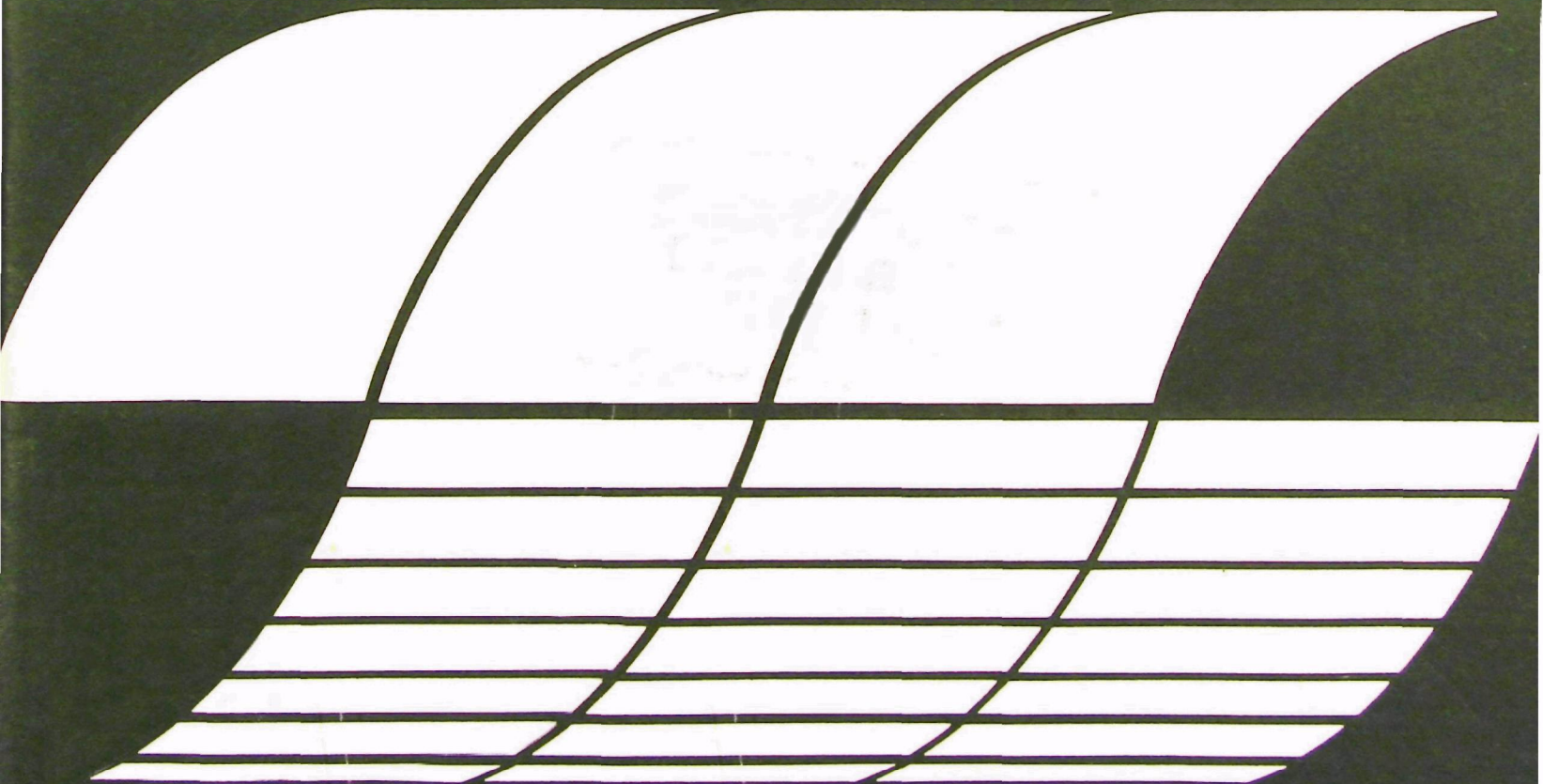


# **RENOVATION OF POWER PLANT COOLING TOWER BLOWDOWN FOR RECYCLE BY EVAPORATION: CRYSTALLIZATION WITH INTERFACE ENHANCEMENT**

Interagency  
Energy-Environment  
Research and Development  
Program Report



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# **RENOVATION OF POWER PLANT COOLING TOWER BLOWDOWN FOR RECYCLE BY EVAPORATION: CRYSTALLIZATION WITH INTERFACE ENHANCEMENT**

by

**Hugo H. Sephton**

**The University of California  
Campus Research Office  
M-11 Wheeler Hall  
Berkeley, California 94720**

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**EPA Project Officer: Fred Roberts**

**Industrial Environmental Research Laboratory  
Office of Energy, Minerals, and Industry  
Research Triangle Park, N.C. 27711**

