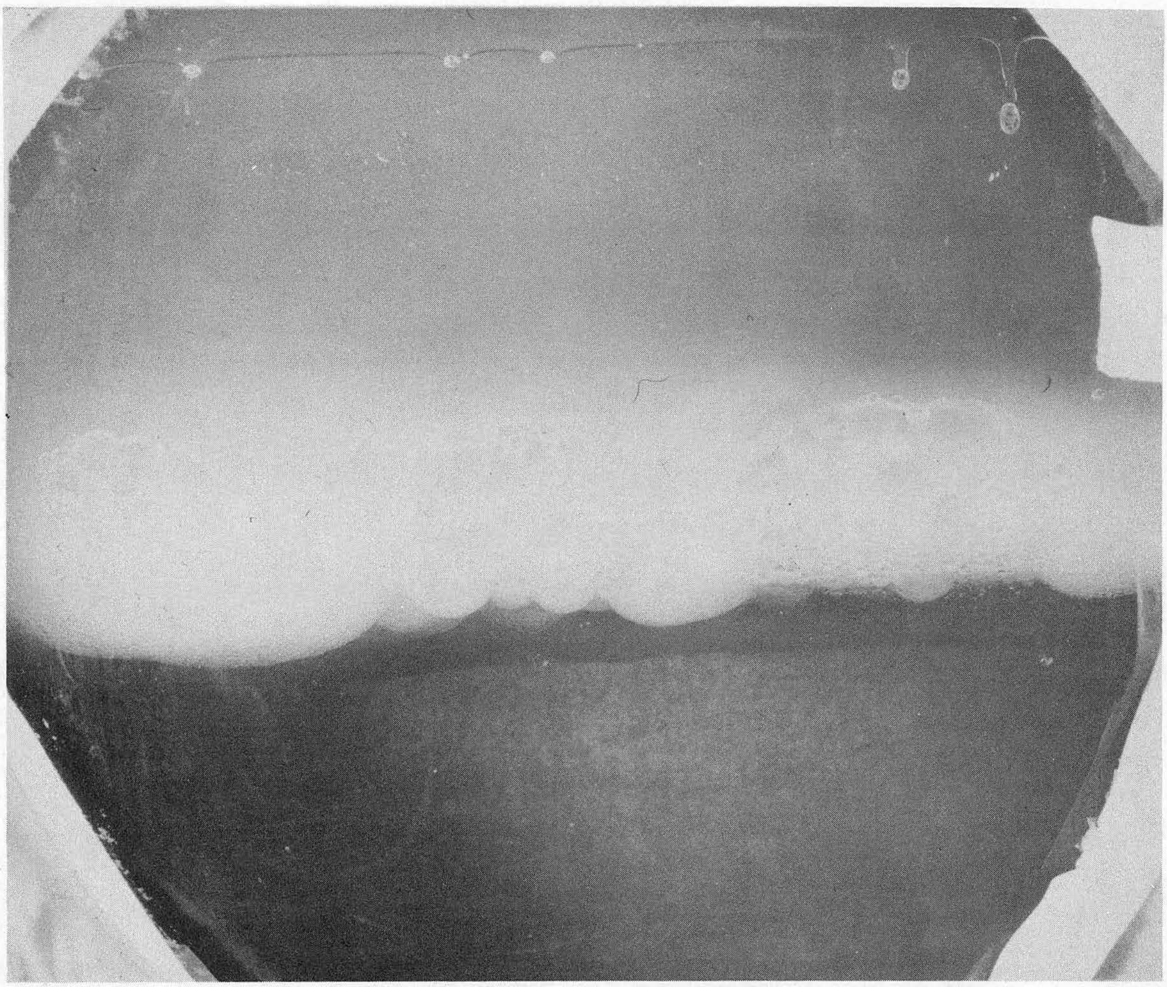
Coalescence and collapse of droplets in the emulsion sent showers of tiny water droplets swirling down into the Aroclor layer. Some droplets from the showers penetrated several inches down into the Aroclor layer. Most, however, penetrated less than an inch, and this accounts for the whitish cloud seen cloud seen just below the emulsion layer in Fig. 7.

At high Q_A' s, the composition of the emulsion layer seemed to vary with the height of the inlet above the Aroclor-water interface, Z_I. When the Z_I was an inch or less, the emulsion layer was composed mainly of small water droplets, and the boundary between the emulsion and Aroclor layers was poorly defined. At these Z_I' s the incoming jet was injected into the emulsion layer, trapping many water droplets in both the emulsion and Aroclor layers.

At larger Z_I' s, more clusters of Aroclor droplets appeared in the emulsion layer. When the incoming jet was injected into the water layer several inches above the interface, the Aroclor in the jet broke up into droplets, which accounted for the Aroclor droplets observed in the emulsion layer. Also, fewer water droplets were observed in the emulsion and Aroclor layers as the Z_I was increased.

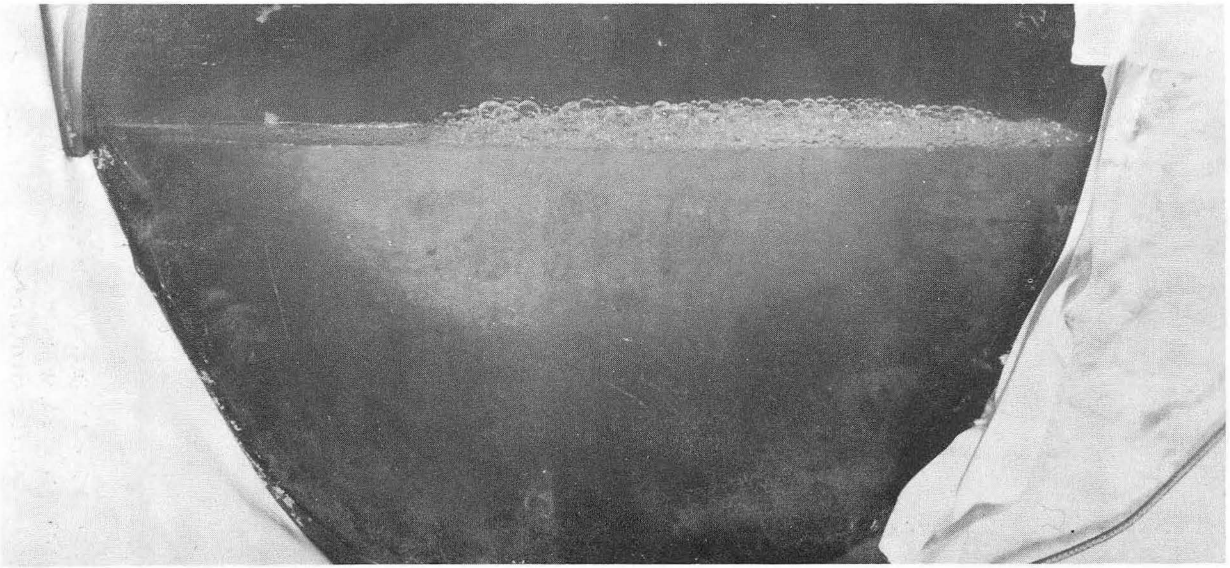
At lower $C_{W(0)}$, the emulsion layer was less thick, the Aroclor layer became clearer and fewer water droplets were observed in it.

Lower Q_A' s produced less emulsion. A photograph of the emulsion layer at a low Q_A is shown as Fig. 8. The thinner emulsion layer was composed mainly of clusters of large Aroclor droplets.



ZN-3088

Fig. 7. Emulsion layer as described in the text, in which Q_A = 0.291 ft^3/min, initial water concentration = 15.2%, L = 2 ft, Z_A = 6 in., baffle distance = 3-1/2 in., front entrance temp = 200° F, and N_{RE} = 2950.



ZN-3089

Fig. 8. Emulsion layer, in which Q_A = 0.097 ft^3/min, initial water concentration = 5.4%, L = 2 ft, baffle distance = 3-1/2 in., and front entrance temp = 200° F.

The Aroclor layer was only slightly clouded with water droplets, and no Aroclor droplets were seen in the water layer.

Higher values of the N_{RE} in the entrance pipe produced more emulsion. At an N_{RE} of 5900 the emulsion layer was 1-1/2 in. thick, what at 2950 the emulsion thickness was only 1 in. thick. At higher N_{RE} values the water layer was clouded with Aroclor droplets and the Aroclor layer contained more water droplets.

The emulsion layer was less thick at a point 3 ft downstream from the entrance than at 1 ft from the entrance, owing to the collapse and coalescence of droplets in the emulsion layer. At the lowest flow rate, no emulsion layer was visible 3 ft from the entrance of the 4-ft settler.

VII. DISCUSSION OF RESULTS

A. Distance of Feed Inlet above Interface (Z_I)

Figure 9 shows the effect of Z_I on C_W at $Z_A = 0.25$ ft. The data are shown in Table IV.

This figure and table also show that at the highest Q_A (0.291 ft^3/min) and $C_{W(0)}$ (15.2%), C_W decreased rapidly with increasing Z_I. Under these conditions, and for $Z_I = 1$ in, C_W was 2.6 times as great as C_W at $Z_I = 5$ in. Also, in the observations of the emulsion layer for high Q_A's, it was noted that the composition of the emulsion layer varied with Z_I. That is, at low Z_I's, the observed emulsion and Aroclor layers contained more water droplets than at high Z_I's, and the data indicate that more water droplets were dispersed into the Aroclor layer at low Z_I's.

For discussion purposes, consider the settler (see Fig. 2) to be divided into three zones: the inlet, outlet, and settling zones. The inlet zone consists of the first section of the settler between the feed inlet and the baffle. The outlet zone consists of the last inch of the settler directly above the Aroclor outlet. The section of the settler between the inlet and outlet zones is the settling zone.

