DIRECT CONTACT HEAT TRANSFER BETWEEN IMMISCIBLE PHASES IN A SPRAY COLUMN

by

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B. Ch. E., University of Minnesota, 1967

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1970

Approved by:

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INTRODUCTION

This research on heat transfer between two immiscible species in a spray column was inspired by the Inversion Freezing Process presented by C. Y. Cheng and S. W. Cheng (1).

The Inversion Freezing Process is based on the abnormal melting point curve of water [$\frac{dP}{dT}$ melting < 0]. In their presentation, the Cheng's noted that if a straight-chained organic mixture in a partially frozen slurry were contacted with brine at atmospheric pressure, partial freezing of the saline water would occur. If the ice crystals were then separated from the brine, recontacted with the spent organic mixture, and pressurized, the ice would melt, producing fresh water, and the organic slurry would be partly regenerated. As a method of desalination, this process has the advantages of involving only condensed phases and not requiring extensive heat transfer surfaces which would be susceptible to fouling.

In a further development of their work, S. S. Maa (2) studied the thermodynamics of the process and found some suitable organic mixtures. Chiu (3) studied the ice-making process using several of Maa's suggested organic mixtures in conjunction with a mixer-settler contacting device. Chiu reported that the Inversion Freezing Process produced good sized ice crystals.

Processes such as the Inversion Freezing Process which involve a close temperature approach are often more efficiently carried out by continuous counter-current contacting instead of discrete methods such as those employed by Chiu. The Inversion Freezing Process' requirement for direct contact suggested the use of a spray column. Though all direct contact spray column freezing processes had used a vaporizing refrigerant, some direct contact heat exchange work between oils and brine (4) indicated the possibility of using a spray column as an ice maker.

The objectives of this research were: first, to study the heat transfer characteristics of a spray column in two phase (brine and organic liquid), three phase (brine, organic solid, and organic liquid) and four phase (brine, fresh water ice, organic solid, and organic liquid) operation; and second, to investigate the feasibility of using direct countercurrent contact between an organic slurry (52 vol. % n-$C_{13}H_{28}$ and 48 vol. % $C_{14}H_{30}$) and saline water to make fresh ice crystals. This second objective required special emphasis on problems of droplet formation, flooding, and recovery of a pure ice product.

LITERATURE SURVEY

The flooding point in a spray column has been defined as that point at which the discontinuous phase becomes continuous or where the discontinuous phase is forced out of the column at the continuous phase exit. Flooding point prediction is extremely important since, once the flow rates are set, the flooding point determines the minimum diameter of the column.

Sakiadis and Johnson (5) developed a generalized flooding correlation of flooding rates in spray columns which applies to solid-liquid, liquid-liquid and gas-liquid systems. Their analysis started with Bernoulli's equation applied separately to each phase. The use of Bernoulli's equation implied an irrotational flow regime of an inviscid incompressible fluid and visualized the dispersed phase drops as a continuum within the column. Operating with these assumptions, Sakiadis and Johnson developed a flooding equation for spray columns which correlated 429 spray column runs with an average error of 12.5%. The final correlation included the effect of continuous phase viscosity.

McCaskill (6) recently gave a qualitative description of the hydrodynamic regimes which prevail at different flow rate combinations in a spray column. McCaskill made his observations in a rectangular spray column (a constant cross-section of 4 1/16-in. by 3 3/8-in., 4-feet long) using a Humbletherm 500 - water system. He categorized the column's various hydrodynamic regimes according to droplet appearance and behavior which he observed through pyrex faces on the front and back sides of the column . McCaskill's five categories are listed as he presented them in his Table III. This type of description, though not quantitatively precise, is useful to the equipment designer and operator in characterizing the flow regimes. McCaskill's table is given below.

McCaskill's Table III (6)

Categories of Column Behavior with
Regard to Backmixing and Coalescence

1. IDEAL BEHAVIOR:	Drops travel straight up column; drop size is uniform.
2. PSEUDO-IDEAL BEHAVIOR:	Drops hesitate and follow crooked path but always travel upward; drop size is uniform.
3. TURBULENT BEHAVIOR:	Swarms of Drops can be seen swirling in the column, sometimes in a downward direction; very little coalescence so drop size is fairly uniform.
4. COALESCENCE & BACKMIXING:	Swarms of drops eddying violently, swirls of drops fall below the distributor plate; drops hitting sides of the column coalesce into larger drops and slugs resulting in a distribution of drop sizes.

5. FLOODING: Drops constantly swirling below distributor plate; no drops of small size reaching top of column, only large slugs; very wide distribution of drop sizes exist in the column; interface level very difficult to maintain. (6)

In conjunction with desalination work, gas-liquid spray columns have become increasingly important. A visual description of the operation of a three phase spray column in which one of the liquid species vaporizes is given by Sideman and Gat (7).

Drop size is an important factor in determining the hydrodynamics of spray columns and their heat transfer capabilities. Sakiadis and Johnson (5) noted that ". . . an error of 10% in diameter determination will result in an estimated error of 10% in predicting flood rates."

Garwin and Smith (8) used a photographic technique for drop size determination which took into account the photographic distortion caused by the circular glass column. Their technique seems the most precise method available for use with transparent systems. Though providing a measure of droplet diameter, Garwin and Smith's method determines drop size at only one position in the column, whereas drop size distribution is probably also a function of radial position.

The mode of heat transfer from drops is complicated by such factors as continuous films, mixing in the continuous phase, drop distortion, and the presence or lack of circulation within a drop.

Hughes and Gilliland (9) reviewed literature in 1952 concerning drop motion and analyzed the effect of drop distortion on internal circulation and the effect of acceleration on drag.

In 1956 McDowell and Myers (10) did a theoretical analysis of heat transfer from a continuous liquid to a drop and made predictions of transfer based on system models which considered internal drop circulation and film resistances. They found that there was a small outside film resistance and negligible internal circulation in the case of high viscosity oil drops passing through water. McDowell and Myers found agreement between the model of internal circulation with no outside film resistance for the case of kerosene, xylene and organic-liquids of lower viscosity rising through water. For the case of water drops rising through mixed organic liquids they achieved close agreement with their ". . . internal circulation - appreciable outside film resistance" model. The result of their research provided some insight into the heat transfer mechanisms which can be expected for liquids with different viscous properties. Their results also provided interesting qualitative correlation with the ideas of the film theory and of a droplet's internal mixing.

The most significant advance in the theory of spray column heat transfer mechanisms has been given by Letan and Kehat (11). Measuring the oil and water temperature profiles in a column, Letan and Kehat found that they could not get a heat balance to check. Further investigation led them to advance a model wherein a drop, upon formation, acquires a wake of continuous fluid which accompanies it through the lower part of the column. In the central part of the column wakes are shed and new ones attach to the drop. At the coalescence interface the wakes are finally detached and mix with incoming continuous fluid producing a "mixing" region where all phases are isothermal.

The wake-model presented by Letan and Kehat (11) was further investigated by Markowitz and Bergles (12). They used 4 inlet-outlet thermocouples and 7 thermocouples inside the column to measure both temperature profiles along the column's length and a temperature profile through a drop at the first three positions in the column. In the column each thermocouple bead (diameter = 0.02-in.) was contacted alternately by the continuous aqueous phase and then enveloped by an upcoming droplet. From their 200 experimental runs Letan and Kehat's wake mechanism was found to be the only acceptable model of column heat transfer. This finding led Markowitz and Bergles to the conclusion that volumetric heat transfer coefficients are not the most useful form of heat transfer correlation since they are not based on the more complicated wake heat transfer mechanism.

Recently, however, volumetric heat transfer coefficients have been used to correlate spray column heat transfer data. Even Markowitz and Bergles have presented some of their data as volumetric heat transfer coefficients versus holdup to validate their work against that of Steinmeyer and Woodward (13).

Volumetric heat transfer coefficients are calculated from the equation:

$$U_{\text{w. vol.}} = \frac{q_d}{[\Delta T_{LM}]\ [\text{column volume}]}$$

In this equation, q_d, the overall heat transfer rate between the two phases, is evaluated from dispersed phase temperatures so that errors from heat losses to the surroundings can be minimized. ΔT_{LM} is the log-mean temperature difference between the two phases over the entire column. Its use results from the assumption of ideal countercurrent flow in the column. The column volume is defined as that working volume in which contact occurs between the two phases.

Pierce, Dwyer, and Martin (14) reported volumetric heat transfer coefficients for the mercury-water system using 2 different column diameters: 1-in. and 2-in. I. D. Their drop size varied between 0.027 and 0.115-in. while the number of nozzles used varied between 9 and 29. The volumetric heat transfer coefficients reported by Pierce, Dwyer, and Martin (14) vary between 5,600 BTU/(hr-ft^3-°F) and 48,300 BTU/(hr-ft^3-°F). Their report on mercury-water column hydrodynamics (14) stated that ". . . considerable water bypassed the stream of drops (flowing down the center of the column) while some (water) surrounding the drops flowed downward." This caused two countercurrent streams of water to form in the column and made the major heat transfer resistance that of the continuous phase. The importance of this finding was realized by the three investigators (Pierce, Dwyer, and Martin) in 1959, but it was not until 1968 that Letan and Kehat (11) were able to explain the phenomenon with their wake mechanism.

Woodward (4), using a Shell-oil and water system, studied the operation of closed loop dual spray columns. Woodward's columns were 4-in. pyrex glass pipe and Woodward varied his nozzle size between 0.040 and 0.0625-in. while the number of nozzles ranged between 104 and 450. Volumetric heat transfer coefficients reported by Woodward range from about 2,770 BTU/(hr-ft^3-°F) at 20% holdup to maximum values of 11,500 BTU/(hr-ft^3-°F), top of column hot (dispersed phase hot), and to 8,500 BTU/(hr-ft^3-°F), top of column cold (dispersed phase cold). This dependence of volumetric heat transfer coefficient on the direction of heat transfer will be discussed later. Woodward found no appreciable end effects when he varied his column length between 5.3-feet and 8.7-feet.

Garwin and Smith (8) report volumetric heat transfer coefficients for the benzene-water system based on experimental runs in a 2-in. I. D., 6-foot long pyrex glass column. The benzene (dispersed phase) flow rate ranged from 3,672 to 10,050 lbm/(hr-ft^2) and the water (continuous phase) flow rate ranged from 4,050 to 11,550 lbm/(hr-ft^2) while drop diameter, a function of both benzene flow rate and direction of heat transfer, varied from 0.277 to 0.319-in. Garwin and Smith report volumetric heat transfer coefficients ranging from 390 BTU/(hr-ft^3-°F) to 4,220 BTU/(hr-ft^3-°F) for heat transfer from the continuous phase to the dispersed benzene phase. For heat transfer from the dispersed benzene to the continuous aqueous phase their volumetric heat transfer coefficients vary between 1,035 and 6,650 BTU/(hr-ft^3-°F).

Volumetric heat transfer coefficients for a Humbletherm 500 and water system are reported by McCaskill (6) from his work with a 4-foot long rectangular spray column of 3 3/8-in. by 4 1/16-in. dimensions. McCaskill used 136 nozzles and three different nozzle diameters: 0.0400, 0.0625 and 0.0937-in. Four different oil flow rates from 4,002 lbm/(hr-ft^2) to 9,979 lbm/(hr-ft^2) were tested with five different water flow rates varying from 2,524 to 12,600 lbm/(hr-ft^2). Within these flow rate ranges the three nozzle diameters produced droplets with average diameters (assuming spherical drops) of about 0.12, 0.192, and 0.252-in., respectively; enabling McCaskill to classify his results by small, medium, or large size drops.

Utilization of the counter-current flow, negligible heat loss model of the spray column heat transfer process lends itself to data correlation through calculation of the Height of a Transfer Unit (HTU). To use this correlation technique, the overall number of transfer units in the spray column, NTU_{od}, is first calculated from the equation

$$\mathrm{NTU}_{od} = \int_{\mathrm{Top}}^{\mathrm{Bottom}} \frac{dT_d}{(T_d - T_c)} \doteq \frac{(T_{d,\mathrm{Bot}} - T_{d,\mathrm{Top}})}{\Delta T_{LM}}$$

This calculation is based on the temperature of the dispersed phase to minimize the effect of column heat losses and assumes that there is nearly true countercurrent flow in the column. From the equation it is seen that the overall number of transfer units is the number of times that the average temperature driving force will divide into the change in organic temperature. The distance over which continuous and discontinuous phases contact each other, Z, can be measured and the height of a transfer unit based on the dispersed phase can be obtained from

$$\mathrm{HTU}_{od} = {}^{Z}/_{\mathrm{NTU}_{od}}$$

As a characteristic parameter of a spray column, HTU's sometimes give a better correlation than the volumetric heat transfer coefficient. Theoretical manipulations indicate that HTU's can be arithmetically correlated with mass phase flow rate ratios. Plots of HTU vs G_d/G_c are given by McCaskill (6). He derived a correlation of HTU for the Humbletherm 500-water system.

The work of Garwin and Smith with a benzene-water system, that of Woodward with a Shell-oil and water system, and that of McCaskill with a Humbletherm 500-water system made note of, whether at any pair of flow rates, the direction of heat transfer in the column affected the volumetric heat transfer coefficient. Woodward and McCaskill both found that heat transfer coefficients were larger when heat was transferred from the continuous phase to the discontinuous phase (top of the column hot). Garwin and Smith reported the opposite result.

Sideman (15) attempted to explain Woodward's result as being due to increased backmixing caused by natural convection currents in his column. The discrepency between Woodward's and Garwin and Smith's results were explained by Sideman (15) as being caused by the large decrease in volumetric heat transfer coefficient with increased dispersed phase viscosity of Woodward's Shell-oil.

Many specific effects which are important in spray columns such as turbulence (19) and axial mising (10, 20) have been the topic of recent works.

APPARATUS

The equipment used in these experiments is presented schematically in Figure 1 and is pictured in Figures 2 and 3. A detailed description of the equipment and equipment modifications is given below.

The Column. The column was constructed of 3-in. I. D. pyrex glass pipe made by the Corning Glass Company. The column (from top to bottom) contained one 6-in. long straight section, four 12-in. long straight sections, and one 3 x 2-in. tee reducer, all connected by aluminum flanges.

The inlet water pipe (1/2-in. pipe size) was inserted concentrically 5 inches into the top of the column (see Figure 4a). The pipe had four 0.265-in. holes drilled in its sides at 90 degree intervals, 3/8-in. above its end. The end of the pipe was plugged with a flat piece of polyvinylchloride (PVC). A stainless steel screen to aid in coalescing the organic drops was mounted on the pipe two inches above the inlet holes. The screen formed jets of liquid organic at high flow rates and collected solid organic

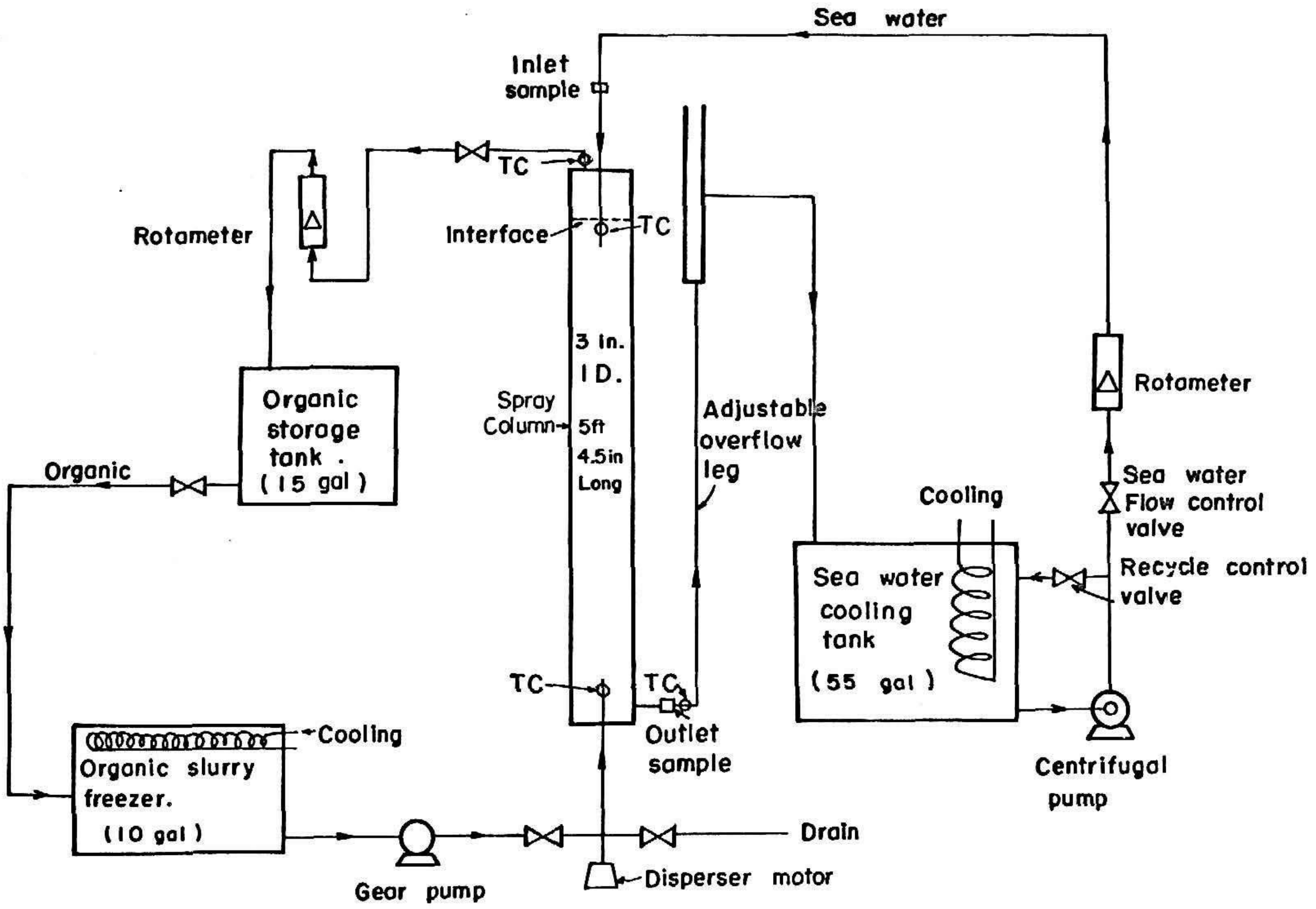

Figure 1. Schematic diagram of spray column heat transfer apparatus.