**Prepared for**

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## ABSTRACT

This work confirms the effectiveness of interface enhancement<sup>\*</sup>; a novel method applied to the evaporation of liquids; it reduces the energy and capital cost requirements for the renovation-recycle of industrial wastewaters. Interface enhancement<sup>\*</sup> depends upon foamy two-phase vapor-liquid flow induced during the evaporation of a liquid flowing over a heat transfer surface; this mode of flow provides a substantial increase in the rate of evaporation of the liquid, after the addition of a selected surfactant. The objectives of this work included the construction of two new vertical tube evaporation (VTE) pilot plants. A two-effect upflow-downflow VTE of 10,000 gallons per day capacity was constructed by adding an identical second effect to an existing single effect, upflow VTE pilot plant; the second effect was operated in the downflow VTE mode for the work reported here. This pilot plant was used to obtain comparative data on the concentration of saline water by upflow VTE and downflow VTE, and by interface-enhanced upflow and downflow VTE. These data indicate that while conventional downflow VTE has a higher heat transfer performance than upflow VTE, the interface-enhanced method of upflow VTE operation provides a higher performance than both the interface-enhanced and conventional downflow operations.

A second pilot plant facility assembled for this work, was a 5,000 gallon per day vertical tube evaporator-crystallizer (EC), tested in the downflow mode. This facility was first used with low temperature steam heating, for crystallizing sodium sulfate and for reducing Mohave power plant cooling tower blowdown to a 30-fold concentrate, at about 51.5°C (125°F). The objectives of these tests were to determine the feasibility of renovating cooling tower blowdown with waste heat available within a conventional power plant cooling cycle; feasibility was indicated by the test results. Secondly, this EC was operated with a vapor compressor (VC) in the evaporation temperature range 101.5°C-107°C (215-224°F). This series of tests were for the concentration, by both conventional and interface-enhanced modes of operation, of saline agricultural drainage water and industrial cooling tower blowdown. In each of these cases, it was found that the heat transfer performance of the VTE was increased while its energy requirements were simultaneously reduced, by applying interface enhancement (1), except in the case of crystallizing sodium sulfate.

<sup>\*</sup>U.S. Patent No. 3,846,254, Nov. 5, 1974 and foreign counterpart patents.

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## SECTION I

### CONCLUSIONS

The following conclusions were drawn from, or confirmed by the work presented in this report:

(1) Renovation of cooling tower blowdown by vertical tube foam evaporation (VTFE) can be carried out with a heat transfer performance substantially enhanced in comparison to conventional evaporation. This enhanced performance VTFE is sufficiently effective at relatively low temperatures to substantially improve the prospects of performing this renovation with waste heat available within the usual power plant cooling cycle.

(2) The upflow mode of VTFE operation is more effective in terms of heat transfer performance and energy requirements than the downflow VTFE mode or conventional downflow VTE. The level of surfactant additive required to provide an adequate foaminess in the wastewater feed flow for maximal heat transfer enhancement is about 10 parts per million (ppm) for upflow VTFE and 20 ppm for downflow VTFE. At these conditions the upflow VTFE provides about a 100 percent heat transfer rate enhancement, and the downflow VTFE about 50 percent enhancement over the conventional vertical tube evaporation (VTE) procedures.

(3) Multieffect upflow and downflow VTFE, over the usual evaporation temperature range, offer a basis for substantial capital and energy cost savings over conventional multieffect VTE. The increased hydrodynamic stability of upflow VTFE permits an increased performance ratio, compared to VTE.

(4) Preconcentration of industrial wastewaters such as cooling tower blowdown should be considered before evaporation-crystallization (EC) of the concentrate. The addition of a surfactant for VTFE was beneficial in each preconcentration carried out in this work, and in each but one EC. The latter was the case of crystallizing a relatively soluble salt (sodium sulphate) from aqueous solution at low temperature. In such cases removal of the surfactant additive from the preconcentrate for recycle to the preconcentrator VTFE is indicated to be advantageous. Centrifugation of the slurried saline-crystalline blowdown from the EC was effective for the separation of crystalline product.

(5) The use of a surfactant additive and imposed foamy flow for the evaporation-concentration of cooling tower blowdown by vapor compression (VC) VTFE has significant advantages. The evaporation rate is increased while the power required is simultaneously reduced. This holds generally but not without exceptions, for evaporation-crystallization by VC-VTFE;

it was found to be beneficial for each cooling tower blowdown examined in this work.

(6) In the case of cooling tower blowdown obtained from the Mohave power plant, a 30-fold concentration was readily obtained at low temperature (125°F) with high heat transfer coefficients by VTFE and EC after the addition of 15 ppm surfactant to the blowdown. This indicates that waste heat should be sufficient to convert this wastewater into 96 percent pure distilled water of potable or reuse quality, and 4 percent slurried concentrate for disposal.

(7) Evaporation-concentration of presoftened agricultural wastewater by VC-VTFE provides a 20-fold concentrated solution for disposal or possible regeneration of the ion-exchange softeners while 95 percent of it becomes available for reuse, as pure distilled water. The addition of a surfactant to this wastewater provided a 37 percent increase in the rate of its evaporation, and a simultaneous reduction in the energy requirements.