Now, to explain the effect of Z_I on C_W at $Z_A = 0.25$ ft, consider the inlet zone. The Aroclor-water feed stream was injected into the water layer of the settler as a dispersion of water droplets in Aroclor. As expected from the observations by Ohnesorge and Richardson, the Aroclor in the incoming feed stream broke up into droplets which were deflected by the baffle and settled at the Aroclor-water interface. The majority of the droplets coalesced as they collided with each other, or when they reached the Aroclor layer. Some, however, did not coalesce immediately at the interface. These droplets became part of the emulsion described in Section VII.

The Aroclor droplets of the feed stream contained dispersed water droplets. As the Aroclor droplets descended through the water layer to the interface, water droplets escaped from their surfaces

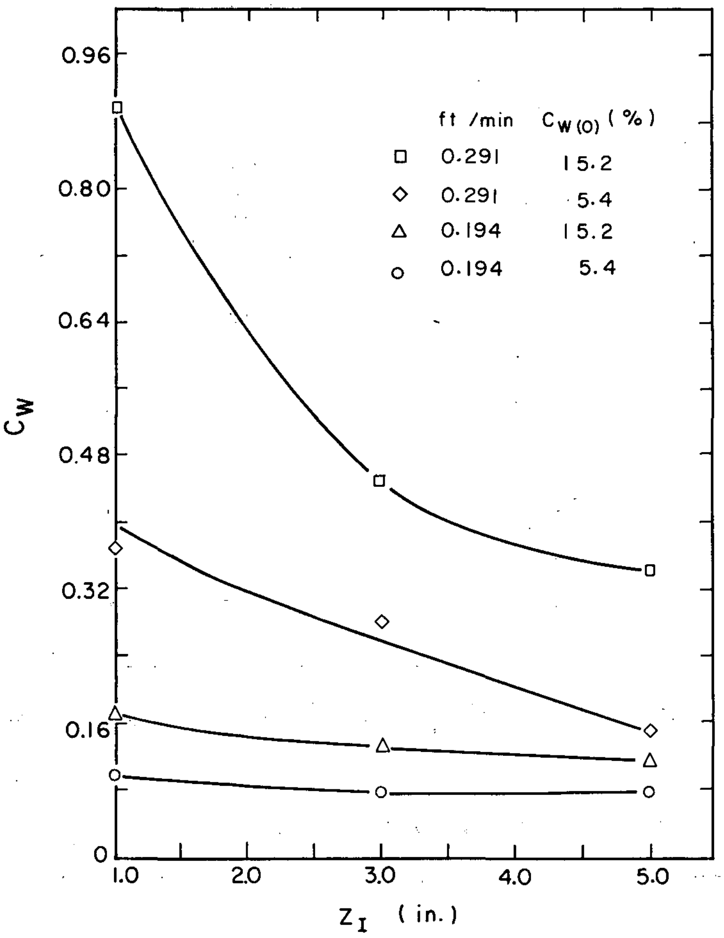


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Fig. 9. C_W vs Z_I.

Table IV. The effect of Z_I on C_W at various values of Q_A and $C_{W(0)}$ for Z_A = 0.25 ft.[a]

Run No.	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	Z_I (in.)	C_W[b] (weight %)
A-4	0.291	15.2	5	0.343
A-5	0.291	15.2	3	0.450
A-6	0.291	15.2	1	0.897
B-4	0.194	15.2	5	0.114
B-5	0.194	15.2	3	0.134
B-6	0.194	15.2	1	0.169
D-4	0.291	5.4	5	0.147
D-5	0.291	5.4	3	0.282
D-6	0.291	5.4	1	0.369
E-4	0.194	5.4	5	0.074
E-5	0.194	5.4	3	0.075
E-6	0.194	5.4	1	0.098

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[a] For all runs shown, N_{RE} = 2800 to 2950, Z_A = 0.25 in., L = 2 ft, and X_b = 3.5 in.

[b] Average values of duplicate runs.

and coalesced into the water layer. Since the time required to reach the interface was very short, only large water droplets near the surface of the Aroclor droplets were able to coalesce in this manner.

At the highest Z_I, the Aroclor droplets had farther to travel to reach the interface than at lower Z_I's. This allowed more water droplets to escape from the Aroclor droplets at high Z_I's, which would explain the lower values of C_W at high Z_I's.

Another factor that would account for more water droplets' being dispersed in the Aroclor layer at low Z_I's was the turbulence created by the incoming feedstream. At low Z_I's, the feedstream was injected into the water layer close to the Aroclor layer, and the turbulence created in the latter tended to entrain water droplets in the Aroclor at low Z_I's.

Since decreasing Z_I at the highest Q_A with $Z_A = 0.25$ ft increased C_W, one might expect a similar result at $Z_A = 0.50$ ft. However, the data in Table II show that this was not the case. Comparing data A-2 with A-3 shows that decreasing Z_I from 4 in. to 2 in. had little effect on C_W. However, at $Z_A = 0.50$ ft as at $Z_A = 0.25$ ft, the composition of the emulsion layer varied with Z_I. At $Z_A = 0.50$ ft, as Z_I was decreased more water droplets appeared in both the emulsion and Aroclor layers. This was likewise the case at $Z_A = 0.25$ ft.

These seemingly contradictory data and observations can be explained by considering the outlet zone. From consideration of the flow pattern around the Aroclor outlet, the downward vertical component of Aroclor velocity above the Aroclor outlet would be expected to decrease with distance above the Aroclor outlet. Consider, for instance, a point located at a given distance (i. e., 1 in.) below the emulsion layer and directly above the Aroclor outlet. For a given value of Q_A, the downward vertical component of the Aroclor velocity at this point ought to be greater at $Z_A = 0.25$ ft than at $Z_A = 0.50$ ft.

However, a water droplet at this point is acted upon by a buoyancy force and a downward-drag force of the Aroclor as it flows downward toward its outlet. If the buoyancy force is the larger, the droplet will move up to the interface and coalesce. If the drag force is the larger, the droplet will be entrained in the effluent Aroclor. Since the downward velocity of the Aroclor at this point is greater at $Z_A = 0.025$ ft than at $Z_A = 0.50$ ft, larger droplets will be entrained in the effluent Aroclor at $Z_A = 0.25$ ft for a given Q_A.

The water droplets dispersed into the Aroclor by lowering the Z_I are large enough so that, at $Z_A = 0.50$ ft, their buoyancy forces are sufficient to overcome the drag force of the downward-flowing Aroclor above its outlet. This would explain why Z_I had little effect on C_W at $Z_A = 0.50$ ft. At $Z_A = 0.25$ ft, however, the downward velocity of the Aroclor above its outlet was large enough to entrain additional droplets dispersed because of the lowering Z_I. This resulted in a variance of C_W, with Z_I at $Z_A = 0.25$ ft at the highest Q_A.

Again, Fig. 9 and Table IV show that at the lower Q_A (0.194 ft^3/min) C_W was not greatly affected by Z_I. More water droplets were dispersed in the emulsion and Aroclor layers at low Z_I's than at high Z_I's, as was the case at $Q_A = 0.291$ ft^3/min. However, at the lower Q_A, the smaller value of V_o and lower downward-moving Aroclor velocities above the Aroclor outlet allowed more of these droplets to reach the interface and coalesce. This reduced the effect of Z_I on C_W at the lower values of Q_A.

For all the data discussed up to this point, the feed stream was injected into the water layer. The effect of injecting the feed stream into the Aroclor layer is shown in Table V. Injecting the feed into the Aroclor layer resulted in C_W values roughly seven times as great as corresponding values of C_W for injecting the feed into the water layer.

These results can be explained by considering conditions in the inlet zone. When the feed stream was injected into the water layer, the Aroclor in the feed stream broke up into droplets. Some of the water droplets dispersed in the Aroclor droplets escaped into the water layer as the Aroclor droplets descended to the interface. This separation process did not occur when the feed stream was injected into the Aroclor layer. Also, when the feed stream was injected into the Aroclor layer, turbulence caused by the incoming feed stream caused additional mixing of the water droplets in the Aroclor in the inlet zone.

Table V. Effect of injecting the feed stream below the Aroclor-water interface.

For all runs shown:

N_{RE} = 2660 to 2950
Q_A = 0.194 ft^3/min
Z_A = 0.25 ft
L = 2 ft
X_b = 3.5 in.
V_o = 3.24×10^{-3} ft/sec

Run No.	$C_{W(0)}$ (weight %)	Z_I (in.)	C_W (weight %)
N-1	5.4	5	0.166
M-1	5.4	-1	1.12
N-1	15.2	5	0.359
M-2	15.2	-1	2.74

B. Overflow Velocity (V_o)

Figure 10 shows C_W plotted vs V_o from data shown in Table VI, Figure 10 and Table VI show the following

(a) for a given value of V_o, L, and $C_{W(0)}$, the values of C_W obtained were fairly consistent;

(b) the slopes of the C_W-vs-V_o curves increased rapidly with increasing V_o;

(c) for a given V_o and $C_{W(0)}$, C_W was lower at L = 2 ft than for C_W at L = 4 ft;

(d) the curves of C_W vs V_o did not approach the origin as V_o approached 0, but seemed to level out at approx C_W = 0.04%.