Figure 2. Front view of equipment.

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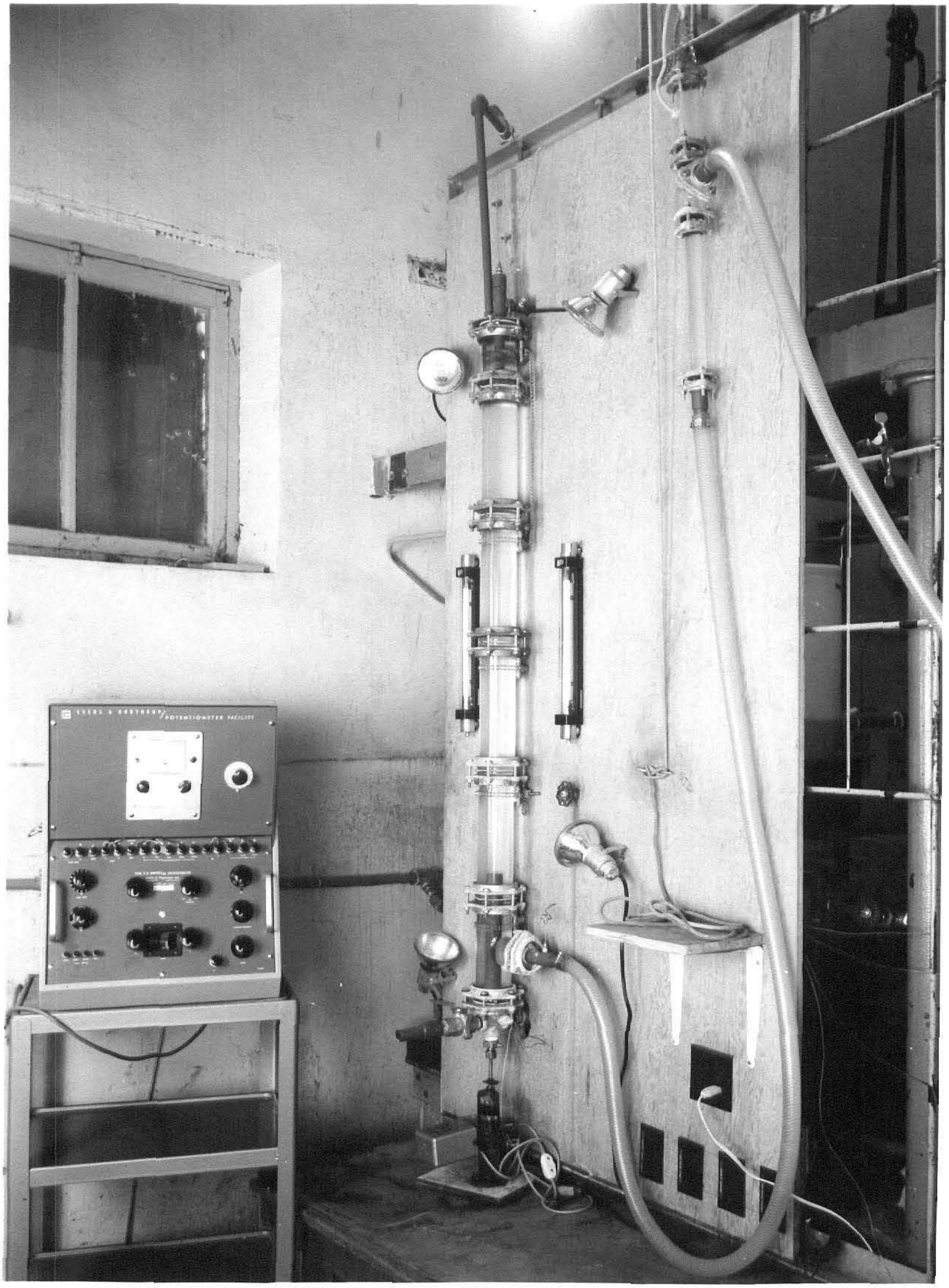
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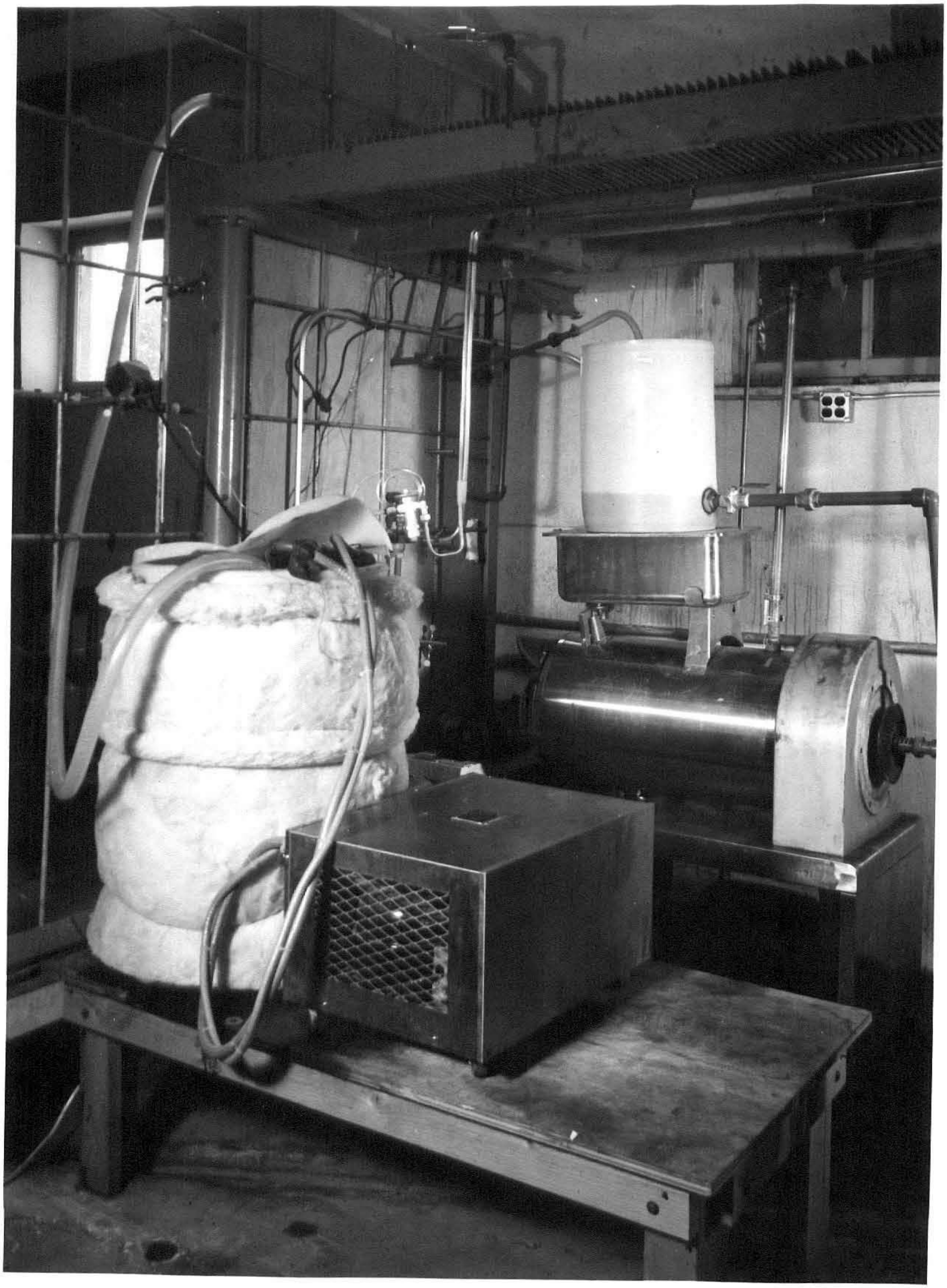
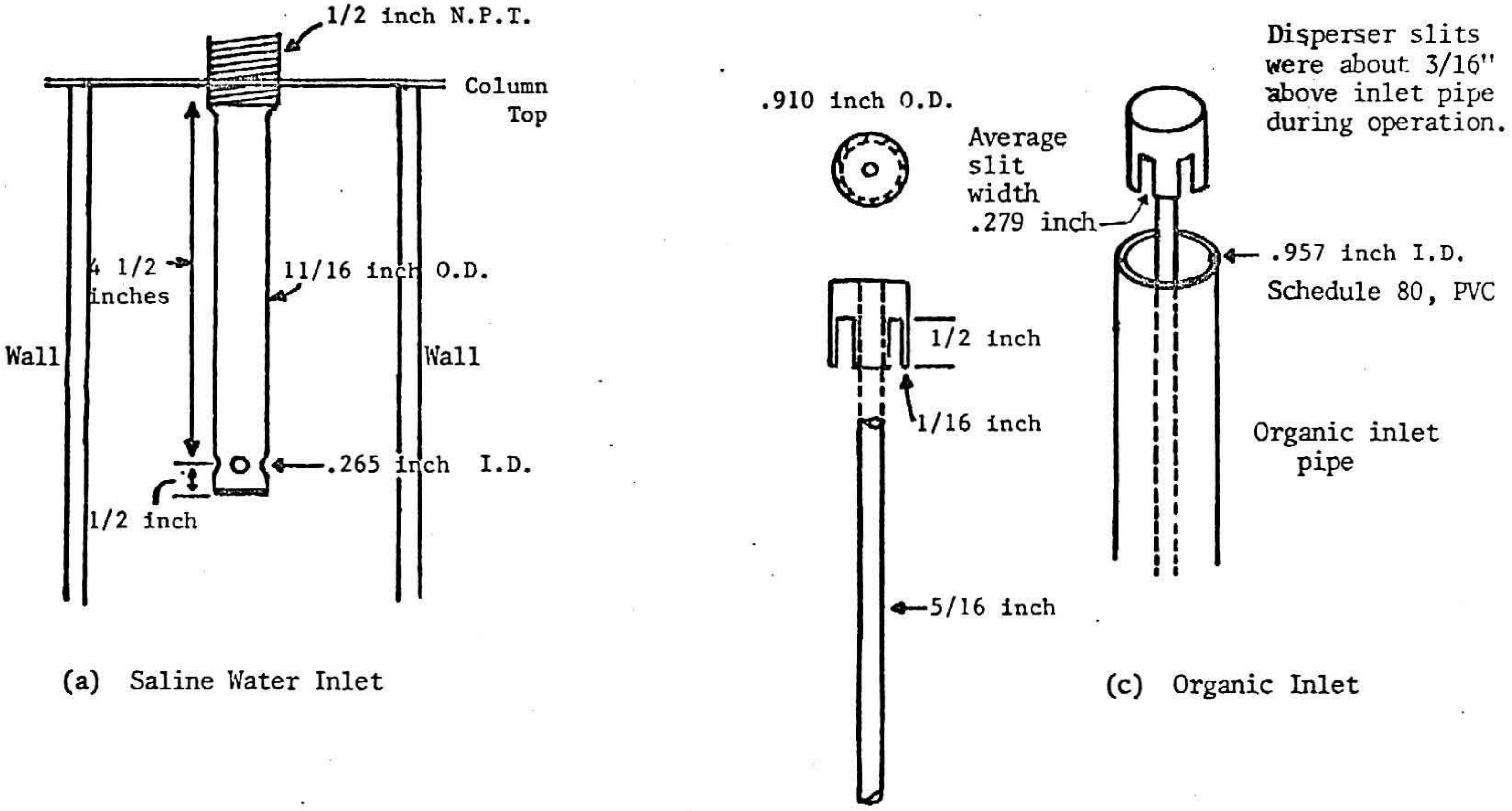
Figure 3. Back view of the equipment.





(a) Saline Water Inlet

(c) Organic Inlet

(b) Disperser

Figure 4. Saline water and organic mixture inlet pipes.

when slurry was present, so it was removed in the last stages of this work.

The organic inlet pipe was inserted concentrically 12-in. into the bottom of the column. This placed it 6-in. above the water outlet. Anticipating that the solids in an organic slurry might be trapped behind dispersing nozzles, a flat bladed (zero pitch) impeller was placed concentrically about 1/4-in. above the organic inlet to break up the incoming organic stream. Though the impeller succeeded in breaking up the stream, downcoming water filled the organic inlet pipe. This condition was intolerable as the water could freeze at the lower temperatures and plug the line.

To overcome this problem a covered disperser was designed which fit inside the pipe and could be raised or lowered to adjust the mean droplet size (see Figure 4b and 4c). This disperser was a cylindrical brass cap with four 9/32-in. wide slits cut into its perimeter. The bottom of the disperser always remained in the inlet pipe.

During the final stages of this work, smaller temperature differences were used, and insulation was required. The column was surrounded by 2-in. thick styrofoam on 3 sides and by 1-in. styrofoam in back. The void spaces were filled with spun fiberglass.

Adjustable Overflow Leg. In order to keep the organic-saline water interface at a constant position for all flow rates, some type of pressure controller was required on the exit aqueous line. A needle valve could not be used since the desired ice product would jam up behind it. An adjustable overflow leg was chosen to control the pressure on the effluent aqueous line. The adjustable overflow leg was made of 1-in. Tygon tubing attached to a 1-in. glass pipe and tee which were made by Corning Glass Company. The adjustable overflow leg was positioned by use of an overhead pulley (see

Figure 2).

<u>Organic Slurry Freezer</u>. The organic slurry freezer was a 10 gallon Emery Thompson (Model 40 HF) soft ice cream machine. The whipper blades were removed so that the organic line could enter through the rear center of the vessel. Some leakage was noted underneath the machine which may have been due to a poor seal at the rear of the freezing vessel.

The scraper blades inside the vessel provided agitation to produce a homogeneous slurry. The blades were not generally used when only liquid organic was desired.

The front door on the ice cream machine was sealed by making a gasket of silicone rubber which adhered to the door. The door was then secured by 6 screws which pressed it into place. The silicone rubber tended to loosen from the metal by action of the organic and was not very satisfactory. A 1-in. hole was tapped into the door to provide an organic outlet.

<u>Tanks</u>. The polyethylene 15 and 50 gallon storage tanks, for the organic (15 gallon) and the saline water (50 gallon) were purchased from Cole-Parmer. Outlet lines and the salt water control line entered the tanks through PVC bulkhead fittings.

<u>Pumps</u>. The (3/4-in. x 1/2-in.) bronze centrifugal saline water pump and coupled motor were made by Oberdorfer and supplied by S. Gelber & Sons. (Product No. 4GCC). The 1-in. bronze gear pump on the organic slurry line was made by Oberdorfer and supplied by McMaster Carr (#4271N16).

The gear pump was powered by a 1/2 HP. (220 Volt A. C., 1725 rpm) motor made by General Electric. The motor speed was reduced before going to the pump by a Zero-Max continuously variable (0-400 rpm, 25 in-lb_f/torque) speed reducer. When liquid organic was pumped, a direct connection was made between the speed reducer and the pump. When organic slurry was pumped, it was necessary to connect the pump to the speed reducer through a 3:1 belt pulley system so that the speed reducer's maximum torque (25 in-lb_f) was not exceeded. The flow rate of organic was always varied by changing the variable speed reducer.

Rotameters. The rotameters were made by Fischer and Porter. These stainless steel rotameters (series 10A3500) had a maximum range of 0 to 1.5 gpm for the saline water and of 0 to 1.0 gpm for the organic phase. The rotameters could be read to ± 0.005 gpm and were provided with external scales calibrated for the expected liquid densities (1.027 for salt water and 0.765 for the organic). The rotameters were calibrated in place and the calibrations are presented in Appendix B. When organic slurry was pumped to the column it became necessary to remove the organic rotameter bob and measure each organic flow rate by timing the volume of flow out of the top of the column.

Fittings and Valves. PVC fittings filled most needs. An exception was at the bottom of the column where a cracked PVC cross was replaced by a steel cross.

The two control valves shown in Figure 1 were bronze globe valves. All other valves were used in on-off applications and were bronze ball valves.

Process Piping. Whenever some solid content was anticipated, 1-in. piping was used. Thus, the piping between the organic freezer and the column, and all lines on the overflow leg were 1-in. In all other applications, smaller diameter pipe (generally 1/2-in.) was used.

Tygon tubing was used whenever flexibility or a short length easily disconnected was desired. The lines on the overflow leg, the lines between the slurry freezer and the column, and the line between the organic rotameter and the organic storage tank were made of Tygon tubing. The Tygon was connected to solid piping with hose clamps. Most stationary piping was schedule 80 Polyvinylchloride (PVC) made by McMaster Carr. None of the piping was insulated.

Cooling. The main cooling unit was a Freon compressor (not shown) which supplied Freon to the shell of the ice cream machine and to a 50-foot long epoxy painted copper cooling coil in the salt water tank.

To control the temperature of the organic pumped to the column, an on-off temperature controller was initially employed to open and shut the ice cream machine expansion valve. The temperature of the organic fluctuated too widely (about 15 times the span of control) and the controller was removed. To control the pressure in the ice cream shell and in the sea water coil a pressure regulating valve made by the Controls Company of America was employed. Thus the operating temperatures were fixed by operating conditions once the pressure regulating valve was set.

Additional cooling was available for the saline water through use of a portable cooling unit (Model PCC-3A, 250 watts, 115 volts, temperature range -23° to +5° C.) made by the Blue M Electric Company.

Organic Disperser Motor. After a few months of operation, salt water and oil leaking through the organic disperser bearing had made the first organic disperser motor inoperable and it was replaced by a Lighting Model F Mixer (220 volts) for the duration of this work.

Thermocouples. All thermocouples were Copper-Constantan, Type 5D0811S sheathed in stainless steel (O. D. = 1/16-in.), made by the Thermo Electric Company.

During the initial stages of this work the thermocouples were all joined to a common ice bath junction. A short circuit developed, however, and when it could not be traced, a separate ice junction was made for each thermocouple.

The bottom organic thermocouple (14-in. immersion length) was inserted into the bottom plate, through the water, and then through the wall of the inlet organic tube about 2-in. below the organic inlet. Many fine organic drops were emitted from the hole where the thermocouple entered the tube. The edges of the hole were sealed with epoxy glue before any data was taken. The tip of the inlet organic thermocouple was positioned 3/16-in. from the wall of the pipe.

The outlet (bottom) water thermocouple (11-in. immersion length) was initially inserted 10.5-in. into the column and was positioned 3/4-in. from the column wall. With this arrangement, however, we could not reproduce heat transfer coefficients from which to evaluate heat losses. It was further expected that the temperature measured would not be the cup mixed temperature due to the channeling at low flow rates. Thus the outlet water temperature was measured by a thermocouple mounted in the outlet aqueous line about 2-in. from the outer edge of the tee.

The outlet (top) organic temperature was initially measured by a thermocouple (8-in. immersion length) inserted into the column 3/4-in. above the interface and 1/2-in. from the column wall. This thermocouple was used in the first part of this work. It was later replaced by a thermocouple (3-in. immersion length) located in the outlet organic line 1.25-in. above the column. This change was made to insure that we had a cup mixed temperature and to insure that solid organic would not cling to the thermocouple.

The inlet water temperature was measured by a thermocouple (immersion length of 14-in.) which was inserted concentrically in the inlet water pipe. The tip of this thermocouple was generally visible in the water inlet holes (always within 1/4-in. above them).

Potentiometer. To read the thermocouples an L & N K-3 Potentiometer facility was used (see Figure 2). This facility is ". . . a guarded system comprising the 7553-6 Type K3 Universal Potentiometer, the 9834-1 Electronic Null Detector, and the 099034 Constant Voltage Power Supply." On calibrating the thermocouples against an ice bath standard, it was found that the thermocouples read within ± 0.0002 millivolts of the ice standard. The limits of error for the potentiometer facility were ± (0.015% of reading + 0.5 μv).

Conductivity Meter. The salinity of the aqueous phase was measured by a conductivity meter. The conductivity meter was a Type RA5 Solu Meter made by Beckmann Instruments, Incorporated. Since this meter was used in conjunction with a conductivity cell whose constant = 10, the meter's scale had a range of 0 to 5,000 ± 20 micromhos/cm.

PROCEDURE

Saline Water Preparation. The saline solutions were prepared from city water and U. S. P. NaCl. The salt concentration was measured by Beckmann Conductivity Meter described previously.

To make a concentration determination, 10 ml. of the saline solution was poured into 250 ml. of distilled water. The diluted solution was then tested at about 23° C. and the salt concentration obtained from the calibration (Figure 15 in Appendix B).

A 3.3 weight-% salt solution was used in the first portion of this work (when the largest log-mean temperatures were used), and a 3.5 weight-% salt solution was used in the latter part. Physical properties of the saline solutions were taken from (16) and (17).

Organic Mixture Preparation. The 52 volume-% n-tridecane ($C_{13}H_{28}$) 48 volume-% n-tetradecane ($C_{14}H_{30}$) mixture was prepared from technical grade n-tridecane and technical grade n-tetradecane purchased from the South Hampton Company, Houston, Texas. The n-tridecane and n-tetradecane were mixed using a 2 liter graduate cylinder. The physical properties of n-tridecane and n-tetradecane are given in Appendix C.

Startup Procedure. The potentiometer which was used to read the thermocouples was always turned on one hour prior to an experimental run. The potentiometer was standardized at the commencement of operations and the thermocouple reference junction was placed in a distilled water-ice slurry.

All the experimental data was taken at temperatures below the ambient (from 20° C. to -3° C.). To cool down the fifty gallons of saline water, fifteen gallons of organic mixture, and all the piping in the system to the desired operating temperature as rapidly as possible, the following procedure was used: First, the water circulation control valve was opened and the water pump turned on to recirculate the saline solution through the tank. Next the water valve to the column was opened and the water overflow leg adjusted to keep the oil-water interface constant at 2.5-in. below the top of the column. With saline water circulating through the entire system, the Freon compressor was started to provide cooling to the heat transfer fluids. For small log-mean temperature differences, the Freon valves to both the organic slurry freezer and to the saline water cooling coil were opened. For larger differences, only one of these Freon valves was opened, the choice depending on the desired direction of heat transfer. Whenever Freon temperatures near the freezing point of the organic mixture were encountered, the scraper blades in the organic slurry freezer were activated.

To achieve initial hydrodynamic stability, it was found that it was easiest to set the organic flow rate first before letting water flow through the column. Thus, when the system temperatures were within 5° F. of desired operating temperatures, the water valve to the column was shut and the organic mixture was started circulating through the column. To start the organic flow, the valve between the gear pump and the column was opened and the gear pump started. With the organic disperser plugging the inlet organic pipe, the drain valve was opened to insure that any accumulated water was discharged from the organic inlet line. Then the drain valve was shut, the organic disperser was raised to the desired slit height, and the overflow leg was adjusted. Next the organic disperser motor was started and its speed set at 136 rpm., which was the speed used in every run. Finally, the water valve to the column was opened, set at the initial flow rate, and a final adjustment made on the overflow leg to keep the organic-water interface 2.5-in. below the top of the column.

Operating Procedure. It was experimentally determined that the system took five minutes to reach a thermal steady state after the organic and saline water flow rates were set. Thus, five minutes after hydrodynamic stability was achieved the four thermocouples (inlet and outlet organic temperatures and inlet and outlet water temperatures) were read. All four thermocouples could be read within two minutes. In every case, the organic flow rate was maintained constant until all values of saline water flow rate had been used and the temperature data recorded. To conduct another run, a new organic flow rate was chosen and data taken over the entire range of saline water rates.

DISCUSSION OF SPRAY COLUMN OPERATION

The purpose of this research was to build a spray column and operate it with four phases (organic solid, organic liquid, saline water, and fresh water ice) present in a desalination operation. The organic mixture was composed of 52 volume-% tridecane and 48 volume-% tetradecane. Saline solutions of 3.3 weight-% NaCl and 3.5 weight-% NaCl were used. Due to time limitations, the four phase operation was never achieved but a column was built and operated with three phases present (organic solid, saline water, and organic liquid).

The organic inlet disperser was not designed like a nozzle because it was anticipated that solids would become clogged behind it. At the outset, a flat bladed impeller set 1/4-in. above the organic inlet leg was used; it was removed and replaced by the cap disperser (see Figure 4b) when downcoming water filled the organic inlet leg. With the cap disperser operating at 3/16-in. slit height and at one set of flow rates, a photographic determination of drop size was made, taking into account the distortion introduced by the curved column according to Garwin's (8) technique. The drop size was expected to vary with dispersed phase flow rate at constant slit height, with continuous phase flow rate, and with temperature of the fluid. Average drop size determinations were not made for every run. It was expected that drop size would not vary strongly with continuous phase flow rate and since the fluids in the column became opaque at low temperatures (0° C.), it was decided to rely on the implication of the slit height parameter to explain any major changes in heat transfer coefficients.

The slit height of 3/16-in. was chosen because that height produced drops that were quite uniform in size, with no slugs or fines. The disperser cap was set on a rotating rod positioned concentrically in the organic inlet pipe to counteract anticipated streaming of the organic slurry. Though the cap was hollow and had no radial vanes installed, its rotary motion tended to shear the upflowing organic stream as it passed through the slit and influenced drop formation. The disperser's rotary speed was held constant during the experiment at 136 rpm, a value picked empirically. The rotary motion proved necessary for liquid runs and especially for slurry runs, where solids tended to become clogged as expected.

The rotary motion of the cap helped insure that the drop distribution had no angular prejudice by spewing drops in all directions. Except for a bottom end effect, the drops were observed to be flowing at all radial positions in the column, and no radial distribution function was obvious. The bottom end effect was involved at low continuous and discontinuous phase flow rates where the drops seemed to move first towards the column center, then out to all positions under the influence of the continuous phase motion.

Due to the difficulty of fabrication, the column itself was not of the Elgin type. Rather, it was constructed of sections of pyrex (3-in. I. D.) glass pipe, with the diameter constant throughout. This construction caused some concern, since two types of solids, organic and ice, were expected at the bottom of the column, which should be of a larger diameter in accordance with the preferred Elgin design. The constant diameter column handled the three phase (solid organic, organic liquid and saline water) operations very well and also operated satisfactorily when four phases were present in the column.

The saline water inlet nozzle was built in accordance with Woodward's (4) design and is presented schematically in Figure 4a. The interface was held at a constant position 2 1/2-in. above the four 1/4-in. water inlet holes; in this position the incoming water caused very little agitation of the organic interface. The large diameter of the water inlet holes helped reduce the pounding of upflowing organic drops to a minimum. Of course, a slight swirling top end effect was present. To avoid flooding at the bottom of the column the pumps were chosen with flow rates less than 50% of the flooding velocity predicted by Sakiadis and Johnson's (5) generalized flooding correlation. In fact the column never did flood, though there was considerable backmixing and agitation or "turbulent behavior" (see page 3) at the maximum flow rates. Heat transfer data was obtained in the region of "pseudo-ideal behavior."

A column I. D. of 3-in. was chosen to minimize wall effects while remaining within the limits of laboratory size equipment. During operation, the droplets were frequently forced against the side of the column but did not seem to stick nor was their upward motion greatly impaired.

The size of the piping had been set at 1-in. wherever solids had been expected. A gear pump was selected to handle the organic slurry. The organic solids did sometimes clog up behind the rotating pump when the organic was not moving through the transparent suction line. There was no difficulty with organic slurry or with ice in any other portion of the piping system. Another consideration of system design was the question: At what ice particle size and at what brine velocity will ice begin to rise through the column under the influence of buoyancy forces? A preliminary calculation using

Stoke's law indicated that the rising of ice particles would not occur. Ice particles of 0.5-mm diameter were present in the column during some of the runs and they all passed directly out of the column through the overflow leg, fulfilling the expectations of Stoke's equation.

The overflow leg was acceptable except in one facet of this investigation. At organic temperatures of about 0° C., the organic-water interface tended to remain at a stable height in the column (eg. the organic may have been sticking to the side, having become a frozen block). When any solid organic was present above the interface, the organic tended to set up in the manner of a thixotropic fluid (Bingham plastic). That is, to keep the interface from dropping, the overflow leg had to be raised up a 2-foot increment in order to start the organic flowing out of the column again. Once flowing, the organic flow rate went to a very high value and it was difficult to keep the interface in the column.

The organic rotameter was useless when any solids were present in the stream leaving the column. In these cases the bob had to be removed and each organic flow rate measured by timing a sample volume. With the rotameter bob removed there was no indication if the organic rate changed slightly and so it was assumed constant as long as the Zero-Max speed controller had not been adjusted.

The coalescing screen (4) which was placed in the column hindered the stability of the column's flow regime. That is, when the interface rose above the screen at high organic flow rates, the screen formed jets of organic through the continuous saline phase and made recoalescence of the jets more difficult. The screen was expected to become clogged if any solids passed it so it was finally removed.

DISCUSSION OF RESULTS

The original data are presented in Appendix D.

Heat transfer coefficients for heat transfer from the continuous saline water phase to the dispersed organic phase are shown in Figure 5. The data at a dispersed phase flow rate, G_d, of 2,648 lbm/(hr-ft^2) (slit height = 3/16-in.) fell well below the data at G_d = 5,670 lbm/(hr-ft^2) and the same slit height. This result is expected since an increased dispersed phase flow rate will give increased holdup.

The data at G_d = 4,486, (slit height = 4/16-in.) generally falls well below that at G_d = 5,210 and the same slit height. However, at the low end of the range of continuous phase flow rates the heat transfer coefficient is almost identical for both cases.

The data at G_d = 4,486, (slit height = 4/16-in.) lie well above the data at G_d = 2,648, (slit height = 3/16-in.). Thus, the increased holdup produced by a larger dispersed phase flow rate has overcome the effect of increased specific surface area (surface area of dispersed phase/volume of dispersed phase) caused by the smaller slit size. Moreover, the data at G_d = 5,670, (slit height = 3/16-in.) lies slightly higher than that for G_d = 5,210 (slit height = 4/16-in.) as is expected for specific surface area and holdup considerations. However, the difference is so minute that neither effect seems very large at these flow rates.