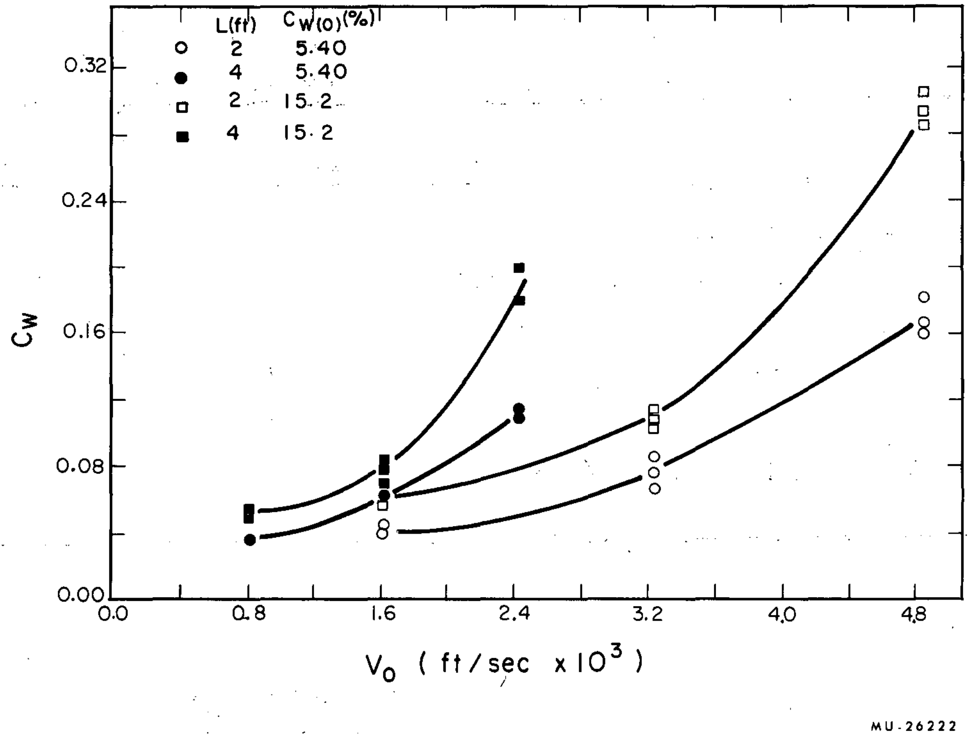

Fig. 10. C_W vs V_o.

Table VI. The effect of V_o on C_W for ranges of values of $C_{W(0)}$, Z_A, and L.

Run No.[a]	V_o (ft/sec × 10^3)	$C_{W(0)}$ (weight %)	Z_A (ft)	L (ft)	C_W[b] (weight %)
A-1	4.85	15.2	0.75	2	0.285
A-2	4.85	15.2	0.50	2	0.293
A-3	4.85	15.2	0.50	2	0.305
B-1	3.24	15.2	0.75	2	0.103
B-2	3.24	15.2	0.50	2	0.113
B-3	3.24	15.2	0.50	2	0.111
C-1	1.62	15.2	0.75	2	0.070
C-2	1.62	15.2	0.50	2	0.057
C-3	1.62	15.2	0.25	2	0.059
D-1	4.85	5.4	0.75	2	0.165
D-2	4.85	5.4	0.50	2	0.181
D-3	4.85	5.4	0.50	2	0.160
E-1	3.24	5.4	0.75	2	0.084
E-2	3.24	5.4	0.50	2	0.067
E-3	3.24	5.4	0.50	2	0.076
F-1	1.62	5.4	0.75	2	0.040
F-2	1.62	5.4	0.50	2	0.043
F-3	1.62	5.4	0.25	2	0.043
G-1	2.43	15.2	0.75	4	0.180
G-2	2.43	15.2	0.50	4	0.199
H-1	1.62	15.2	0.75	4	0.080
H-2	1.62	15.2	0.50	4	0.077
H-3	1.62	15.2	0.25	4	0.084
I-1	0.81	15.2	0.75	4	0.054
I-2	0.81	15.2	0.50	4	0.051
I-3	0.81	15.2	0.25	4	0.055

Table VI. (continued)

Run No.[a]	V_o (ft/sec × 10^3)	$C_{W(0)}$ (weight %)	Z_A (ft)	L (ft)	C_W[b] (weight %)
J-1	2.43	5.4	0.75	4	0.113
J-2	2.43	5.4	0.50	4	0.111
K-1	1.62	5.4	0.75	4	0.060
K-2	1.62	5.4	0.50	4	0.060
K-3	1.62	5.4	0.25	4	0.058
L-1	0.81	5.4	0.75	4	0.036
L-2	0.81	5.4	0.50	4	0.037
L-3	0.81	5.4	0.25	4	0.039

[a] For all runs shown: N_{RE} = 2660 to 2950, and X_b = 3.5 in.

[b] These are average values of C_W for duplicate runs.

These results can be explained by considering the behavior of the water droplets and the emulsion layer in the settling zone. In the settling zone, water droplets dispersed in the Aroclor rose toward the interface at various rising velocities U_∞, depending on the droplet size. Whether or not a given droplet reached the interface depended on (a) its depth in the Aroclor, (b) the horizontal velocity of the Aroclor, (c) the length of the settler, and (d) the rising velocity of the droplet. From the discussion of design methods based on V_o, one would expect consistent values of C_W for a given V_o. The data in Table VI show that this was indeed true, provided that L and $C_{W(0)}$ were also constant. Also, values of C_W obtained at $Z_A = 0.25$ ft with $V_o \geqslant 2.43 \times 10^3$ ft/sec were omitted from the data in Table VI for reasons discussed in Section VII-C below.

In the observations of the emulsion layer in the settling zone, it was noted for the highest Q_A that collapsing and coalescing droplets in the emulsion layer sent showers of water droplets into the Aroclor layer. Most of these water droplets penetrated less than an inch into the Aroclor layer and were observed as a whitish cloud just below the emulsion layer already refered to. At lower values of Q_A, the emulsion layer contained fewer droplets, fewer droplets were showered into the Aroclor layer from coalescing droplets, and the cloudy layer was not observed.

These observations provide part of the explanation of the rapidly increasing slope of C_W-vs-V_o curves. At higher Q_A's (i.e., high V_o's) more droplets of water were showered into the Aroclor layer from coalescing droplets in the emulsion layer. This would increase C_W at high V_o's.

Also, increasing V_o increases the U_∞ required of a droplet to reach the interface. Since U_∞ is proportional to the second power of the droplet radius, increasing V_o would increase the droplet size required of a droplet to reach the interface. This would result in more droplets' being entrained in the effluent Aroclor at large values of V_o.

A third factor that would account for the rapid increase of the C_W-vs-V_o curve is the downward velocity of the Aroclor in the outlet zone. As Q_A (i. e., V_o) was increased during the experiment the downward velocity of the Aroclor above the Aroclor outlet was increased, and, therefore, larger water droplets were entrained in the effluent Aroclor.

For a given V_o and $C_{W(0)}$, C_W was lower at L = 2 ft than at L = 4 ft. Figure 10 illustrates this clearly by showing that the curves of C_W vs V_o obtained for L = 2 ft do not match up with curves obtained for L = 4 ft. To obtain the same value of V_o, the value of Q_A at L = 4 ft must be twice as great as Q_A at L = 2 ft. At higher Q_A's required for L = 4 ft, the emulsion layer was thicker and more water droplets were showered into the Aroclor layer from collapsing and coalescing droplets in the emulsion layer than at the lower flow rates at L = 2 ft. Also, the downward vertical velocity of the Aroclor above its outlet entrained more water droplets in the effluent Aroclor at the higher Q_A's. This produced higher values of C_W for L = 4 ft than for L = 2 ft, for equal values of V_o and $C_{W(0)}$.

At the lowest Q_A (0.097 ft^3/min), decreasing V_o from 1.62×10^{-3} to 0.81×10^{-3} ft/sec by increasing L from 2 to 4 ft resulted in little reduction of C_W. In Table VI, comparing data F=1, F-2, and F-3 for L = 2 ft with data L-1 and L-2 for L = 4 ft shows that C_W was reduced from about 0.043% to 0.036%. This is also seen in comparing data C-1, C-2, and C-3 for L = 2 ft with data I-1 and I-2 for L = 4 ft. For these data, C_W was reduced from about 0.060% to 0.052% by increasing L from 2 to 4 ft.

At Q_A = 0.097 ft^3/min, the water droplets dispersed in the Aroclor effluent appeared as a cloudy haze of very tiny droplets. These droplets became dispersed in the Aroclor layer by turbulence and condensation of soluble water as the Aroclor was cooled from the storage tank temperature of from 220° to 240° F to the operating temperature of 200° F. Owing to their low U_∞, these droplets practically constitute a stable emulsion. Since decreasing V_o would have little effect on these tiny droplets, increasing L from 2 to 4 ft resulted in little reduction in C_W at the lowest Q_A (0.097 ft^3/min).

C. Aroclor Depth (Z_A)

The correlation between C_W and V_o, shown in Fig. 10, did not hold for high values of V_o ($\geq 2.43 \times 10^{-3}$ ft/sec) obtained at the lowest Z_A(0.25 ft). As the data in Table II show, for high values of V_o($\geq 2.43 \times 10^{-3}$ ft/sec), C_W decreased as Z_A was increased from 0.25 to 0.50 ft.

This result can be explained by considering the behavior of the emulsion layer and the downward Aroclor velocity in the outlet zone. In the observations of the emulsion layer it was noted that, for the highest Q_A, collapsing and coalescing droplets in the emulsion layer sent showers of droplets into the Aroclor layer. Most of these droplets penetrated less than an inch into the Aroclor layer and were observed as a whitish cloud just below the emulsion layer. As pointed out in the discussion in Section VII-A the downward Aroclor velocity in the cloudy area was greater at Z_A = 0.25 ft than at Z_A = 0.50 ft. This means that larger droplets from the cloudy area were entrained at Z_A = 0.25 ft. This would account for the high values of C_W at Z_A = 0.25 ft for high V_o's). Consequently, the high values of C_W obtained at the highest V_o's, when Z_A = 0.25 ft, were omitted from Table VI and Fig. 10 because they were outside the range of correlation.