A comparison of our data with that of Garwin and Smith (8) for heat transfer from the continuous saline water phase to the dispersed organic phase is also shown in Figure 5. Our data at a dispersed phase flow rate of 2,648 lbm/(hr-ft^2), (drop diameter = 0.32-in., slight height = 3/16-in.) fell well below that of Garwin and Smith at a dispersed phase flow rate of

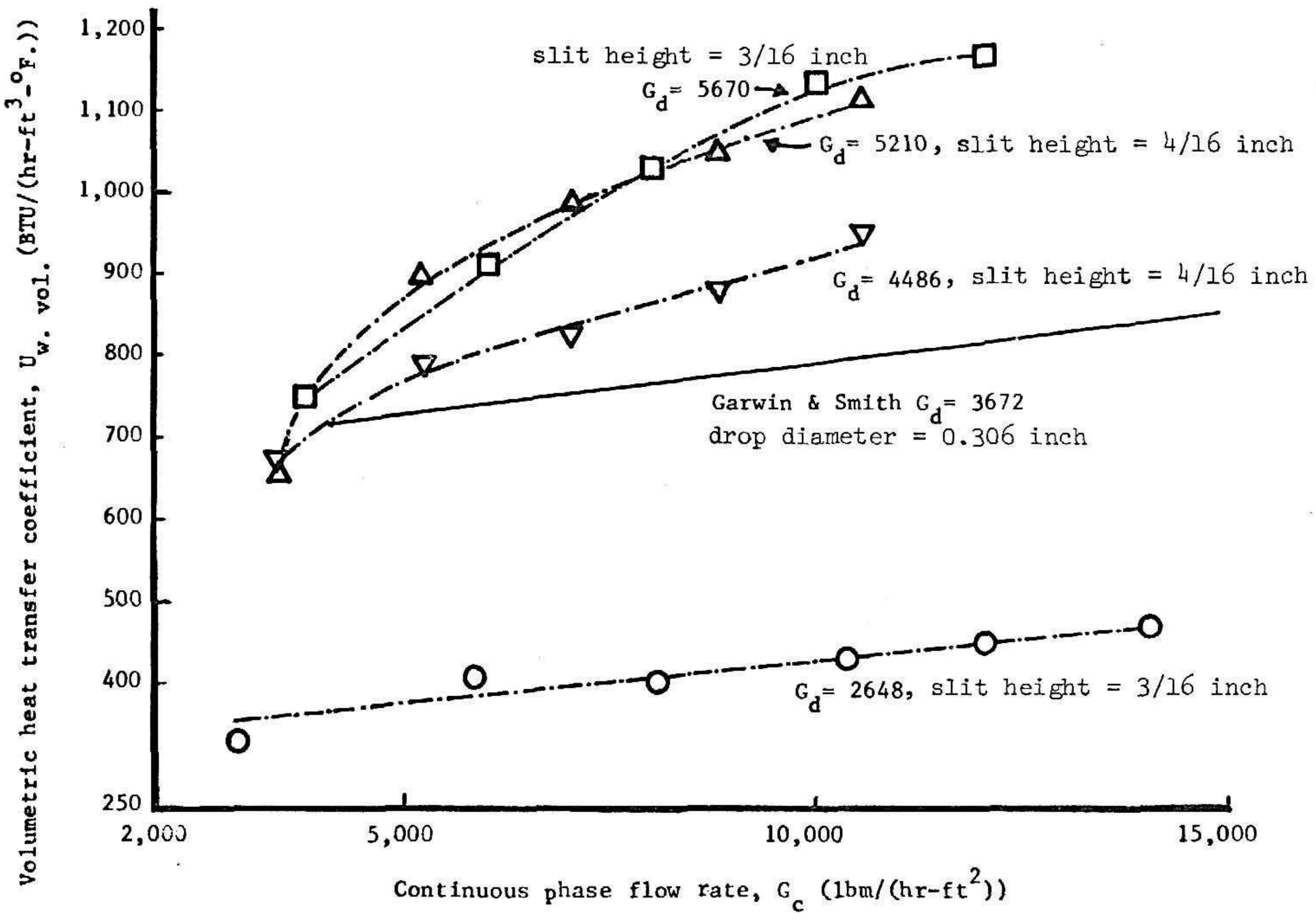


Figure 5. Effect of continuous phase flow rate on volumetric heat transfer coefficient at different dispersed phase flow rates. *The direction of heat transfer was from the saline water to the dispersed organic phase; no solid phases present.*

3,672 lbm/(hr-ft^2) (drop diameter = 0.306-in.). This result is expected from considerations of both flow rate and drop size. The data at a dispersed phase flow rate of 4,486 lbm/(hr-ft^2), (slit height = 1/4-in.) fell slightly above that of Garwin and Smith (G_d = 3,672, drop diameter = 0.306-in.) which indicates that the large dispersed phase flow rate raised the heat transfer coefficient despite Garwin and Smith's slightly smaller drop size.

Liquid-liquid heat transfer in the opposite direction, from the dispersed organic phase to the aqueous phase is the subject of Figure 6. The data at G_d = 3,473 lbm/(hr-ft^2), (slit height = 3/16-in.), lie well below the data at G_d = 6,510 and the same slit height. The data at G_d = 5,210, (slit height = 1/4-in.) lie only slightly above the data at G_d = 3,473 which seems to indicate that the increase in slit height has had a large effect on the column's heat transfer effectiveness.

For the case of heat transfer from the dispersed organic phase to the continuous aqueous phase the correlations through our data are compared with that of Garwin and Smith (8) and that of McCaskill (6) in Figure 6. At a low dispersed phase flow rate, G_d = 3,473 lbm/(hr-ft^2), (slit height = 3/16-in.), the data fell just below McCaskill's correlation for G_d = 4,002 (average drop diameter = 0.252-in.). The result was anticipated since our dispersed phase flow rate was lower (lower holdup) and our drop size was larger (provided less heat transfer surface per unit volume of organic).

Garwin and Smith's correlation for a dispersed phase flow rate of 3,672 lbm/(hr-ft^2), (drop diameter = 0.281-in.) fell considerably higher than our data at a dispersed phase flow rate of 3,473 lbm/(hr-ft^2). This was an expected result since Garwin and Smith's flow rate is larger and their drop size is smaller.

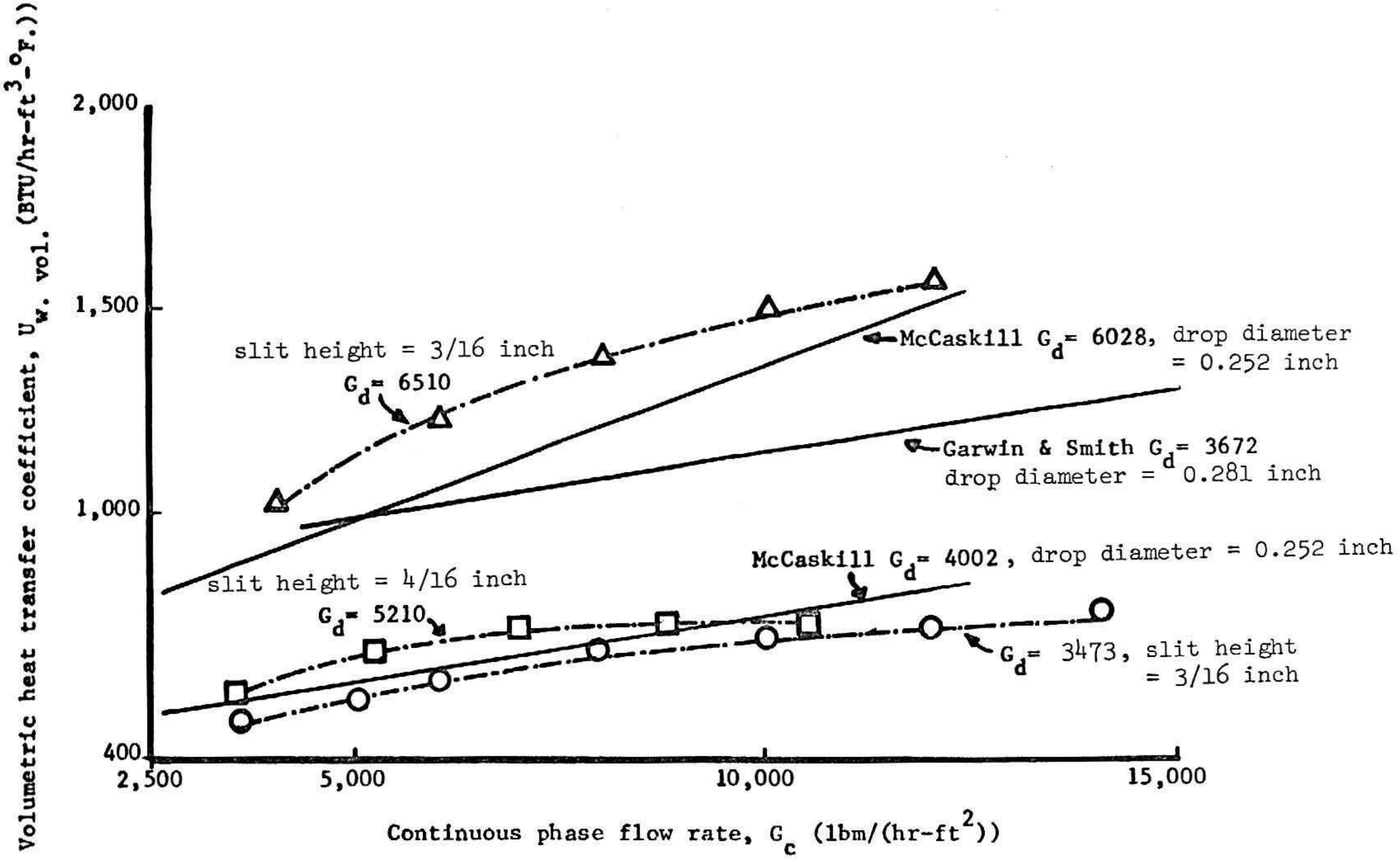


Figure 6. The effect of continuous phase flow rate upon volumetric heat transfer coefficient at different dispersed phase flow rates. *The direction of heat transfer is from the dispersed organic phase to the saline water; no solid phases are present.*

It is noted that Garwin and Smith's correlation at G_d = 3,672 lbm/(hr-ft^2), (drop diameter = 0.281-in.) lies considerably higher than McCaskill's correlation at G_d = 4,002, (drop diameter = 0.252-in.). Consideration of increased heat transfer area due to smaller drop size and increased holdup at higher dispersed phase flow rate contradict this result. The contradiction can possibly be explained by the larger wall effect (greater holdup) in Garwin and Smith's smaller (2-in. I. D.) column.

Continuing the analysis of our correlations in Figure 6 it is noted that our data at a dispersed phase flow rate of 6,510 lbm/(hr-ft^2), (slit height = 3/16-in.) lie slightly above McCaskill's correlation at a dispersed phase flow rate of 6,028 lbm/(hr-ft^2), (drop diameter = 0.252-in.). This result could be inferred from the fact that our flow rate is higher but this effect would be countered by our larger drop size which tends to lower the heat transfer coefficient. The data at G_d = 6,510 is 45% lower than the data of Garwin and Smith's for G_d = 6,290 lbm/(hr-ft^2) drop diameter = 0.271-in. (not plotted). Garwin and Smith's volumetric heat transfer coefficients at G_d = 6,290 increased with continuous phase flow rate 260% faster than ours at G_d = 6,510. These two results emphasize the importance of wall effects in Garwin and Smith's 2-in. I. D. column.

Near the termination of this work, equipment wear made it increasingly difficult to operate at a disperser height of 3/16-in., and a 1/4-in. height was used. One set of data taken at a disperser height of 1/4-in. and a dispersed phase flow rate of 5,210 lbm/(hr-ft^2) is shown in Figure 6. It can be seen that this data is only about 6% greater than the data taken at G_d = 3,473 and a slit height of 3/16-in. The very slight increase of volumetric heat transfer coefficient with a 50% change is dispersed phase flow rate at the largest slit height is taken as evidence of a drop size increase with the increased slit height.

The data presented in Figure 7 were taken at a constant slit height and constant organic rate at two different conditions: The top of the column hot (cold organic) and the top of the column cold (hot organic). The data for the top of the column hot (cold organic) lies well above the data for the top of the column cold, in agreement with Teynham Woodward's work (4), which was done for smaller drops.

The result in Figure 7 is contradicted by the work of Garwin and Smith however. Garwin and Smith (8) reported smaller volumetric heat transfer coefficients at a constant organic flow rate, G_d = 3,672, with the top of the column hot (cold organic). Garwin and Smith's results may be consistent within themselves, however, since their apparatus produced smaller drops (greater interfacial area) for the top of the column hot (cold organic operation).

Sideman's (7) attempt to explain this apparent discrepancy between Garwin and Smith's result and Woodward's result is presented in the literature survey.

The case of the heat transfer between an incoming organic slurry and warmer aqueous phase is presented in Figure 8. The data at G_d = 5,819, (slit height = 4/16-in.) lies well above the data at G_d = 4,517 and the same height. Thus, as in the case of heat transfer between liquids, increased holdup has increased the heat transfer coefficient.

Figure 9 shows the effects of the different organic phase solid contents upon heat transfer coefficient when slit height and dispersed phase flow rate are held constant. It must be pointed out that the heat transfer coefficient

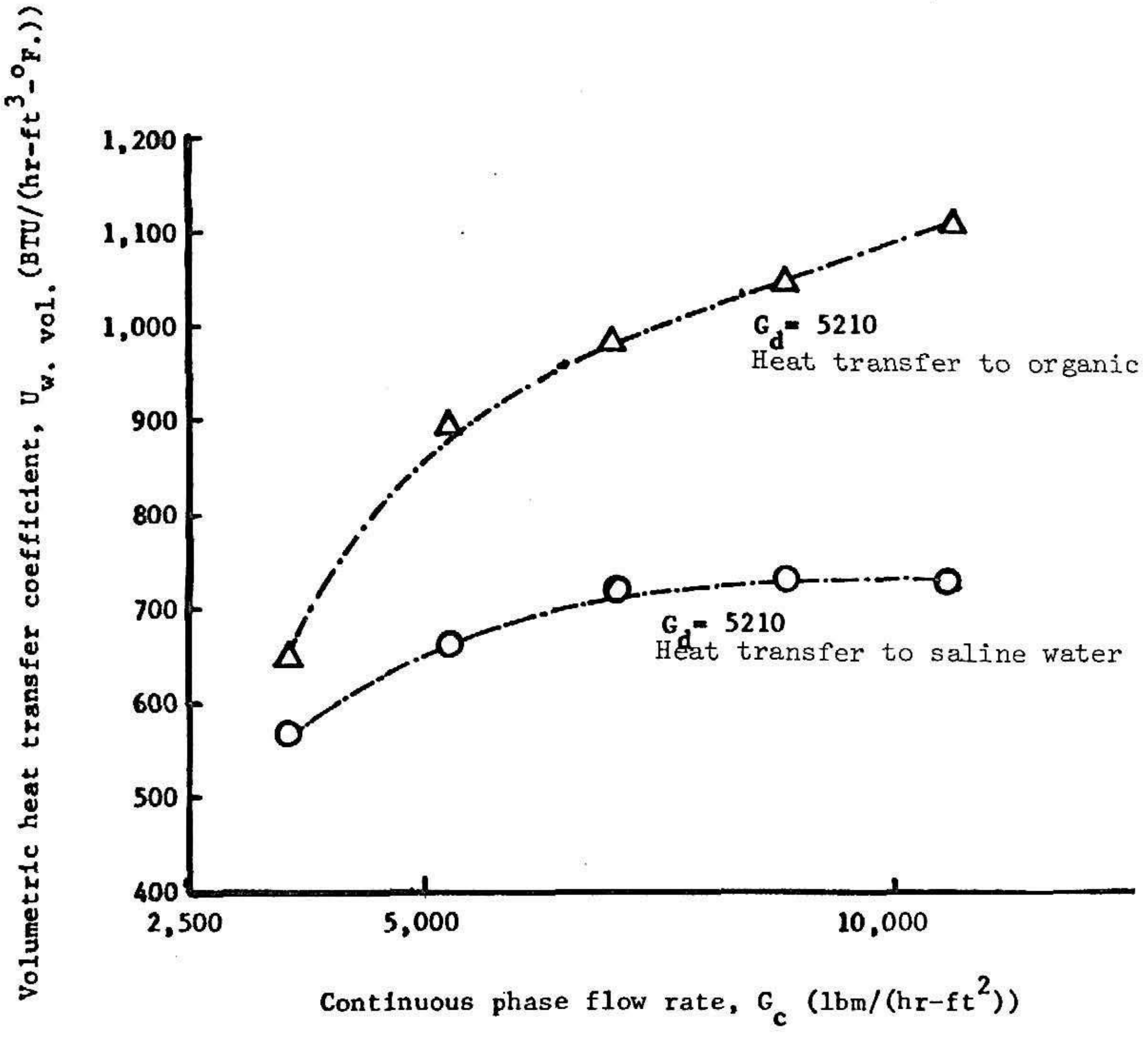


Figure 7. Effect of continuous phase flow rate on volumetric heat transfer coefficient at a constant dispersed phase flow rate. *Heat is transferred to organic and to water; no solids are present.*

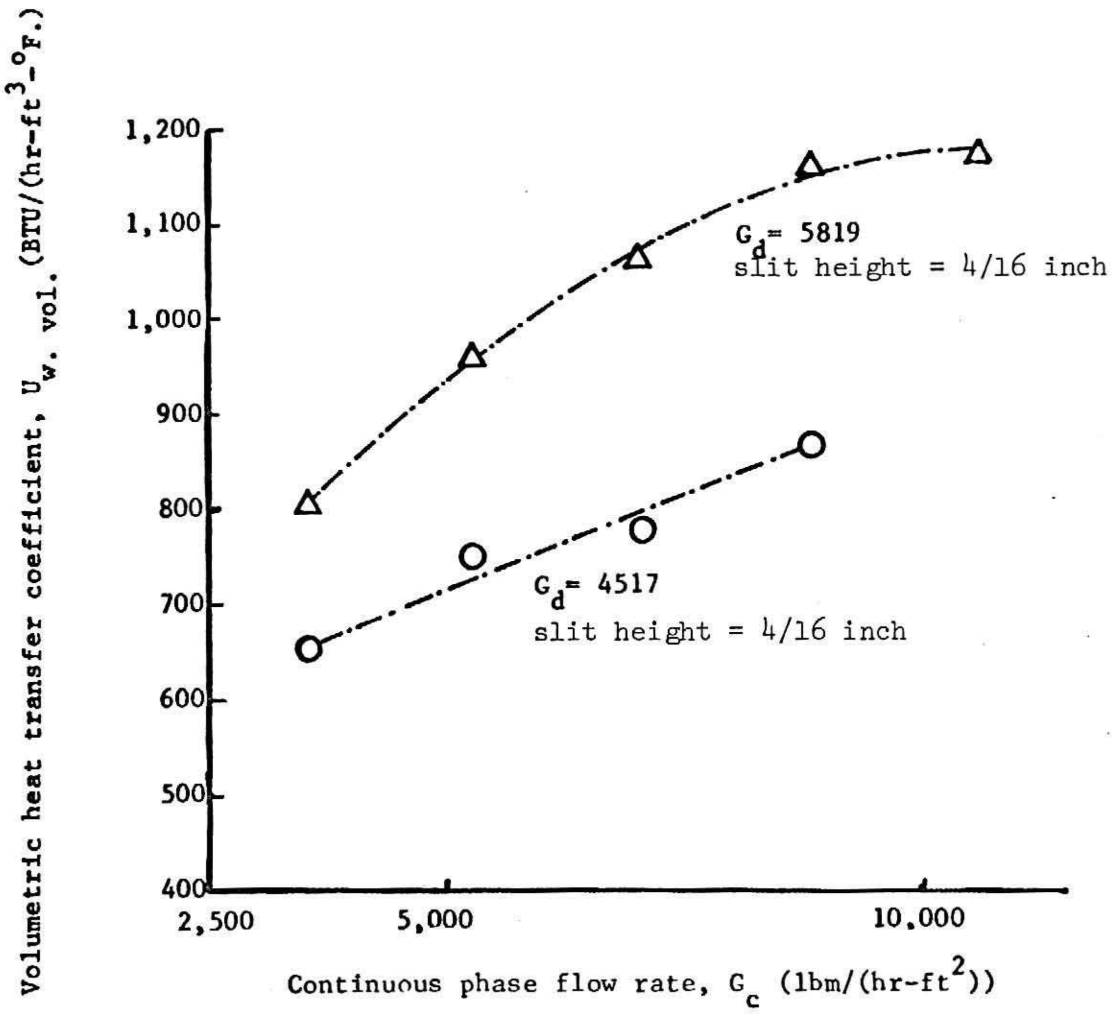


Figure 8. The effect of continuous phase flow rate on volumetric heat transfer coefficients at two different dispersed phase flow rates. *The direction of heat transfer is from the saline water to organic and solid organic is present in the entering stream only.*

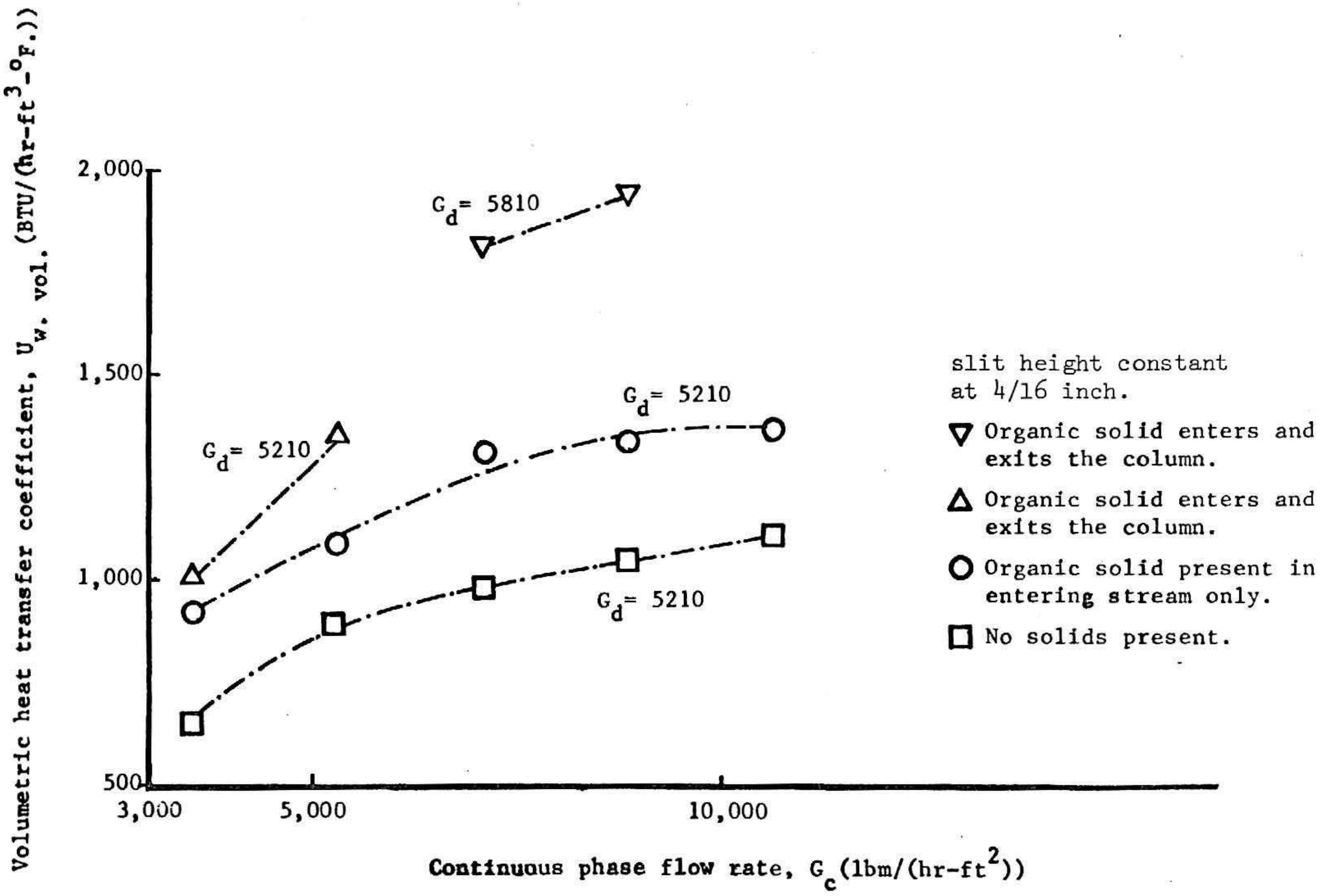


Figure 9. Effect of continuous phase flow rate on volumetric heat transfer coefficient for various dispersed phase solid contents at a constant slit height. *The direction of heat transfer is from the saline water to the organic.*

in the slurry case were calculated from the change in water temperature over the column, since the necessary thermodynamic data was not available for the organic slurry.

With no organic solids present in the column the lowest coefficients are obtained. A 30% increase in heat transfer coefficient is obtained when organic slurry enters the column and organic liquid is exhausted. It is seen that another increase in heat transfer coefficient was obtained when organic solids were present in both entering and exit streams. These increases do not necessarily indicate an improvement in heat transfer in the column, since the log mean temperature difference will be smaller than the actual temperature difference whenever slurry is present.

The correlations between the overall height of a transfer unit and the mass flow rate ratio, G_d/G_c, at different dispersed phase flow rates are seen in Figures 10 and 11. At any given continuous phase flow rate and constant slit height, there is a decrease in HTU (e.g. improvement in heat transfer) with increased dispersed phase flow rate. This result is due to the increased interfacial contact area which accompanies increased holdup in the column.

It is also notable that in Figure 10 the data obtained for a slit height of 1/4-in. lies near or below the data taken at $G_d = 5,670$, (slit height = 3/16-in.), despite the fact that the dispersed phase flow rates are smaller. This decrease in HTU may be due to an increased internal circulation in the larger droplets.

In Figure 11, the opposite result obtains, wherein the data taken at $G_d = 5,210$, (slit height = 1/4-in.) has a greater HTU, than any data taken at a 3/16-in. slit height. This result can only be attributed to the smaller

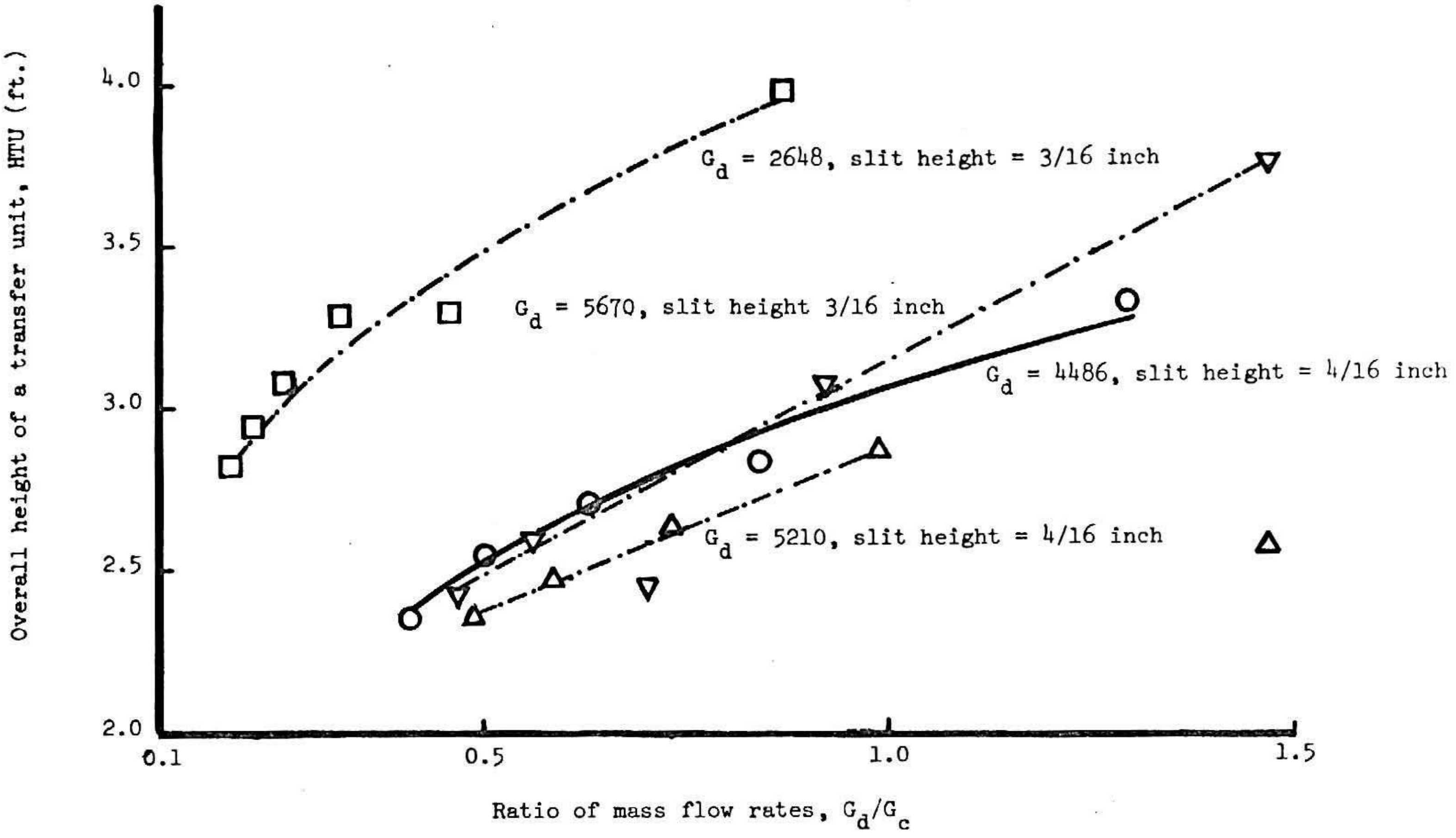


Figure 10. Effect of mass flow rate ratio on overall height of transfer unit at different dispersed phase flow rates. *The direction of heat transfer is from the saline water to organic; no solids present.*

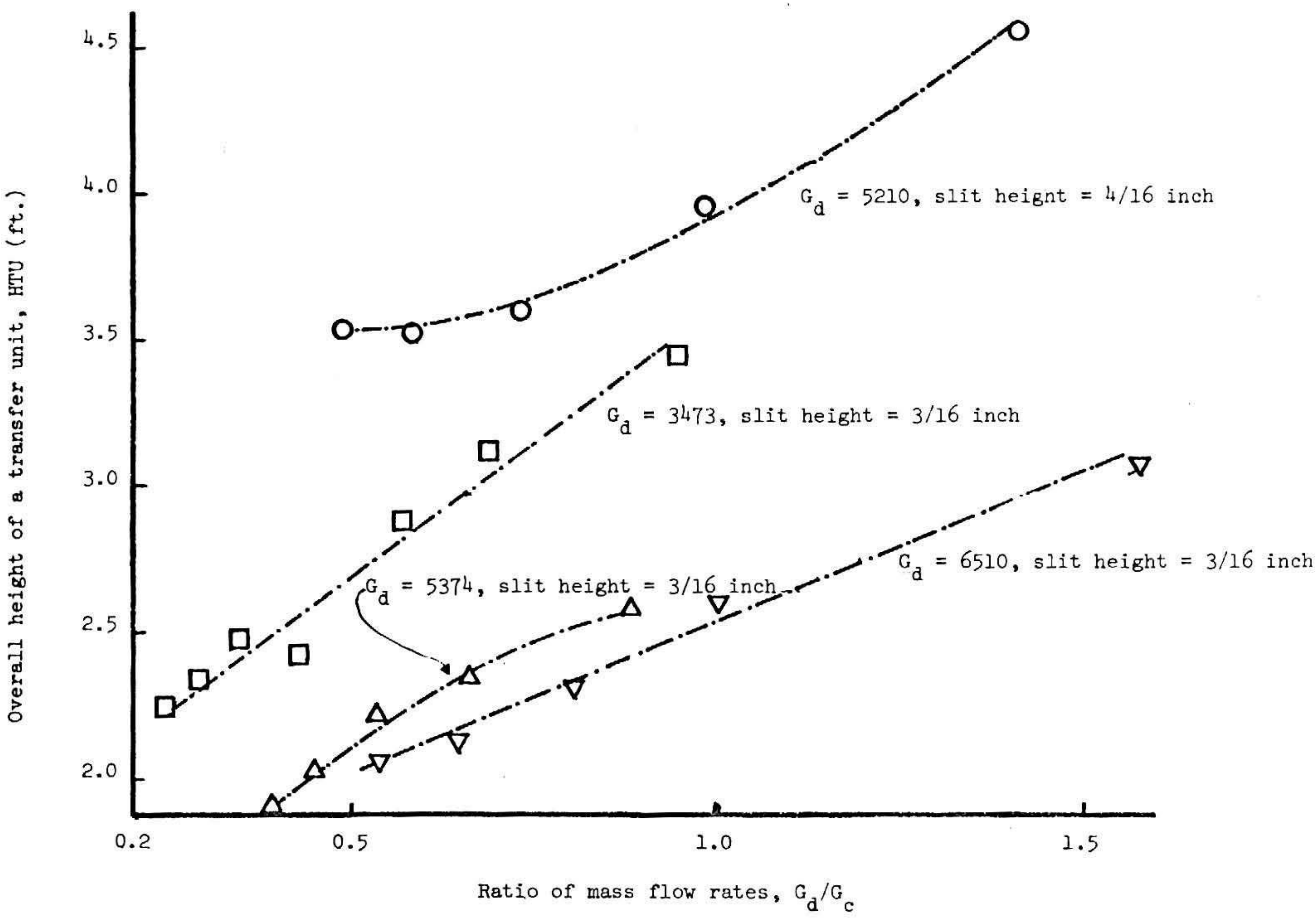


Figure 11. Effect of mass flow rate ratio on overall height of transfer unit at different dispersed phase flow rates. The direction of heat transfer was organic to saline water; no solids present.

drop size and greater interfacial area which would prevail at the smaller slit height.

CONCLUSIONS AND RECOMMENDATIONS

The spray column heat transfer data for the 52 volume-% n-tridecane ($C_{13}H_{28}$) - 48 volume-% n-tetradecane ($C_{14}H_{30}$) and saline water system reported here indicate that this system has heat transfer characteristics similar to other two phase (water and liquid organic) systems which have been investigated recently (4, 6, 8). Three phase (solid organic, liquid organic, and saline water) operations caused some improvement in the volumetric heat transfer coefficient. Stable four phase (solid organic, liquid organic, saline water, and fresh water ice) operation was not obtained.

To extend this work into four phase operations the following equipment modifications should be made: first, a mixer should be placed in the outlet saline water line to insure a cup-mixed temperature is obtained; second, a turbine type organic pump should be used to insure a positive flow of slurry; third, the flowmeter on the outlet organic line should be compatible with a slurry flow; and fourth, an automatic interface controller should be used to reduce interface positioning problems.