At the lower values of V_o, there was little difference per given value of V_o between C_W at Z_A = 0.25 ft and C_W at Z_A = 0.50 or 0.75 ft. At these V_o's, the emulsion layer was less thick and no cloudy layer was observed below the emulsion layer. Also, at lower V_o' the downward velocity of the Aroclor was less above its outlet, and therfore water droplets showered into the Aroclor by droplets collapsing in the emulsion layer were less likely to be entrained in the effluent Aroclor.

D. Residence Time (L/U_A)

The data in Table II showed no correlation whatsoever between C_W and residence time L/U_A. For instance, for data E-1(b), E-2, and E-5(a), the residence times were 155, 103, and 52 sec. However, the C_W values for E-1(b), E-2, and E-5(a) were 0.080, 0.077, and 0.78, respectively. The values of C_W remained essentially constant even though the residence times were varied by a factor of 3. A correlation based on residence time alone would not adequately account for the effect of the Aroclor depth Z_A.

E. Mixing in the Inlet Pipe and Inlet Velocity

The inlet velocity to the settler and the degree of turbulence in the mixing pipe were regulated as described in Section IV-C. As a measure of the degree of turbulence and inlet velocity for given Q_A, the Reynolds Number N_{RE} was used. Since the viscosity of the Aroclor-water mixture could not be determined, the viscosity of the continuous phase (Aroclor) was used to calculate N_{RE}.

Table VII shows that, for a given V_o and $C_{W(0)}$, when N_{RE} is doubled C_W is greatly increased. Apparently, increasing the Reynolds number in the entrance pipe created more turbulence which in turn caused more small water droplets to be dispersed into the Aroclor. This agrees with Clay's observations that increasing the Reynolds number decreases the average droplet size of a dispersion. Decreasing the size of the dispersed water droplets decreased the rising velocity, of the water droplets U_∞, and thereby reduced the degree of separation in all three zones.

At the higher Reynolds numbers and inlet velocities, Aroclor droplets were observed in the water effluent from the 2-ft settler. None was observed in the water from the 4-ft settler. Table III shows the Aroclor concentration in the water effluent at various N_{RE} and $C_{W(0)}$.

Table VII. Effect of N_{RE} on C_W

Run No.[a]	L (ft)	$C_{W(0)}$ (weight %)	V_o (ft/sec × 10^3)	N_{RE}	C_W (weight %)
E-4	2	5.4	3.24	2660	0.077
N-5	2	5.4	3.24	5320	0.173
K-2	4	5.4	1.62	2660	0.060
N-6	4	5.4	1.62	5320	0.089
B-4	2	15.2	3.24	2950	0.114
N-3	2	15.2	3.24	5900	0.337
H-2	4	15.2	1.62	2950	0.077
N-4	4	15.2	1.62	5900	0.191

[a] For all runs shown:

Q_A = 0.194 ft^3/min

Z_A = 0.50 ft

Z_L = 4.0 in.

X_b = 3.5 in.

Richardson's work, which has been discussed in Section II-B, indicates that increasing the inlet velocity would decrease the mean droplet diameter. This would explain the presence of Aroclor droplets in the water layer at an N_{RE} of 5320 and 5900, although none were observed at N_{RE} of 2660 and 2950. Richardson's formula would also explain why the Aroclor concentration was higher for the highest $C_{W(0)}$, since the inlet velocity is greater at the higher initial water concentration.

F. Initial Water Concentration ($C_{W(0)}$)

At all values of V_o, increasing $C_{W(0)}$ gave higher values of C_W. This is clearly illustrated in Fig. 4.

Higher $C_{W(0)}$'s merely produced more small droplets that would not separate from the Aroclor in any of the three zones. In addition, higher initial water concentrations caused higher N_{RE}'s in the mixing pipe and therefore more turbulent mixing.

G. Baffle Position (X_b)

Table VIII compares the values of C_W obtained at different baffle positions for a given Q_A and $C_{W(0)}$. Using a side inlet with no baffle produced values of C_W that did not differ significantly from values obtained by using a front entrance and a baffle with $X_b = 3.5$ in. When the baffle-to-inlet distance X_b was decreased to 1 in., much higher values of C_W resulted.

Apparently more mixing occurred at $X_b = 1$ in. Also, the force with which the feedstream impinged on the baffle was greater at $X_b = 1$ in. than for $X_b = 3.5$ in. These two facts caused more small water droplets to be dispersed in the Aroclor, and reduced the degree of separation in all three zones for $X_b = 1$ in.

Table VIII. Effect of X_b on C_W.

Run No.[a]	V_o (ft/sec × 10^3)	$C_{W(0)}$ (weight %)	X_b (in.)	C_W (weight %)
O-1	3.24	5.4	1	0.135
E-2	3.24	5.4	3 1/2	0.077
P-1	3.24	5.4	no baffle[b]	0.084
O-3	4.85	5.4	1	0.275
D-2	4.85	5.4	3 1/2	0.181
P-3	4.85	5.4	no baffle[b]	0.169
O-2	3.24	15.2	1	0.196
B-2	3.24	15.2	3 1/2	0.113
P-2	3.24	15.2	no baffle[b]	0.133
O-4	4.85	15.2	1	0.416
A-2	4.85	15.2	3 1/2	0.293
P-4	4.85	15.2	no baffle[b]	0.319

[a] For all runs shown:

N_{RE} = 2600 to 2950

Z_I = 4.0 in.

Z_A = 0.50 ft

L = 2 ft

[b] Feed inlet on side of settler

VIII. SUMMARY AND CONCLUSIONS

C_W was fairly consistent for a given V_o for low V_o's, and for high V_o's at high Z_A's, provided that X_b, N_{RE}, $C_{W(0)}$, and L were constant. C_W increased rapidly with increasing V_o. At low V_o's, the C_W values leveled off to about 0.04%, because of very tiny water droplets that did not separate from the Aroclor. Increasing N_{RE}, $C_{W(0)}$, or L resulted in an increase in C_W. Decreasing Z_A or Z_I for high V_o's at low Z_A's, and decreasing X_b, also increased C_W. Injecting the feed stream into the Aroclor layer produced much higher values of C_W than injecting the feed stream into the water layer. At high inlet velocities N_{RE}, Aroclor was carried over in the water effluent.

These results are explained by considering the separation and dispersion mechanism in each of the three zones: the inlet zone, the settling zone, and the outlet zone. In the inlet zone, when the feed stream was injected into the water layer, the separation of water from Aroclor was aided by the breaking up of the Aroclor in the feed into droplets. Turbulence caused by the incoming feed stream hindered separation of water from Aroclor when the inlet was near the Aroclor layer, or when it was in the Aroclor layer. In the settling zone, the separation depended on the size of the dispersed water droplets and V_o. Also, in the settling zone, droplets that collapsed and coalesced in the emulsion layer caused water droplets to be dispersed in the Aroclor. In the outlet zone, the downward velocity of Aroclor above its outlet, and the rising velocities of the dispersed water droplets, were the most important elements in the separation process.

The fact that C_W varied with N_{RE} and X_b indicates that mixing upstream from the settler and at the inlet to the settler were important variables to be considered in the development of a scale-up method for gravity settlers. Lack of criteria for duplicating the upstream mixing in laboratory-scale settlers that occurs in the operation of full-scale settlers limits the usefulness of V_o as a design criterion.

Further study of this problem is necessary before a practicable design procedure for gravity settlers can be successfully developed.

The emulsion layer was another element limiting the use of V_o. For a shallow laboratory-scale settler, coalescing droplets in the emulsion layer sent showers of water droplets into the Aroclor layer, which influenced C_W at the higher values of Q_A. This influence in full-sized settlers would be difficult to estimate, because of the greater distance of the emulsion layer above the outlet.

For equal values of V_o obtained at different values of L, C_W varied. Also, for a given V_o, C_W varied with $C_{W(0)}$. These two relationships will further complicate the problem of devising a scale-up method.

The residence time L/U_A was of no value in correlating C_W with settler dimensions and flow rates. Residence times do not take into account the effect of Z_A, nor the variables $C_{W(0)}$, L, N_{RE}, and X_b.

PART II

GRAVITY SETTLING WITH INDUCED COALESCENCE

I. INTRODUCTION

Many investigators have found that emulsions of two immiscible liquids could be separated by passing the emulsion through a porous material that caused the droplets to break and coalesce. This method of "breaking" emulsions has been used extensively in the petroleum industry to separate emulsified water from oil and petroleum products.

A wide variety of materials has been suggested as emulsion breakers. Water has been separated from oil field emulsions by passing the emulsions through excelsior,[32] Fiberglas[4, 33] glass chips,[34] porous carbon particles,[35] steel wool,[36] wire mesh,[5] pumice stones,[37] calcium carbonate,[38] fuller's earth,[39] bauxite,[40] quartz,[41] filter cloth,[42] and even beds of Raschig rings.[5]

It was noted in Part I of this paper that separation of the Aroclor 1248-water mixture was hindered by the formation of an emulsion layer of water and Aroclor. One possibility for improved separation is to induce coalescence of the emulsion by passing it through a porous material. In this case, the porous material might also aid the separation by inducing coalescence of droplets dispersed in either the water or the Aroclor. The purpose of Part II of this work was to investigate Fiberglas coalescing materials in order to improve separation in gravity settlers.

II. MECHANISM OF INDUCED COALESCENCE

The mechanism by which a relatively fine, porous, fibrous membrane induces coalescence of an emulsion when the latter is passed through it is described by Jordon.[43] The first step is the rupture of the film encasing the dispersed droplets, which occurs at or near the external surface of the membrane. The second step is the coalescence and conglomeration of the dispersed droplets as they are brought into intimate contact, by the channeling effect of the fibrous membrane.