When selecting an ice-making vessel, the merits of the mixer-settler and the spray column must be compared. It is noted that the contact area and the degree of mixing in a mixer-settler may be adjusted by a simple change of stirrer speed. Moreover, the separation of the organic and saline water phases is easily achieved in a mixer-settler. In a spray column there is the problem of interface stability which can be controlled only by the ice-brine outflow. It is thought that a series of counter-current mixer-settler devices (3) would be more suitable than a spray column for use as an ice-maker.

DEFINITIONS

Cp	Heat capacity of organic mixture on a volume basis, $(\frac{\text{BTU}}{\text{gal - °C.}})$
$\overline{\text{C}}\text{p}$	Average molar heat capacity of the organic mixture, $(\frac{\text{cal}}{\text{gmole - °C.}})$
$\tilde{\text{C}}\text{p}$	Molar heat capacity, $(\frac{\text{cal}}{\text{gmole - °C.}})$
CSA	column cross-sectional area, 0.049 ft^2
$(\frac{dP}{dT})_{\text{melting}}$	rate of change of pressure with temperature at a melting point, $(\frac{\text{psi}}{\text{°F.}})$
G	specific mass flow rate, based on column cross-sectional area, $\text{lbm/(hr-ft}^2)$
HTU	height of a transfer unit, $\frac{\text{Z (ft)}}{\text{NTU}}$, ft
HTU_{od}	overall height of a transfer unit, based on the dispersed phase, $\frac{\text{Z (ft)}}{\text{NTU}_{\text{od}}}$, ft
H_{s}	cap disperser slit height, inch
I.D.	inner diameter, inch
LOG_{e}	Logarithm of base e.
MW	molecular weight, gm/gmole
N.P.T.	national pipe thread
NTU_{od}	number of transfer units, based on dispersed organic phase, $\int \frac{dT_d}{(T_d - T_c)} \doteq (\frac{T_{\text{bot, org.}} - T_{\text{top, org.}}}{\Delta T_{\text{LM}}})$
O.D.	outer diameter, inches
q	heat transfer rate within the column, (BTU/min.)

T	temperature, °C.
TC	thermocouple
ΔT_{LM}	log-mean temperature difference, $\frac{[(T_{bot,\ org.} - T_{bot}) - (T_{top,\ org.} - T_{top})]}{\mathrm{LOG}_e\ [\frac{(T_{bot,\ org.} - T_{bot})}{(T_{top,\ org.} - T_{top})}]}$, °C.
$U_{w.\ vol.}$	overall volumetric heat transfer coefficient, $\frac{q}{(\Delta T_{LM})(Z)(CSA)}$, $(\frac{BTU}{hr\text{-}ft^3\text{-}^\circ F.})$
$\dot{V}$	volumetric flow rate, (gal/min)
Z	working column height or contact height, 4.21 ft

Subscripts:

bot = bottom of the column

c = continuous phase

C_{13} = n-tridecane

C_{14} = n-tetradecane

org = organic mixture

sw = saline water

ACKNOWLEDGMENTS

The author wishes to express his sincere gratitude to his major advisor, Dr. Richard G. Akins, for his constant advice and guidance. The author wishes to thank Dr. L. T. Fan and Dr. William Honstead for their help. Special thanks are given to Miss Diana Goertz for her encouragement and assistance in the preparation of this manuscript.

The author wishes to thank the Kansas State University Engineering Experiment Station and the Office of Saline Water for their financial support in this work.

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APPENDIX A

SAMPLE CALCULATIONS

Sample Calculations

Run Number 7-1

Direction of Heat Transfer: Organic to saline water. No solids present.

The rate that heat is given up by the organic q_{org}, is obtained from

$$q_{org}\left(\frac{BTU}{min}\right) = C_{p_{org}}\left(\frac{BTU}{gal\ °C}\right)\left[\dot{V}_{org}\left(\frac{gal}{min}\right)\right]\left[T_{bot,\ org} - T_{top,\ org}\ (°C.)\right]$$

Heat capacity of 52 vol. % n-$C_{13}H_{28}$ - 48 vol. % n-$C_{14}H_{30}$ mixture

For n-tetradecane

$$\tilde{C}_{p_{C_{14}}} = 0.497\left(\frac{cal}{gm - °C}\right) 198.38\left(\frac{gm}{gmole}\right)$$

$$\tilde{C}_{p_{C_{14}}} = 98.6\left(\frac{cal}{gmole - °C}\right)$$

For n-tridecane

$$\tilde{C}_{p_{C_{13}}} = 0.499\left(\frac{cal}{gm - °C}\right) 184.36\left(\frac{gm}{gmole}\right) = 92.0\left(\frac{cal}{gmole - °C}\right)$$

The molar average heat capacity of the mixture is given by:

$$\overline{C}_p = x_{C_{14}}\ \tilde{C}_{p_{C_{14}}} + x_{C_{13}}\ \tilde{C}_{p_{C_{13}}}$$

for the 52 vol. % n-$C_{13}H_{28}$ solution

$$x_{C_{13}} = 0.536 \quad \text{and} \quad x_{C_{14}} = 0.464$$

Thus

$$\bar{C}_p = 0.464(98.6 \frac{cal}{gmole - °C}) + 0.536(92.0 \frac{cal}{gmole - °C})$$

$$\bar{C}_p = 95.1 \frac{cal}{gmole - °C}$$

Returning to a specific volume basis

$$C_{p_{org}} = \frac{\bar{C}_p}{MW} = \frac{95.1 (\frac{cal}{gmole\ °C})}{190.9 (\frac{gm}{gmole})}$$

$$C_{p_{org}} = \frac{0.498\ cal}{(gm - °C)} = 5.708 (\frac{BTU}{gal\ °C})$$

$$q_{org} = 5.708 (\frac{BTU}{gal - °C}) [0.445 (\frac{gal}{min})] [9.682 - 3.868] (°C)$$

$$q_{org} = 14.76 (\frac{BTU}{min})$$

The log mean temperature difference ΔT_{1m}, is calculated by

$$\Delta T_{1m}(°C) = \frac{[(T_{bot, org} - T_{bot, sw}) - (T_{top, org} - T_{top, sw})]}{LOG_e \frac{(T_{bot, org} - T_{bot, sw})}{(T_{top, org} - T_{top, sw})}}$$

where all temperatures are in °C.

$$\Delta T_{1m}(°C) = \frac{[(9.682 - 3.592) - (3.868 - 2.374)]}{LOG_e \frac{(9.682 - 3.592)}{(3.868 - 2.374)}}$$

$$\Delta T_{1m} = 3.271°C$$

The overall volumetric heat transfer coefficient, $U_{w.vol.}$, based on the working volume of the column, is calculated from

$$U_{w.vol.} = \frac{q_{org}\ (\frac{BTU}{min})}{(\Delta T_{1m})\ (\text{working column volume})}$$

Working column volume calculation:

> The working height, Z, of the column is the length over which the continuous and discontinuous phases normally contact. It is the length from the organic nozzle to the organic-saline water interface; Z = 4.21 feet in our column.

Column I.D. = 0.25 ft

$$\text{Column cross-sectional area (CSA)} = \frac{\pi D^2}{4} = \frac{\pi}{4}\ (0.25\ \text{ft})^2$$

$$= 0.049\ \text{ft}^2$$

$$\text{Working column volume} = (\text{CSA})\ (Z)$$

$$= (4.21\ \text{ft})\ (0.049\ \text{ft}^2) = 0.0206\ \text{ft}^3$$

$$U_{w.vol.} = \frac{q_{org}\ (\frac{BTU}{min})\ [(\frac{60\ min}{hr})\ (\frac{1°C}{1.8°F})]}{\Delta T_{1m}(°C)\ (\text{working column volume})\ (\text{ft}^3)}$$

$$U_{w.vol.} = \frac{q_{org}\ (\frac{BTU}{min})}{\Delta T_{1m}(°C)\ (0.206\ \text{ft}^3)}\ [33.33\ (\frac{min\ \ °F}{hr\ \ °C})]$$

$$U_{w.vol.}\ (\frac{BTU}{hr\text{-}ft^3\text{-}°F}) = [161.4\ (\frac{min}{hr}\ \frac{°C}{°F}\ \frac{1}{ft^3})]\ \frac{q_{org}\ (\frac{BTU}{min})}{\Delta T_{1m}(°C)}$$

$$U_{w.vol.} = 161.4\ (\frac{min}{hr}\ \frac{°C}{°F}\ \frac{1}{ft^3})\ \frac{14.76\ (\frac{BTU}{min})}{3.271\ (°C)}$$

$$U_{w.vol.} = 728.5\ (\frac{BTU}{hr\text{-}ft^3\text{-}°F})$$

The number of transfer units based on the dispersed organic phase, NTU_{od}, is given by

$$NTU_{od} = \frac{(T_{bot,\ org} - T_{top,\ org})}{\Delta T_{1m}}$$

$$NTU_{od} = \frac{(9.682 - 3.868)\ °C}{3.271\ (°C)} = 1.778$$

The height of a transfer unit based on the dispersed organic phase, HTU_{od}, is given by

$$HTU_{od} = \frac{Z\ (ft)}{NTU_{od}} = \frac{4.21}{1.778} = 2.37$$

Run 61-1

Direction of Heat Transfer: Saline water to organic. Organic solids present.

> The rate of heat transfer was taken as equal to the rate of heat loss by the saline water, q_{sw}, since the thermodynamic data available on the organic slurry were inadequate.

$$q_{sw}\ (\frac{BTU}{min}) = C_{p_{sw}}\ (\frac{BTU}{gal-°C})\ (\dot{V}_{sw})\ (\frac{gal}{min})\ (T_{top,\ sw} - T_{bot,\ sw})\ °C$$

Where $C_{p_{sw}}$ for normal sea water (3.5 weight % NaCl) from (19) is

$$C_p = 0.952\ \frac{BTU}{lbm-°F} \qquad \text{Specific gravity} = 1.027$$

$$C_{p_{sw}} = 0.952\ (\frac{BTU}{lbm°F})\ 1.8\ (\frac{°F}{°C})\ \frac{1.027}{0.1198\ (\frac{gal}{lbm}\ \text{pure water})}$$

$$C_{p_{sw}} = 14.69\ (\frac{BTU}{gal°C})$$

$$q_{sw}\ (\frac{\text{BTU}}{\text{min}}) = 14.69\ (\frac{\text{BTU}}{\text{gal-°C}})\ (1.017\ \frac{\text{BTU}}{\text{min}})\ (8.987 - 4.338)\ °\text{C}$$

$$q_{sw} = 54.60\ \frac{\text{BTU}}{\text{min}}$$

The log mean temperature difference ΔT_{lm} is calculated as for the case of no solids present

$$\Delta T_{lm}\ (°\text{C}) = \frac{[(T_{bot,\ org} - T_{bot,\ sw}) - (T_{top,\ org} - T_{top,\ sw})]}{\text{LOG}_e \frac{(T_{bot,\ org} - T_{bot,\ sw})}{(T_{top,\ org} - T_{top,\ sw})}}$$

$$\Delta T_{lm}\ (°\text{C}) = \frac{(-3.247 - 5.333) - (4.338 - 8.987)}{\text{LOG}_e\ [(-3.247 - 5.333) - (4.338 - 8.987)]}$$

$$\Delta T_{lm} = -6.415\ °\text{C}$$

$$U_{w.vol.} = 161.4\ (\frac{\text{min}}{\text{hr}}\ \frac{°\text{C}}{°\text{F}}\ \frac{1}{\text{ft}^3}) \left(\frac{-q_{sw}\ (\frac{\text{BTU}}{\text{min}})}{\Delta T_{lm}\ (°\text{C})}\right)$$

$$= 161.4\ (\frac{\text{min}}{\text{hr}}\ \frac{°\text{C}}{°\text{F}}\ \frac{1}{\text{ft}^3}) \left(\frac{-54.60\ (\frac{\text{BTU}}{\text{min}})}{-6.415\ °\text{C}}\right)$$

$$U_{w.vol.} = 1374\ (\frac{\text{BTU}}{\text{hr-ft}^3\text{-°F}})$$

NTU_{od} and HTU_{od} values were not calculated for the cases in which organic solids were present.

APPENDIX B

CALIBRATIONS

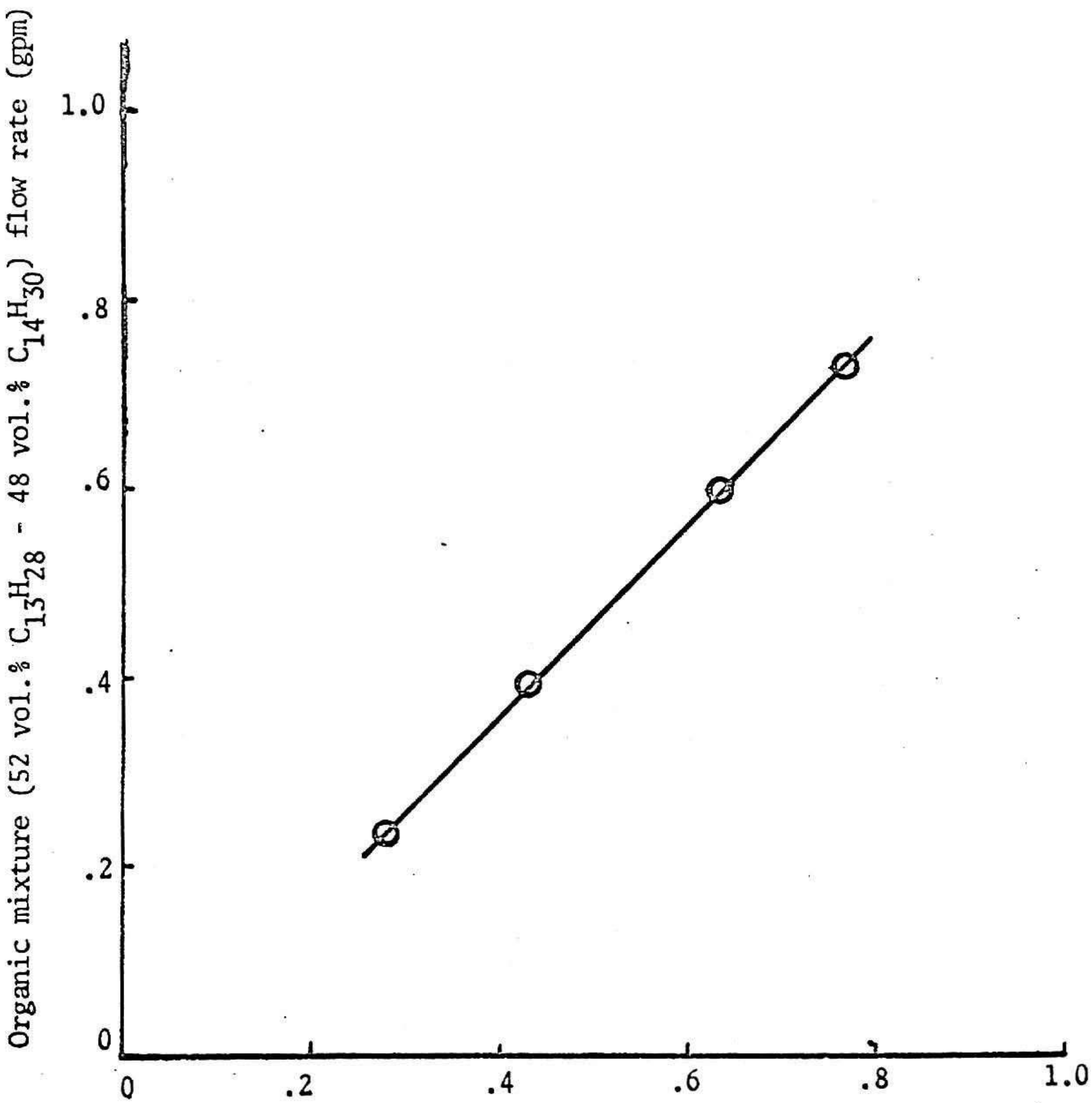

Figure 12. Calibration of organic rotameter for 52 vol.% $C_{13}H_{28}$ and 48 vol.% $C_{14}H_{30}$ mixture.

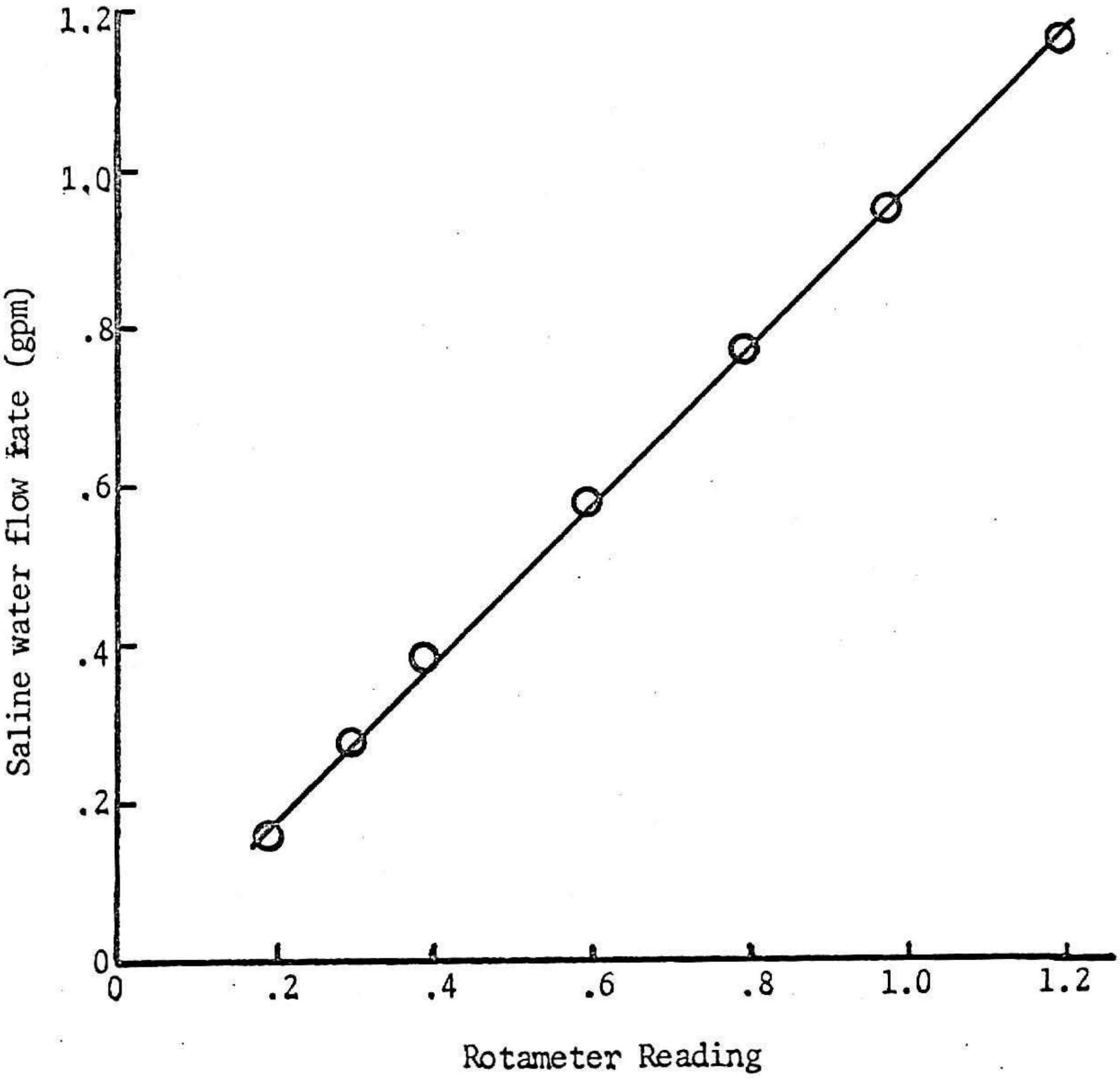

Figure 13. Calibrations of saline water rotameter for 3.3 weight-% salt water at 20.5°C.

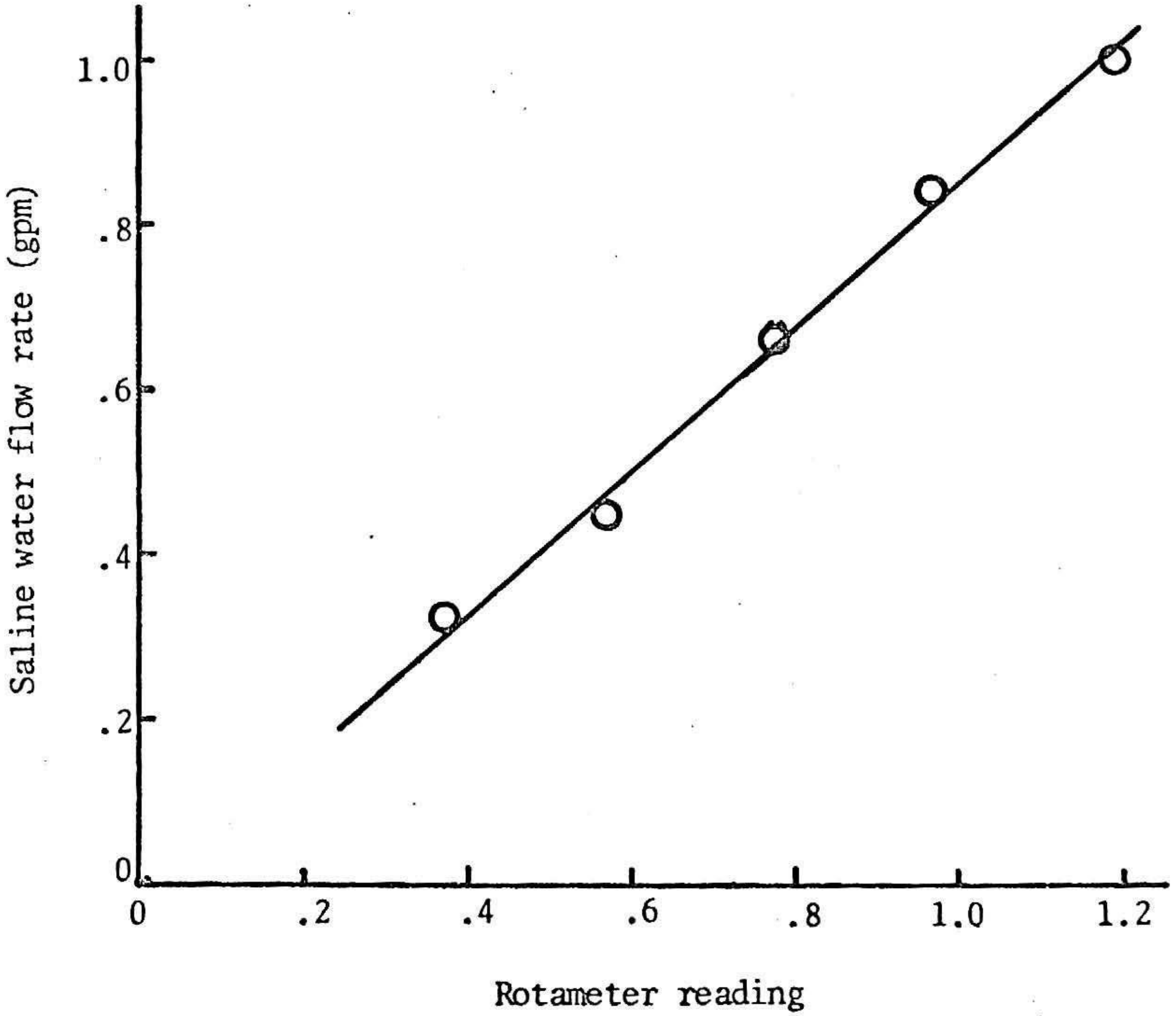

Figure 14. Calibrations of saline water rotameter for 3.5 weight-% salt water at + 1.0°C.

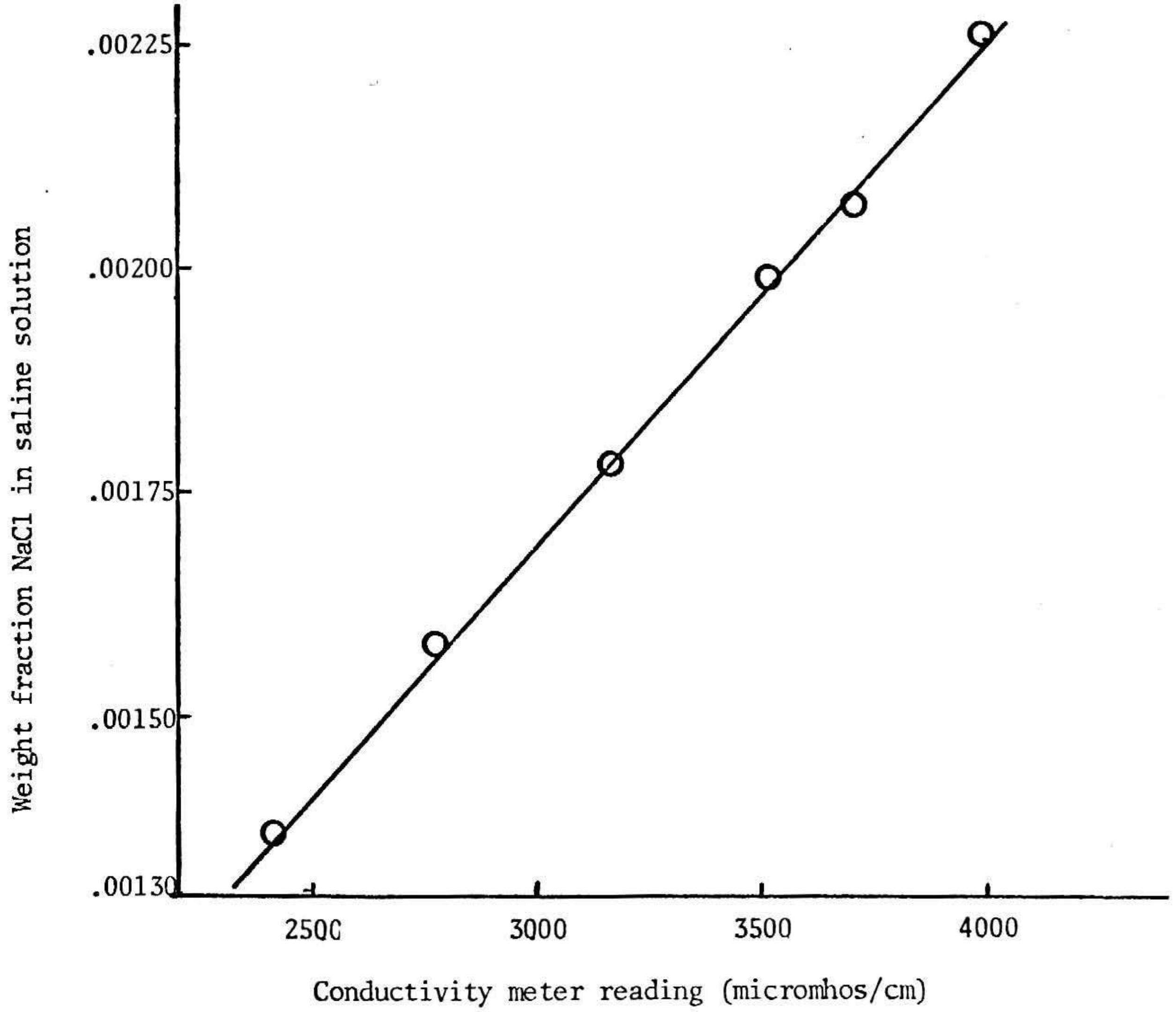

Figure 15. Calibration of Beckmann Conductivity Meter with saline solutions at 22.8 °C.

APPENDIX C

PHYSICAL PROPERTIES

Physical Constants

The physical properties of n-tridecane ($C_{13}H_{28}$) and n-tetradecane ($C_{14}H_{30}$) are as follows:

	$C_{13}H_{28}$	$C_{14}H_{30}$	Reference
Molecular weight, MW	184.36	198.38	(21)
Melting Point (1 atm), (°C.)	-6.2	+5.5	(21)
Specific Gravity, (20°C./4°C.)	0.757	0.765	(21)
Heat of Fusion, (cal/gm)	37.0	54.4	(22)
Solid Heat Capacity, (cal/gm-°C.)	0.577(-13°C.)	0.418(-3°C.)	(22)
Liquid Heat Capacity, (cal/gm-°C.)	0.514(-3°C.)	0.519(+7°C.)	(22)

The freezing point of the 52 vol.% n-$C_{13}H_{28}$, 48 vol.% n-$C_{14}H_{30}$ mixture used in this work was reported by Chiu (3) to be -3.07° C.

APPENDIX D

DATA

Data tables for runs 1 through 69 presenting ambient temperatures (°C.), water rate/organic rate (gpm), and thermocouple temperature readings (°C.), for top water, bottom organic, top organic, and bottom water.

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Salt Water Concentration 3.3 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
1-1	Interface was 1 inch low during this set of readings.	29.0	.349/.445	4.077	9.820	6.451	6.741
1-2		29.0	.349/.445	3.421	10.238	6.290	6.462
2-1		28.6	.483/.446	2.474	10.262	5.049	5.141
2-2		28.2	.483/.446	2.087	10.200	4.831	4.826
2-3		28.0	.483/.446	2.077	10.262	4.744	4.808
3-1		28.1	.581/.446	1.726	10.225	4.410	4.282
3-2		27.5	.581/.446	1.872	10.255	4.410	4.223
3-3		27.3	.572/.446	2.041	10.288	4.405	4.205
4-1		26.0	.769/.441	2.000	10.290	4.010	3.766
4-2		26.0	.769/.441	1.949	10.238	4.000	3.895
4-3		26.0	.769/.441	2.200	10.250	4.072	3.813

Salt Water Concentration [illegible] wgt % 52% (vol) C_{13} – 48% (vol) C_{14}

Stirrer speed 136 rpm Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
5-1		25.0	.963/.446	2.400	9.913	4.082	3.789
5-2		24.8	.963/.446	2.346	9.959	4.077	3.805
6-1		24.7	1.155/.445	2.426	9.823	3.971	3.721
6-2		24.6	1.155/.445	2.410	9.846	3.953	3.634
7-1		25.0	1.353/.445	2.374	9.682	3.868	3.592
7-2		25.0	1.345/.445	2.405	9.638	3.671	3.539
8-1		24.9	.762/.682	2.849	10.012	4.536	4.987
8-2		24.5	.768/.682	2.844	9.859	4.526	5.005
8-3		24.6	.768/.682	2.836	9.769	4.485	4.997
9-1		24.8	.967/.690	2.744	9.369	4.141	4.538
9-2		25.0	.967/.690	2.708	9.305	4.120	4.492

Salt water concentration 3.5 wgt % 52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm Slit height 3/16 inches

RUN #/ POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
9-3		25.0	.963/.690	2.744	9.067	4.044	4.451
10-1	Interface was 3/4 inch low during this set of readings.	24.6	1.162/.696	2.741	9.038	3.934	4.256
10-2		24.6	1.162/.696	2.759	8.872	3.840	4.169
10-3		24.6	1.162/.696	2.744	8.818	3.876	4.182
11-1		24.7	.582/.691	2.795	8.654	4.454	4.977
11-2	Interface was 3/4 inch low during this set of readings.	24.7	.582/.691	2.744	8.644	4.433	5.015
11-3		24.7	.582/.691	2.677	8.600	4.372	4.938
12-1		24.8	.447/.701	2.728	8.759	4.895	5.362
12-2	Interface was 3/4 inch low during this set of readings.	24.8	.433/.701	2.708	8.741	4.803	5.462
12-3		24.9	.447/.701	2.550	8.772	4.851	5.546
13-1		25.8	1.352/.700	2.544	8.554	3.466	3.879

Salt Water Concentration ______ wgt % 52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
14-1		25.6	.388/.823	2.705	8.562	5.177	5.933
14-2		25.4	.388/.823	2.677	8.738	5.149	5.951
14-3		25.4	.388/.823	2.726	8.613	5.102	6.010
15-1	Interface was 1 inch low during this set of readings.	24.9	.582/.831	2.628	8.400	4.492	5.202
15-2	Interface was 1 inch low during this set of readings.	24.9	.582/.831	2.628	8.338	4.410	5.205
16-1		25.3	.775/.827	2.590	8.182	4.020	4.733
16-2		25.3	.769/.827	2.620	8.154	4.000	4.636
17-1		25.3	.968/.836	2.554	7.815	3.682	4.269
17-2		25.3	.968/.836	2.613	7.648	3.647	4.226
17-3	Interface was 1 inch low during this set of readings.	25.3	.956/.836	2.590	7.662	3.724	4.269
18-1	Interface was 1 inch low, got beaten by the water jets.	25.3	1.156/.836	2.590	7.538	3.550	4.000

Salt Water Concentration 3.3 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
18-2		25.3	1.162/.836	2.626	7.465	3.526	4.013
19-1		25.7	.562/.348	9.241	5.482	8.054	8.428
19-2		25.5	.562/.348	9.518	5.964	8.333	8.820
19-3		25.2	.562/.348	9.756	6.138	8.556	9.015
20-1	Interface was 1.5 inches low during readings taken in this set.	25.2	.776/.340	10.752	6.900	9.549	10.212
20-2	Interface appeared scummy.	24.5	.776/.340	11.005	6.885	9.697	10.415
20-3		24.8	.776/.340	11.402	7.038	10.088	10.772
21-1		24.3	.968/.340	12.240	7.540	10.888	11.669
21-2	Interface was 1 inch low during this set of readings.	24.8	.968/.340	12.420	7.575	11.100	11.766
21-3		24.4	.968/.340	12.580	7.692	11.256	11.967
21-4		24.0	.968/.340	12.770	7.700	11.285	12.115