Treybal states that causing an emulsion to flow through a porous substance having a large surface-to-volume ratio, but relatively large capillaries, and which is preferentially wet by the dispersed phase, often induces coalescence.[5] Possibly the coalescence is caused by mechanical destruction of the surface film of the dispersed droplets.

Berkman and Egloff suggest that the film surrounding the dispersed droplets is ruptured by forces created by friction, impact, capillary action, or other causes, when an emulsion is passed through a capillary.[44] They noted that two immiscible fluids flow through capillary openings with different degrees of freedom, depending on whether the resistance to flow is caused by capillary action, frictional resistance due to viscosity, or other reasons. This causes the two immisicible fluids of an emulsion to move through a capillary material with different velocities. This induces coalescence by bringing the droplets of the dispersed phase into intimate contact with each other.

Voyutskii, Fadiman, and Panich used various combinations of hydrophobic (viscose fiber) and hydrophyllic (wool fiber) materials to filter water from emulsions.[45] They proposed that the mechanism of filtration consisted of the following steps:

(a) collision of the microdroplets of water with the fiber ends of the filter,

(b) adhesion of microdroplets to the fiber ends,

(c) coalescence of the microdroplets,

(d) adhesion of the coalesced droplets to the fiber, or

(e) trickling of the water droplets down the filter.

In our experiment, the coalesced water droplets did not pass through the fibers but were actually filtered out of the emulsion. At higher pressures, the water drops would have been forced through the fibers.

From the foregoing information, the method by which a medium induces coalescence seems to involve rupture of the film surrounding the dispersed droplets, followed by intimate contacts between dispersed droplets. Fiberglas coalescers may encourage this process in the following manner:

It is known that if a droplet of water is placed upon a clean, smooth, flat glass surface, the droplet spreads to form a thin film, indicating that the molecular forces of attraction between the molecules of the glass and the molecules of water are greater than the molecular forces of attraction between the molecules of water. The exact strength of molecular attraction between glass and water molecules is not known, but its order of magnitude may be estimated by use of Dupré's equation, $W_A = \sigma(1 + \cos\theta)$,[46] assuming a contact angle $\theta = 0$ deg. To separate the water molecules one molecular distance (1×10^{-7} cm) from the glass would require

$$\frac{2 \times 72.8 \text{ dynes/cm}}{1 \times 10^{-7} \text{ cm}}, \text{ or } 1490 \text{ kg/cm}^2 \ (21200 \text{ lb/in.}^2).$$

However, Rideal and Davies state that in practice this attractive force seldom exceeds 30 kg/cm^2.[47] The reason for this difference is said to lie both in the plastic and viscous flows that precede the fracture and in the dislocation in the surface of the solid.

The molecular forces of attraction between Aroclor and glass are much smaller than those between water and glass. This is observed when a droplet of Aroclor is placed on a flat glass surface. The Aroclor does not spread as water does, but forms a droplet with a contact angle of 14 deg.[48]

As the water droplets dispersed in the Aroclor pass through the Fiberglas coalescer, droplets collide with strands of the glass.

The strong attractive force causes the colliding water droplets to adhere to the glass strand and spread out along it. Other droplets overtake the adhering droplets and coalesce with them. Increasing the size of the adhering droplet by coalescence increases the buoyancy and fluid drag forces on the drop. These augmented forces then cause coalesced droplets to break away from the strand and move toward the interface. This induced coalescence reduces the number of small water droplets and results in the formation of large droplets that separate more readily from the Aroclor.

One of the most extensive investigations of a coalescing material was made by Burtis and Kirkebride,[4] who used beds of Fiberglas to break oil-field emulsions. Their results are summarized as follows:

(a) Better separation could be obtained by increasing the thickness and density of the Fiberglas bed. A bed thickness of 3 to 4 in. and a bed density fo 13.0 lb/ft^3 were found to be sufficient in most cases.

(b) The efficiency of separation decreased when the superficial velocity (i. e., the total voltmetric flow rate divided by the cross-sectional area) of the fluid through the bed was increased, ranging from 0.25 to 1 ft/min.

(c) Raising the temperature improved separation. The authors attributed the improved separation to reduction of the viscosity of the oil phase through increasing temperature.

III. EQUIPMENT

The same settlers, tanks, pumps, and other equipment were used as described in Part I (Section IV). A side entrance at the 10-in. level, with no baffle, was used in all runs, for reasons of convenience. It has been shown in Part I that the side entrance gave only slightly higher values of C_W and that the inlet height made little difference except at the highest Q_A at $Z_A = 0.25$ ft.

Four coalescers were tested under various conditions, whose dimensions and properties are shown in Table IX. The most important features of Fiberglas coalescers are their high percentage voil fraction and large surface-area-to-volume ratios. High percentage void fraction allows for low resistance to fluid flow, and high ratios of surface area to volume allow for large contacting areas in relatively small coalescers.

Pressure taps were installed on either side of the coalescer. The taps were attached to a mercury manometer to observe pressure drops through the coalescers.

Coalescer A was composed of Owens-Corning Fiberglas, No. 600 fiber. This glass comes in $25 \times 12 \times 1/2$-in. mats, and the fibers are arranged in a loose jackstraw pattern and held together by a water-soluble binder. We cut Fiberglas mats with scissors into strips 6 in. wide × 10 in. long and laid these one on top of another in the settler until its cross section was filled. The strips were held in place between two 14-mesh stainless steel screens, positioned on tie rods attached to the front of the settler. The front screen was placed 3-1/2 in. from the front of the settler. No attempt was made to compress the Fiberglas; it remained at its original bulk density of 4.70 lb/ft^3.

The settler was then filled with hot water which dissolved the soluble binder. The settler was rinsed several times to remove the last traces of the binder. After the binder was dissolved the fibers of the mats remained in place even at the highest flow rates.

Table IX. Dimensions and physical properties of Fiberglas coalescers.

Coalescer	Height (in.)	Width (in.)	Depth (in.)	Fiber diameter (in.)	Density (lb/ft^3)	Volume (% void)	Surface area to volume (ft^2/ft^3)	Surface area (ft^2)[a]
A	12	6	10	0.006	4.70	97	237	98.5
B	12	6	20	0.006	4.70	97	237	197
C	12	6	0.25	0.00028	13.0	90^+	15300	145.2
D	12	6	1	0.00028	13.0	90^+	15300	581

[a] The surface area was calculated from the following empirical formula taken from reference 49:

$S = 48/dB$

S = surface area, ft^2/lb,

d = fiber diameter, in.

B = density of glass, lb/ft^3 ($155\ lb/ft^3$)

Coalescer B, also composed of No. 600 fibers, was installed in the same manner. This Fiberglas, however, was cut into 6 × 20-in. strips.

Coalescer C was made from Owens-Corning Fiberglas, basic fiber No. 28C, which comes in sheets 10-3/4 in. wide and 1/8 in. thick, wrapped in 1/2-lb rolls. A weighed quantity of the Fiberglas was compressed to a density of 13.0 lb/ft^3 and a thickness of 1/4 in. between two 14-mesh stainless-steel screens. Care was taken to get as uniform a thickness and density as possible. The Fiberglas was allowed to overlap the edges of the screens slightly, so that a tight fit was obtained when the screens were inserted into the settler. The coalescer was placed 3-1/2 in. from the front of the settler. The screens were mounted on four tie rods attached to the front of the settler.

Coalescer D was installed in the same manner. It was also composed of Owens-Corning basic fiber No. 28C, but was packed to a thickness of 1 in.

IV. OPERATING PROCEDURE

The procedure for this experiment was identical to the one used in Part I (Section V), except that the pressure drop through the coalescer was recorded for the data taken in Part II.

V. OBSERVATIONS

A. Emulsion Layer

No emulsion layer was observed for coalescers A and B at any flow rate. Droplets reaching the interface from either phase were caused to coalesce by the glass fibers.

Some emulsion layer was observed at the higher flow rates for coalescers C and D. This emulsion layer consisted mainly of large droplets, and in the worst case resembled the emulsion layer shown in Fig. 8. The emulsion layer was composed of droplets that passed through the coalescer, reached the interface, and remained there for a short time before coalescing.

B. Pressure Drop

In all runs the pressure drop through the coalescers was too small to be observed on a mercury manometer.

VI. DISCUSSION OF RESULTS

A. Flowrate

The effect of the four Fiberglas coalescers on C_W at various Q_A and initial water concentrations is shown on Figs. 11 and 12. The graphs were plotted from data in Table X.

All the C_W-vs-Q_A curves for the coalescers were well below the curve for the gravity settler with no coalescer. However, the degree of separation attained by each coalescer was different. From Figs. 11 and 12 it was noted that coalescer D seemed to be the most effective; coalescer B was next, followed by coalescers C and A, respectively.

This may be explained by comparing the surface areas of the four coalescers:

Coalescer	Surface area (ft^2)
A	98.5
C	145.0
B	197.0
D	582.0

It is to be expected that the coalescers with the most surface area for water droplets to impinge on and coalesce would give the best degree of separation, and Figs. 11 and 12 show that this was the case.

The effectiveness of coalescers A, B, and C seemed to decrease with increasing Q_A. This agreed with Burtis and Kirkbride's results. Increasing the velocity of the Aroclor through the coalescer decreased the residence time of the water droplets in the coalescer, thereby decreasing the possibility of collisions between the water droplets and the Fiberglas. Also, increasing this velocity increased the drag force of the Aroclor on the water droplets that adhered to the Fiberglas. This tended to pull the droplets from the glass and reduced the possibility of collision and coalescence between the adhering droplets and droplets dispersed in the Aroclor.