Salt Water Concentration 3.3 wgt % 52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
21-5		24.0	.968/.340	13.026	7.962	11.538	12.385
22-1		24.1	1.162/.340	13.692	8.177	12.150	13.118
22-2		24.0	1.162/.340	13.882	8.182	12.428	13.295
22-3		23.9	1.162/.340	14.302	8.410	12.802	13.720
23-1	Interface was 1 inch low during this set of readings.	23.2	1.352/.340	14.665	8.720	13.259	14.188
23-2		23.3	1.352/.340	14.985	8.862	13.459	14.432
23-3	Bottom organic thermocouple fluctuated; took five minutes to read it.	23.4	1.352/.340	15.150	8.872	13.592	14.602
24-1	Interface was hard to control.	23.3	.389/.340	15.965	8.844	13.031	13.990
24-2		23.1	.377/.340	16.165	8.887	13.226	14.102
24-3	Bottom organic thermocouple fluctuated during Run 24-1, 2, and 3.	23.0	.389/.340	16.260	8.944	13.156	14.300
25-1		22.5	.369/.728	16.990	11.338	14.278	14.048

Salt water concentration ________ wgt % 13 14

Stirrer speed 136 rpm Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
25-2		22.2	.369/.728	17.260	11.492	14.588	14.255
25-3		22.0	.369/.728	17.322	11.650	14.670	14.338
26-1		22.0	.582/.722	17.280	11.718	15.325	15.110
26-2		21.9	.582/.722	17.410	11.723	15.365	15.212
26-3		21.7	.582/.722	17.412	11.872	15.472	15.235
27-1	Interface fell to the water nozzle.	21.7	.775/.728	17.502	11.977	15.838	15.753
27-2		21.7	.775/.728	17.465	12.088	15.905	15.850
27-3		21.7	.775/.728	17.568	12.122	15.982	15.475
27-4		21.7	.775/.728	17.600	12.240	16.050	15.925
28-1		21.3	.967/.728	17.642	12.268	16.215	16.262
28-2		21.3	.967/.720	17.662	12.352	16.275	16.350

Salt Water Concentration 3.3 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
28-3	Interface dropped while the top organic was read.	21.3	.967/.728	17.635	12.360	16.380	16.380
29-1		21.3	1.162/.732	17.698	12.480	16.540	16.530
29-2		21.3	1.140/.732	17.738	12.465	16.578	16.672
29-3		21.3	1.162/.732	17.852	12.375	16.610	16.708

Salt Water Concentration 3.5 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 7/32 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
30-1		28.3	.506/.580	5.205	-3.379	-1.369	1.590
30-2		28.2	.497/.580	5.595	-3.366	-0.816	1.882
30-3		28.3	.497/.580	6.074	-3.358	-0.105	2.449
31-1		27.8	.338/.580	7.225	-3.305	0.095	2.231
31-2		28.5	.338/.580	7.565	-3.300	0.263	2.310
32-1		28.1	.667/.580	8.026	-3.326	2.233	4.654
32-2		28.0	.668/.580	8.215	-3.290	2.449	4.820
32-3		28.0	.668/.580	8.318	-3.316	2.508	4.951
33-1	Disperser motor may have stopped in Runs 32 and 33	28.1	.848/.580	8.820	-3.424	1.777	5.420
33-2		28.1	.848/.580	8.910	-3.434	1.618	5.451

Salt Water Concentration 3.5 wgt %

Stirrer speed 136 rpm

52% (vol) C_{13} - 48% (vol) C_{14}

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
34-1	Organic rotameter bob and stops were removed. Measured slit height=3/16"	26.5	.668/.747	14.238	-3.524	3.432	7.028
34-2		26.5	.677/.747	13.903	-3.503	3.066	6.833
34-3	Disperser motor may have stopped.	26.5	.677/.747	13.651	-3.500	2.633	6.820
35-1	Interface contained a 1.5 inch thick layer of scum.	27.3	.847/.747	12.638	-3.579	2.660	6.715
35-2		27.3	.847/.747	12.250	-3.589	1.962	6.462
35-3		27.3	.847/.747	12.090	-3.566	1.436	6.218
36-1	7/8 inch layer of scum was visible below the interface.	27.0	1.017/.747	11.192	-3.618	1.041	6.267
36-2		26.9	1.017/.747	10.838	-3.634	0.421	6.015
36-3		26.6	1.017/.747	10.590	-3.655	-0.105	5.751
37-1	Measured slit height was 2.5/16 inch.	26.6	.503/.747	8.851	-3.740	-0.947	2.462
37-2		26.4	.503/.747	8.756	-3.800	-0.971	2.395

Salt Water Concentration 3.5 wgt %　　　52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm　　　Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
37-3		26.4	.503/.747	8.605	-3.813	-0.592	2.526
38-1		26.0	.338/.747	8.802	-3.960	-1.872	1.256
38-2		26.0	.338/.747	8.695	-3.987	-2.074	1.200
38-3		26.0	.338/.747	9.144	-3.568	-1.628	1.285
39-1		25.9	.847/.351	-0.645	-3.658	-3.195	-1.167
39-2	Outflowing organic appeared to contain some solids.	25.9	.847/.351	-0.624	-3.695	-3.132	-1.090
39-3		25.9	.847/.351	-0.582	-3.687	-2.753	-0.971
39-4		25.9	.847/.351	-0.524	-3.629	-2.290	-0.855
40-1		27.0	.677/.351	-0.210	-3.526	-3.053	-0.858
40-2	Measured slit height was 3/16 inch.	27.0	.677/.351	-0.276	-3.553	-3.076	-0.908
40-3		27.0	.667/.351	-0.303	-3.605	-3.216	-0.974

Salt Water Concentration 3.5 wgt %

Stirrer speed 136 rpm

52% (vol) C_{13} - 48% (vol) C_{14}

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
41-1	Measured disperser speed was 184 rpm. Droplets formed normally.	28.3	.505/.351	-0.642	-3.753	-3.350	-1.462
41-2	Salt water rotameter read 0.59 gpm.	28.3	.505/.351	-0.682	-3.734	-3.371	-1.497
41-3	Measured slit height was 3/16 inch.	28.3	.505/.351	-0.710	-3.737	-3.171	-1.487
42-1	Measured disperser speed was 136 rpm. Measured slit height was 3.8/16 inch.	24.5	.676/.576	4.872	-0.560	3.526	3.797
42-2		24.3	.676/.576	5.112	-0.555	3.579	3.842
42-3		24.2	.676/.576	5.080	-0.521	3.610	3.910
43-1		24.9	.847/.576	5.444	-0.566	4.038	4.323
43-2		24.9	.847/.576	5.426	-0.658	4.050	4.410
43-3		25.0	.847/.576	5.615	-0.658	4.167	4.513
44-1	Disperser speed slowed to about 80 rpm. Droplets formed normally.	25.0	1.015/.576	5.795	-0.460	4.549	4.820
44-2	Disperser speed slowed to about 80 rpm. Droplets - normal.	24.8	1.015/.576	6.013	-0.447	4.692	4.949

Salt Water Concentration 3.5 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3.8/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
45-1	Disperser speed restored to 136 rpm.	24.9	.505/.576	6.426	-0.510	4.413	4.551
45-2		24.9	.500/.576	6.580	-0.513	4.520	4.580
45-3		24.9	.505/.576	6.736	-0.540	4.572	4.785
46-1		24.9	.330/.576	7.295	-0.579	4.333	4.385
46-2		24.9	.330/.576	7.435	-0.539	4.390	4.372
47-1		27.0	1.017/.576	1.744	-3.347	-2.800	-0.384
47-2		27.0	1.017/.576	1.602	-3.313	-2.605	-0.329
48-1	Organic solids appeared packed above the interface.	27.5	.846/.576	0.647	-3.300	-2.824	-1.038
48-2		27.5	.846/.576	0.379	-3.295	-2.855	-1.123
49-1	No ice was formed on the saline water cooling coil	27.3	.676/.576	0.315	-3.337	-2.960	-1.564
49-2		27.3	.676/.576	-0.008	-3.368	-3.053	-1.664

Salt Water Concentration 3.5 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3.8/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
49-3	Measured disperser speed was 128 rpm.	27.3	.676/.576	.079	-3.371	-2.984	-1.920
50-1	Organic solids were packed at the interface; no ice visible.	27.3	.506/.576	1.400	-3.342	-2.008	-2.092
50-2	Disperser motor quit and was restored. Small organic droplets produced.	27.3	.506/.576	1.487	-3.416	-2.060	-2.379
51-1	Measured slit height was 3.8/16 inch.	25.8	.331/.668	1.190	4.272	2.754	2.692
51-2		25.8	.331/.668	1.346	4.203	2.846	2.603
51-3		25.8	.331/.668	1.372	4.238	2.897	2.880
52-1	3/4 inch of scum accumulated at the interface.	25.8	.502/.668	1.241	4.164	2.628	2.615
52-2		25.8	.502/.668	1.241	4.136	2.564	2.580
52-3		25.8	.502/.668	1.274	4.095	2.590	2.528
53-1	Measured disperser speed was 113 rpm.	25.8	.673/.668	1.218	3.984	2.359	2.218
53-2		25.8	.673/.668	1.256	4.026	2.359	2.244

Salt Water Concentration 3.5 wgt %

Stirrer speed 136 rpm

52% (vol) C_{13} - 48% (vol) C_{14}

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
53-3		25.8	.673/.668	1.218	3.932	2.333	3.256
54-1	Measured disperser speed was 120 rpm.	25.8	.847/.668	1.513	3.879	2.359	2.172
54-2		25.8	.847/.668	1.439	3.842	2.426	2.233
54-3		24.9	.847/.668	1.551	3.816	2.390	2.223
55-1	Measured disperser speed was 136 rpm.	24.8	1.017/.668	1.664	3.858	2.485	2.269
55-2		25.0	1.017/.668	1.664	3.837	2.487	2.310
56-1		25.0	.338/.668	4.208	-1.077	1.800	1.903
56-2		25.0	.338/.668	4.436	0.568	2.410	2.092
57-1		24.5	.507/.668	5.062	1.625	3.895	3.708
57-2		24.5	.507/.668	5.262	1.831	4.103	3.842
57-3		24.5	.507/.668	5.423	2.100	4.292	4.064

Salt Water Concentration 3.5 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
58-1		24.7	.677/.668	5.972	2.774	5.072	4.905
58-2		24.7	.677/.668	6.205	3.042	5.297	5.144
59-1	Disperser motor raced. (Adjusted.) Measured slit height 1/4 inch.	24.7	.847/.668	6.820	3.737	6.051	5.936
59-2		24.7	.847/.668	7.710	4.705	6.949	6.872
59-3		24.4	.847/.668	7.798	4.820	7.088	7.015
60-1	Disperser quit working but was restored to operation.	24.3	1.017/.668	8.000	4.318	7.250	7.250
60-2		24.3	1.017/.668	8.090	4.241	7.250	7.250
60-3	Freon control valve adjusted for lower temperature.	24.3	1.017/.668	8.462	1.192	6.882	7.050
61-1		24.4	1.017/.668	8.987	-3.247	4.338	5.333
61-2		24.4	1.017/.668	8.949	-3.260	3.724	5.185
61-3		24.4	1.017/.668	8.910	-3.263	3.653	5.126

Salt Water Concentration 3.5 wgt %

52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm

Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
62-1		24.4	.847/.668	8.869	-3.263	2.244	4.205
62-2		24.4	.847/.668	8.818	-3.279	2.374	4.154
62-3		24.4	.847/.668	8.728	-3.326	1.964	4.051
63-1	Measured disperser speed was 200 rpm. Drops forming normally.	24.7	.676/.668	8.628	-3.342	-0.184	2.687
63-2		25.3	.676/.668	8.510	-3.418	-0.779	2.418
63-3		25.6	.676/.668	8.459	-3.432	-1.028	2.346
64-1	Measured disperser speed was 200 rpm. Drops forming normally.	25.6	.505/.668	8.433	-3.216	1.323	2.510
64-2		25.6	.505/.668	8.451	-3.226	1.754	2.820
64-3		25.6	.505/.668	8.462	-3.224	2.151	2.833
65-1		25.7	.338/.668	8.772	-3.276	0.017	1.513
65-2		25.7	.338/.668	8.792	-3.303	-0.776	1.000

Salt Water Concentration 3.5 wgt % 52% (vol) C_{13} - 48% (vol) C_{14}

Stirrer speed 136 rpm Slit height 3/16 inches

RUN # / POINT	NOTES	AMBIENT TEMP (°C)	WATER RATE/ ORG RATE (gpm)	TEMPERATURES (°C)			
				TOP WATER	BOTTOM ORGANIC	TOP ORGANIC	BOTTOM WATER
65-3		25.6	.338/.668	8.759	-3.303	-0.695	1.154
66-1	Water level too high in the organic storage tank. Ice in the column may have	25.4	.338/.668	0.855	-3.539	-3.210	-2.260
66-2	formed in the organic slurry freezer. Organic pump secured after 66-2.	25.4	.338/.668	0.708	-3.632	-3.234	-2.253
67-1	Organic pump controller unchanged. Flow rate assumed same as Run 66.	26.2	.507/.668	1.128	-3.408	-3.066	-1.728
67-2		26.2	.507/.668	1.190	-3.450	-3.063	-1.928
67-3	Organic pump turned off and organic valve to column secured after 67-3.	26.2	.507/.745	1.118	-3.495	-3.103	-1.974
68-1		26.4	.676/.745	1.113	-3.482	-3.066	-1.764
68-2		26.4	.676/.745	1.090	-3.516	-3.071	-1.795
68-3	Some solids collected at the bottom of the interface.	26.4	.676/.745	1.113	-3.550	-3.116	-1.998
69-1	Measured disperser speed was 152 rpm.	26.3	.845/.745	1.000	-3.526	-3.055	-1.744
69-2	Some solids packed at bottom of interface.	26.5	.845/.745	0.908	-3.474	-3.010	-1.667

VITA

Wickham Gregory Smith was born at Fort Benning, Georgia on 3 July, 1945. He attended grammar and high schools in Whittier, California, where he was graduated from La Serna High School in 1963. He entered the University of Minnesota in September 1963 under the Regular NROTC program and was graduated in June 1967 with the degree of Bachelor of Chemical Engineering.

Commissioned upon graduation, Wickham Smith was placed on inactive duty to do graduate work at Kansas State University. Called to active duty in November 1968, Lieutenant Junior Grade Smith, U. S. Navy, is now serving as Main Propulsion Assistant aboard the destroyer USS O'Hare (DD-889) and is a candidate for the Master of Science Degree in the Department of Chemical Engineering, Kansas State University.

DIRECT CONTACT HEAT TRANSFER
BETWEEN IMMISCIBLE PHASES IN A SPRAY COLUMN

by

WICKHAM GREGORY SMITH

B. Ch. E., University of Minnesota, 1967

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1970

This thesis presents the results of construction and operation of a spray column heat exchanger designed to operate with four phases: organic solid, organic liquid, saline water and fresh water ice. The dispersed species was an organic mixture of 52 volume-% n-tridecane ($C_{13}H_{28}$) and 48 volume-% n-tetradecane ($C_{14}H_{30}$). Two different continuous phase aqueous solutions, 3.3 weight-% NaCl and 3.5 weight-% NaCl, were tested.

The pyrex column had an I. D. of 3-in. and the dispersed and continuous species contacted through 4.21 feet of the 5.38 foot high column. Temperatures of incoming and outgoing fluids were measured by 4 stainless steel sheathed (O.D. = 1/16-in.) Copper-Constantan thermocouples. Flow rates ranged from 2,500 $lbm/(hr\text{-}ft^2)$ to 14,000 $lbm/(hr\text{-}ft^2)$ for the continuous saline water phase and from 2,648 $lbm/(hr\text{-}ft^2)$ to 6,510 $lbm/(hr\text{-}ft^2)$ for the dispersed organic phase. Drops formed at an organic disperser had a measured diameter of 0.32-in. for one run; the organic disperser slit height was used as a correlation parameter in lieu of droplet diameter.

Volumetric heat transfer coefficients and heights of a transfer unit are presented for two phase (saline water and liquid organic) operation with heat transfer in both directions; volumetric heat transfer coefficients also are presented for three phase (saline water, organic liquid and organic solid) operation. Although stable four phase operation was not obtained, the problems of flooding and recovery of product ice are discussed herein.