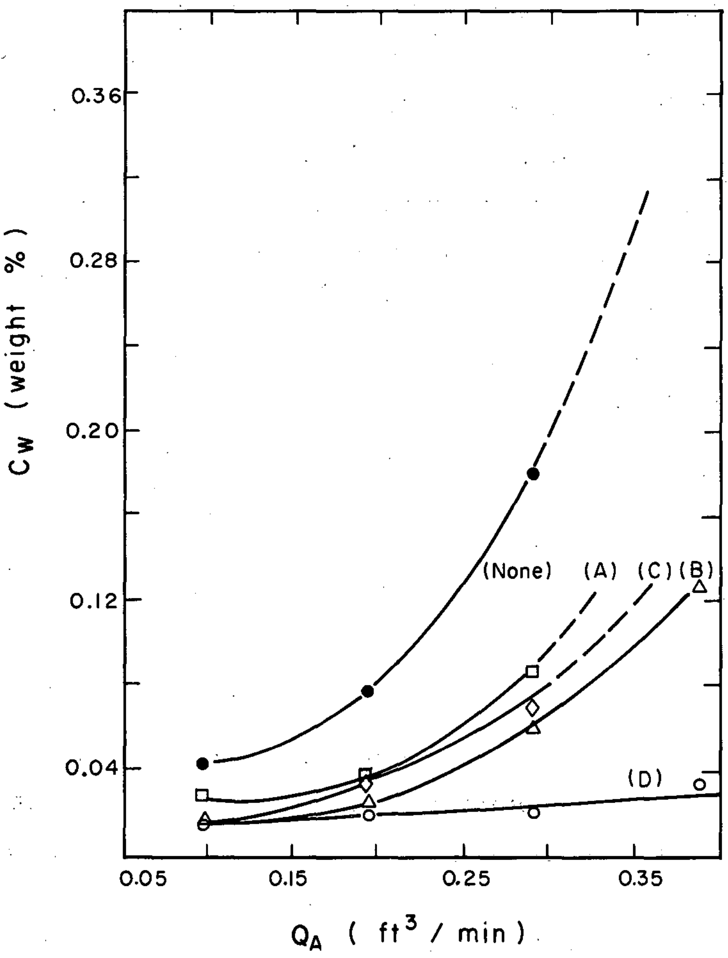

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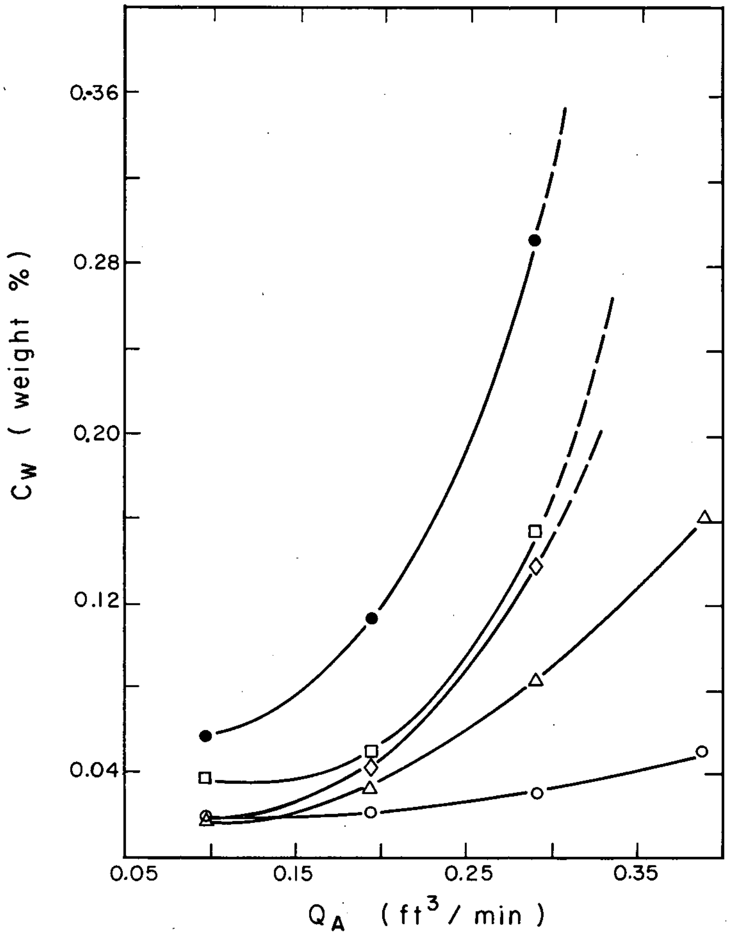
Fig. 11. Effect of coalescer on C_W for various Q_A, where $N_{RE} = 2660$, $L = 2$ ft, $C_{W(0)} = 5.40\%$, $Z_A = 0.50$ ft, $Z_I = 4$ in., $X_C = 3.5$ in., and side entrance temp = 200° F.



MU-26221

Fig. 12. Effect of coalescer on C_W for various Q_A, where N_{RE} = 2950, $C_{W(0)}$ = 15.2%, L = 2 ft, Z_A = 0.50 ft, Z_I = 4 in., X_C = 3.5 in., and side entrance temp = 200° F.

Table X. Experimental data and derived results for the separation of water from Aroclor with coalecers.

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec$\times 10^3$)	Z_A (ft)	L (ft)	V_o (ft/sec$\times 10^3$)	X_C (in.)	N_{RE}	Coalescer	C_W (weight %)
1-N	0.097	5.4	6.46	0.50	2	1.62	--	2660	none	0.043
2-N	0.194	5.4	12.9	0.50	2	3.24	--	2660	none	0.077
3-N	0.291	5.4	19.4	0.50	2	4.85	--	2660	none	0.181
5-N	0.097	15.2	6.46	0.50	2	1.62	--	2950	none	0.057
6-N	0.194	15.2	12.9	0.50	2	3.24	--	2950	none	0.113
7-N	0.291	15.2	19.4	0.50	2	4.85	--	2950	none	0.293
1-A	0.097	5.4	6.46	0.50	2	1.62	3.5	2660	A	0.028
2-A	0.194	5.4	12.9	0.50	2	3.24	3.5	2660	A	0.033
3-A	0.291	5.4	19.4	0.50	2	4.85	3.5	2660	A	0.087
5-A	0.097	15.2	6.46	0.50	2	1.62	3.5	2950	A	0.037
6-A	0.194	15.2	12.9	0.50	2	3.24	3.5	2950	A	0.050
7-A	0.291	15.2	19.4	0.50	2	4.85	3.5	2950	A	0.155
1-B	0.097	5.4	6.46	0.50	2	1.62	3.5	2660	B	0.017
2-B	0.194	5.4	12.9	0.50	2	3.24	3.5	2660	B	0.023
3-B	0.291	5.4	19.4	0.50	2	4.85	3.5	2660	B	0.060
4-B	0.388	5.4	25.9	0.50	2	6.48	3.5	2660	B	0.127
5-B	0.097	15.2	6.46	0.50	2	1.62	3.5	2950	B	0.017
6-B	0.194	15.2	12.9	0.50	2	3.24	3.5	2950	B	0.034
7-B	0.291	15.2	19.4	0.50	2	4.85	3.5	2950	B	0.084
8-B	0.388	15.2	25.9	0.50	2	6.48	3.5	2950	B	0.161

Table X. (continued)

Run number	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	U_A (ft/sec×10^3)	Z_A (ft)	L (ft)	V_o (ft/sec×10^3)	X_C (in.)	N_{RE}	Coalescer	C_W (weight %)
1-C	0.097	5.4	6.46	0.50	2	1.62	3.5	2600	C	0.017
2-C	0.194	5.4	12.9	0.50	2	3.24	3.5	2600	C	0.040
3-C	0.291	5.4	19.4	0.50	2	4.85	3.5	2600	C	0.069
5-C	0.097	15.2	6.46	0.50	2	1.62	3.5	2950	C	0.019
6-C	0.194	15.2	12.9	0.50	2	3.24	3.5	2950	C	0.044
7-C	0.291	15.2	19.4	0.50	2	4.85	3.5	2950	C	0.139
1-D	0.097	5.4	6.46	0.50	2	1.62	3.5	2600	D	0.016
2-D	0.194	5.4	12.9	0.50	2	3.24	3.5	2660	D	0.020
3-D	0.291	5.4	19.4	0.50	2	4.85	3.5	2660	D	0.020
4-D	0.388	5.4	25.9	0.50	2	6.48	3.5	2660	D	0.033
5-D	0.097	15.2	6.46	0.50	2	1.62	3.5	2950	D	0.019
6-D	0.194	15.2	12.9	0.50	2	3.24	3.5	2950	D	0.022
7-D	0.291	15.2	19.4	0.50	2	4.85	3.5	2950	D	0.031
8-D	0.388	15.2	25.9	0.50	2	6.48	3.5	2950	D	0.051
8-N	0.194	5.4	12.9	0.50	2	3.24	3.5	5320	none	0.173
9-N	0.194	15.2	12.9	0.50	2	3.24	3.5	5900	none	0.337
8-A	0.194	5.4	12.9	0.50	2	3.24	3.5	5320	A	0.051
9-A	0.194	15.2	12.9	0.50	2	3.24	3.5	5900	A	0.103

The curves for coalescer D showed little change of C_W with flow rate. Even at Q_A = 0.388 ft^3/min, coalescer D provided about the same degree of separation as the gravity settler with no coalescer provided at Q_A = 0.097 ft^3/min. The greater surface area of coalescer D allowed for adequate coalescence even at the highest flow rate.

Curves for coalescers B, C, and D converged at the lowest flow rate at a value of C_W equal to about 0.02%. This insoluble water probably consisted of very tiny water droplets that had passed through the coalescer without coalescing. These droplets became dispersed in the Aroclor through turbulent mixing and condensation of soluble water when the Aroclor was cooled from the storage tank temperature of about 230° F to the settler temperature of 200° F.

B. Initial Water Concentration ($C_{W(0)}$)

For a given Q_A, increasing $C_{W(0)}$ from 5.4% to 15.2% increased C_W, particularly at the higher Q_A's. This was due to more small water droplets' being dispersed in the Aroclor at the higher initial water concentration. The effect of increasing $C_{W(0)}$ was less pronounced for coalescers B and D because of their greater surface areas.

C. Mixing in the Inlet Pipe, and Inlet Velocity

As discussed in Part I (Section VII-E), increasing N_{RE} in the inlet tube caused smaller water droplets to be dispersed in the Aroclor. This explains why C_W values in Table XII are higher than corresponding values of C_W in Table XI. The effect of higher Reynolds numbers is more pronounced for coalescers A, B, and C than for coalescer D, because these coalescers had less surface area.

In Part I, for the gravity settler with no coalescer, Aroclor droplets were observed in the water layer at the higher Reynolds numbers. The coalescers were successful in causing the Aroclor droplets dispersed in the water layer to coalesce. No Aroclor was detected in the water layer for any coalescer at any flow rate.

Table XI. Effect of surface area of coalescers on C_W at N_{RE} 2660-2950.

Run number[a]	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	Coalescer	Surface area (ft^2)	C_W (weight %)
1-N	0.097	5.4	none[b]	0	0.043
1-A	0.097	5.4	A	98.5	0.028
1-C	0.097	5.4	C	145.2	0.017
1-B	0.097	5.4	B	197	0.017
1-D	0.097	5.4	D	581	0.016
2-N	0.194	5.4	none[b]	0	0.077
2-A	0.194	5.4	A	98.5	0.033
2-C	0.194	5.4	C	145.2	0.040
2-B	0.194	5.4	B	197	0.023
2-D	0.194	5.4	D	581	0.020
3-N	0.291	5.4	none[b]	0	0.181
3-A	0.291	5.4	A	98.5	0.087
3-C	0.291	5.4	C	145.2	0.069
3-B	0.291	5.4	B	197	0.060
3-D	0.291	5.4	D	581	0.020
4-B	0.388	5.4	B	197	0.127
4-D	0.388	5.4	D	581	0.033
5-N	0.097	15.2	none	0	0.057
5-A	0.097	15.2	A	98.5	0.037
5-C	0.097	15.2	C	145.2	0.019
5-B	0.097	15.2	B	197	0.017
5-D	0.097	15.2	D	581	0.019

Table XI. (continued)

Run number[a]	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	Coalescer	Surface area (ft^2)	C_W (weight %)
6-N	0.194	15.2	none	0	0.113
6-A	0.194	15.2	A	98.5	0.050
6-C	0.194	15.2	C	145.2	0.044
6-B	0.194	15.2	B	197	0.034
6-D	0.194	15.2	D	581	0.022
7-N	0.291	15.2	none	0	0.293
7-A	0.291	15.2	A	98.5	0.155
7-C	0.291	15.2	C	145.2	0.139
7-B	0.291	15.2	B	197	0.084
7-D	0.291	15.2	D	581	0.031
8-B	0.388	15.2	B	197	0.161
8-D	0.388	15.2	D	581	0.051

[a] For all runs shown:

X_C = 3.5 in.
L = 2 ft
N_{RE} = 2660 - 2950
Z_A = 0.50 ft
Z_I = 4 in.

[b] Feed inlet on side of settler.

Table XII. Effect of surface area of coalescers on C_W at N_{RE} 5320-5900.

Run number[a]	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	Coalescer	Surface area (ft^2)	C_W (weight %)
8-N	0.194	5.4	none[b]	0	0.173
8-A	0.194	5.4	A	98.5	0.051
8-C	0.194	5.4	C	145.2	0.047
9-B	0.194	5.4	B	197	0.036
9-D	0.194	5.4	D	581	0.024
11-B	0.388	5.4	B	197	0.124
11-D	0.388	5.4	D	581	0.069
9-N	0.194	15.2	none[b]	0	0.337
9-A	0.194	15.2	A	98.5	0.103
9-C	0.194	15.2	C	145.2	0.084
10-B	0.194	15.2	B	197	0.068
10-D	0.194	15.2	D	581	0.028
12-B	0.388	15.2	B	197	0.171
12-D	0.388	15.2	D	581	0.069

[a] For all runs shown:

X_C = 3.5 in.
L = 2 ft
N_{RE} = 5320 - 5900
Z_A = 0.50 ft
Z_I = 4 in.

[b] Feed inlet on side of settler.

D. Settler Length, (L)

Table XIII shows that increasing L from 2 ft to 4 ft did not affect C_W for coalescer D, and reduced C_W only slightly for coalescer C. Although increasing L decreased V_o (i.e., decreased the rising velocity U_∞ required for the dispersed water droplets to reach the interface), C_W was only slightly reduced.

This would indicate that only extremely small water droplets remained in the Aroclor after passing through the coalescer and the settling zone of the 2-ft settler. These small water droplets had U_∞'s so small that decreasing V_o did not improve the separation of water from Aroclor. From the standpoint of settler operation, the tiny droplets that did not separate from the Aroclor when coalescer D was used could be considered to be a stable emulsion.

E. Distance of Coalescer from Inlet (X_c)

Table XIV shows that increasing X_c from 3.5 to 23 increases C_W, particularly at the higher Q_A's. At the largest X_c, the emulsion layer traveled 23 in. down the interface before it reached the coalescer. Coalescing droplets in the emulsion layer sent showers of water droplets into the Aroclor. Some of these passed through the coalescer without coalescing and were entrained in the effluent Aroclor. Also, the water droplets that did pass through the coalescer had less time to rise to the interface at distance X_c = 23 in. This would account for the increase in C_W with X_c.

Table XIII. Effect of increasing L on C_W for coalescers C and D.

Run number[a]	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	V_o ($ft/sec \times 10^3$)	L (ft)	Coalescer	C_W (weight %)
1-C	0.097	5.4	1.62	2	C	0.017
10-C	0.097	5.4	0.81	4	C	0.022
2-C	0.194	5.4	3.24	2	C	0.040
12-C	0.194	5.4	1.62	4	C	0.030
3-C	0.291	5.4	4.85	2	C	0.069
14-C	0.291	5.4	2.43	4	C	0.058
5-C	0.097	15.2	1.62	2	C	0.019
11-C	0.097	15.2	0.81	4	C	0.022
6-C	0.194	15.2	3.24	2	C	0.044
13-C	0.194	15.2	1.62	4	C	0.032
7-C	0.291	15.2	4.85	2	C	0.139
15-C	0.291	15.2	2.43	4	C	0.113
1-D	0.097	5.4	1.62	2	D	0.016
13-D	0.097	5.4	0.81	4	D	0.017
2-D	0.194	5.4	3.24	2	D	0.020
15-D	0.194	5.4	1.62	4	D	0.019
3-D	0.291	5.4	4.85	2	D	0.020
17-D	0.291	5.4	2.43	4	D	0.022
5-D	0.097	15.2	1.62	2	D	0.019
14-D	0.097	15.2	0.81	4	D	0.021
6-D	0.194	15.2	3.24	2	D	0.022
16-D	0.194	15.2	1.62	4	D	0.022
7-D	0.291	15.2	4.85	2	D	0.031
18-D	0.291	15.2	2.43	4	D	0.028

[a] For all runs shown: X_c = 3.5 in.; Z_A = 0.50 ft; Z_I = 4.0 in.; N_{RE} = 2660 to 2950; Side inlet.

Table XIV. Effect of X_c on C_W for coalescer C

Run number[a]	Q_A (ft^3/min)	$C_{W(0)}$ (weight %)	X_c (in.)	C_W (weight %)
10-C	0.097	5.4	3.5	0.022
16-C	0.097	5.4	23	0.023
12-C	0.194	5.4	3.5	0.030
17-C	0.194	5.4	23	0.046
14-C	0.291	5.4	3.5	0.058
18-C	0.291	5.4	23	0.129
11-C	0.097	15.2	3.5	0.022
19-C	0.097	15.2	23	0.022
13-C	0.194	15.2	3.5	0.032
20-C	0.194	15.2	23	0.056
15-C	0.291	15.2	3.5	0.113
21-C	0.291	15.2	23	0.153

[a] For all runs shown:

$L = 4$ ft

$Z_A = 0.50$ ft

$Z_I = 4.0$ in.

$N_{RE} = 2560$ to 2950

Side inlet

VII. SUMMARY AND CONCLUSIONS

All coalescers succeeded in varying degrees in reducing the water concentration in the Aroclor by causing the coalescence of dispersed water droplets both in the emulsion layer and in the Aroclor. The surface area seemed to be the most important element in determining the effectiveness of the coalescers. Coalescers with greater surface areas for the dispersed water droplets to adhere to and coalesce provided the best separation of water from Aroclor.

When the number of small water droplets dispersed was increased by increasing X_c, N_{RE}, or $C_{W(0)}$ the degree of separation attained by the coalescers was reduced. However, these variables affected separation less for coalescers with larger surface areas.

High Aroclor velocities through the coalescers also reduced their effectiveness. Increasing Aroclor velocity resulted in less residence time for the dispersed water droplets in the coalescers, and caused water droplets adhering to the glass fibers to be entrained in the Aroclor.

Increasing the length of the settler downstream from the coalescer resulted in little improvement in the separation of water from Aroclor. This was because of the low settling velocities of the small water droplets that remained in the Aroclor after passing through the coalescer.

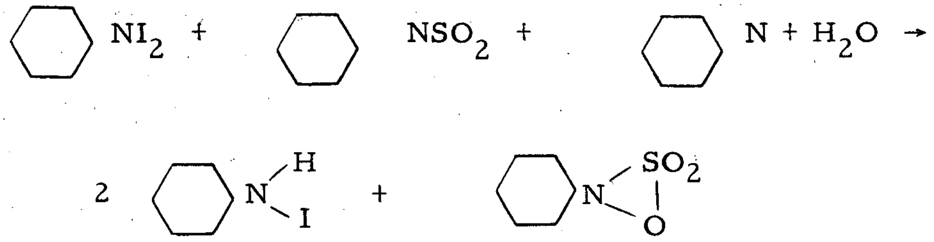
The coalescers successfully removed Aroclor from the water layer and coalesced Aroclor droplets in the emulsion layer. No Aroclor was detected in the water effluent at any flow rate, in the initial water concentration, or in the initial mixing conditions.

APPENDICES

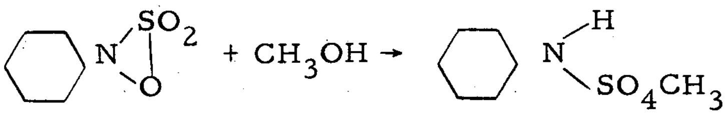
A. Analytical Procedure for the Determination of Water in Aroclor, Using Karl Fischer Reagent

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:[50]



(A. 1)



(A. 2)

Equations (A. 1) and (A. 2) indicate that the maximum absorption 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 include the reaction of iodine and pyridine to form iodopyridine, the reduction of iodine to the iodide ion, and the formation of quaternary methylpyridinium salts.[50] 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 per hour. This is tabulated in Table XV.

Table XV. Change of strength of Karl Fischer reagent.

Time after preparation of the Karl Fischer reagent (hr)	Water equivalents ($mg/ml\ H_2O$)
18	11.23
21	9.51
23	8.74
24.5	8.34
26	8.06
34	7.90
36	7.86
38	7.85

2. Preparation of Karl Fischer Reagent

The Karl Fischer reagent used had the following composition per liter:

Pyridine	316 ml
Iodine	152 g
Methanol	631 ml
Sulfur dioxide	45 ml

A 9.5-liter quantity of stock reagent was prepared by dissolving 1140 g of iodine in 3.0 l of pyridine. When the iodine had dissolved, 6 l of methanol was added.

Two liters of stock solution was transferred to a buret reservior cooled in a slurry of chopped ice. A 90-ml quantity of sulfur dioxide was collected in a cold trap immersed in a methanol-dry-ice bath. The cold liquid SO_2 was then added slowly and carefully to the stock solution in the reservoir. The reagent was set aside for a few days to allow parasitic side reactions to approach equilibrium.

The procedure used was similar to the one suggested by Mitchell[50] except 1.5 times as much iodine and 1.25 times as much pyridine were used. This produced a reagent that had a water equivalence of 7.0 to 8.0 mg/ml, whereas Mitchell's reagent had a water equivalence of 3.5 mg/ml.

3. Apparatus

A 50-ml pyrex automatic buret with 0.1-ml subdivisions was used for the titrations. The buret was connected to a vacuum line for filling and a dry nitrogen line for emptying. A stainless steel hypodermic needle was used as a buret tip.

4. Standardization of the Karl Fischer Reagent

The Karl Fischer Reagent was standardized in the following manner: A magnetic stirring bar and 2 to 4 ml of methanol were placed in a 50-ml Erlenmeyer flask covered with a rubber cap. The buret needle was inserted in the rubber cap, and the methanol titrated to the end point. The methanol was swirled around in the flask to remove any adsorbed water and then retitrated, if necessary.

A weighed quantity (0.25 to 0.35 g) of sodium acetate trihydrate was added to the flask and titrated to the end point. The color change was from yellow to the brownish color of iodine. The end point was the point at which the brownish color of iodine did not fade.

5. Titration of Water in Aroclor

Samples of Aroclor weighing from 40 to 60 g were collected in 100-ml volumetric flasks. The flasks had been dried at 120° C for several hours and then stored in a desiccator. The amount of adsorbed water on the flasks was negligible (less than 0.2 mg).[50] A dry teflon-coated stirring bar was placed in the flask and the mouth of the flask was covered with a rubber cap. The sample was titrated until the reddish-brown color of iodine appeared and did not fade.

B. Determination of Aroclor in Water

Although Aroclor is insoluble in water, some of it was entrained in the water effluent from the settler at certain flow rates. The concentration was measured by dissolving the Aroclor in benzene, separating the Aroclor-rich benzene from the water, and then evaporating off the benzene. A procedural description is given below.

A 500-ml sample was withdrawn into a 500-ml volumetric flask, at the water sampling tap. The sample was allowed to stand until the Aroclor droplets settled to the bottom of the flask. A few small droplets of Aroclor did not settle, but remained as a thin film on the surface of the water.

Five ml of benzene was added to the flask to dissolve the Aroclor on the water surface. The benzene, and about 400 ml of the water, were then carefully decanted to a separatory funnel and separated by their difference in density. The benzene was saved in the separatory funnel.

About 30 ml of benzene was added to the remaining 100 ml of water in the volumetric flask. By gently swirling the benzene, the Aroclor on the bottom of the flask was dissolved. The water and benzene were separated in the separatory funnel and the benzene containing dissolved Aroclor was saved. The volumetric flask was rinsed with 10 ml of benzene and then the benzene in the flask was added to the benzene in the separatory funnel.

The benzene was removed from the separatory funnel and placed in a 100-ml beaker. The funnel was rinsed with 10 ml of benzene and this benzene was added to the 100-ml flask.

The 100-ml beaker was placed on a hot plate and the benzene-Aroclor mixture was evaporated down to about 5 ml. The Aroclor does not evaporate, owing to its low vapor pressure (i. e., 0.16 mm of Hg at 100° C).[50] The remaining 5 ml was transferred to a 12-ml tube with a tapered bottom calibrated in 0.1 ml graduations. The 100-ml beaker was rinsed with 2 to 3 ml of benzene which was then added to the 12-ml tube.

The 12-ml tube was placed in a boiling water bath and the benzene-Aroclor mixture was evaporated gently. When no more benzene evaporates off, the residue in the tube was heated gently over an open flame to drive off the remaining benzene. The residue was cooled and the volume estimated to the nearest 0.01 ml.

C. Physical Properties of Aroclor 1248

Aroclor 1248 is a heat-transfer medium made by the Monsanto Chemical Company.[51] It is essentially tetrachlorobiphenyl with very small amounts of both lower and higher chlorinated biphenyls. Its physical properties are as follows

(a) Density[51]

Temperature (°C)	Density (g/cm^3)
30	1.44
60	1.41
93.4 (200° F)	1.376
100	1.37

(b) Viscosity[51]

Temperature (°C)	Viscosity (centipoises)
30	112
60	17.5
93.4 (200° F)	5.0
100	4.2

(c) Surface Tension and Interfacial Tension

The surface tension and interfacial tension were determined with an interfacial tension meter:

Surface tension of Aroclor against air at
20° C = 42.9 dynes/cm

Interfacial tension of Aroclor against water
at 20° C = 44.6 dynes/cm

E. Solubility

The solubility of water in Aroclor 1248 was determined by titration with Karl Fischer reagent. The plot of mole % vs the reciprocal of the absolute temperature in ^{o}K is shown in Fig. 13.

Aroclor 1248 has no detectable solubility in water.

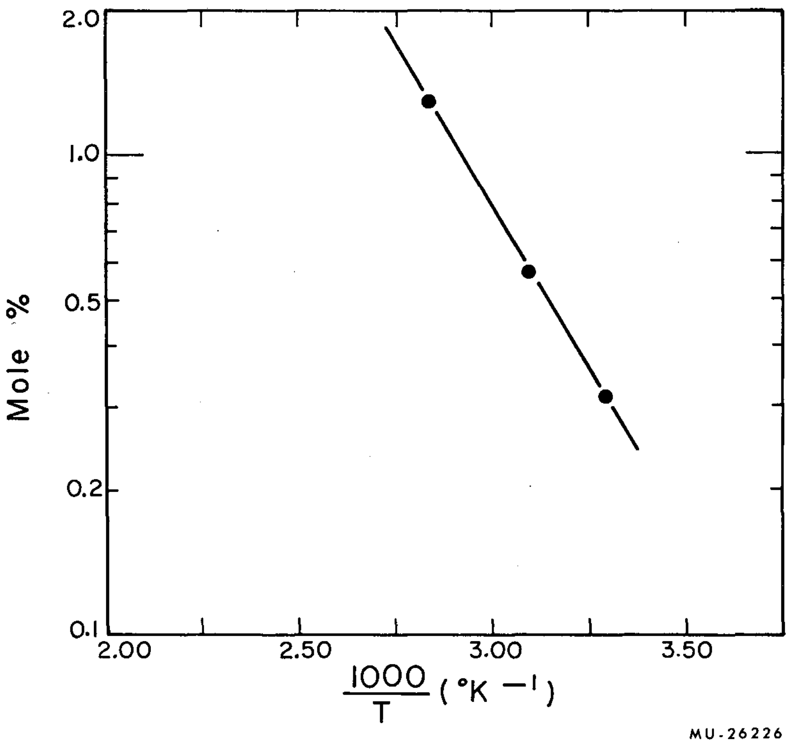


Fig. 13. Solubility of water in Aroclor 1248.

ACKNOWLEDGMENTS

The author wishes to gratefully acknowledge the assistance of Professor Charles R. Wilke during the course of this experiment.

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

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