(8) Concentration, followed by evaporation-crystallization of industrial cooling tower blowdown by vapor compression VTFE, provided clear advantages over conventional procedures. In one case, a 27-fold concentration was obtained with increased performance, providing pure distillate and a slurried concentrate of salts for disposal. In another case, a 20-fold concentration was obtained by VC-VTFE with an increased performance throughout the concentration range. This is clear evidence of the beneficial effects of surfactant additives and the interface-enhanced mode of VTFE operation for the renovation of cooling tower blowdown.

(9) The dispersant effects of the heat transfer enhancement additive can be of significant advantage during distillation of wastewaters. It serves to retain particulates in suspension, keeping heat transfer surfaces clean, and thereby contributes toward maintaining high heat transfer performance and reducing downtime for cleaning or descaling of evaporators.

(10) At a quoted cost of about 25 cents per lb in bulk delivery of 60 percent active solutions, the use of surfactant for heat transfer enhancement (1, 2) in the range of 50 to 100 percent with surfactant concentrations of 10 to 20 ppm added to wastewater feeds for VTFE, it is clear that this technology should be adopted for wastewater renovation, in the interest of both capital and/or energy cost reduction.

## SECTION II

### RECOMMENDATIONS

(1) It is recommended that this work be followed up by the design, construction and field testing of a pilot demonstration facility, to provide field test data on the renovation of power plant cooling tower blowdown of several different types. Such a project, ideally carried out with the collaboration of the electric power utility industry, should provide final feasibility and cost data, and provide confidence in the adoption of VTFE for the complete renovation-recycle of such wastewaters. It should also focus attention on the prospects of integrating blowdown renovation-recycle into the power plant cooling cycle, using low-grade, waste heat for this purpose, and complying with zero-discharge guidelines with minimal cost.

(2) It is recommended that the very promising results obtained in this work on the application of interface enhancement to vapor compression vertical tube evaporation be followed up by the design and construction of a mobile field test pilot plant unit for operation on several industrial wastewaters. Such a project should include as one of its objectives to satisfy potential industrial users of this technology of the merit of this approach to wastewater renovation-recycle. The proposed field tests should be designed to provide a clear view of the feasibility and cost involved. Ideally this field test series should be carried out with the collaboration of industry.

(3) It is recommended that work be carried out to demonstrate regeneration of ion-exchange resins, used for softening irrigation return flows, using a concentrate of the softened water.

## SECTION III

### RENOVATION OF POWER PLANT COOLING TOWER BLOWDOWN FOR RECYCLE

#### BY EVAPORATION-CRYSTALLIZATION WITH INTERFACE ENHANCEMENT\*

#### INTRODUCTION

The novel interface-enhanced method (1, 2) of evaporating liquids applied in this study relies on the addition of a few parts per million of a selected surfactant to the liquid to be evaporated, followed by causing the feed liquid to flow as a foamy layer over a heated surface. As a result of this foamy layer flow, heat transfer from the surface to the feed liquid is augmented by several mechanisms to provide for a significant increase in the rate of evaporation, or heat transfer coefficient (3, 4).

In the work described here, the method of interface enhancement for evaporation of liquids was applied to the vertical tube evaporation (VTE) procedure. VTE accounts for more industrial evaporation in the U.S. than all other methods used and its further improvement by the vertical tube foam evaporation (VTFE) method (5) should be of significant utility. This foam-enhanced method of operation is applicable to both the upflow and the downflow modes of operation, whereby the feed liquid is caused to flow either upward or downward through a bundle of parallel tubes while steam condensing on their outside walls provides the heat of evaporation (6).

Industrial evaporators are usually operated either in single effect or in multi-effect series. In single effect operation the source of condensing steam (heat) is derived by vapor recompression, whereby the vapor generated during evaporation of the feed is simply compressed to a relatively higher pressure and temperature, and caused to condense on the tube outside surfaces. In multi-effect plants the source of heat is usually steam from a boiler condensed on the tubes of the first effect; each of the subsequent effects in, for instance, a typical sextuple effect plant receives heating steam as the vapor from the previous effect. Such a series of evaporators must therefore be operated at a stepwise reduced evaporation temperature series. Good steam economy provides the main advantage of such a series of VTE effects; each pound of boiler steam used provides for about 6 pounds of distillate from a sextuple VTE. This ratio of product to heating steam is referred to as the performance ratio or economy ratio.

The number of effects that can be used for an upflow VTE series is limited by the available temperature span between the steam temperature

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\* U.S. Patent No. 3,846,254, Nov. 5, 1974 & foreign patent counterparts.

and the coolant used to condense the vapor from the final effect; typically three to six effects are used. Another factor that tends to limit the number of upflow effects in a VTE series is the minimal effect  $\Delta T$ , or the temperature difference required to vaporize the feed as it flows upward through the tubes (3, 4). Part of the available  $\Delta T$  is expended to pump the liquid through the tube by a vapor-lift mechanism. The minimal effective  $\Delta T$  therefore depends on the liquid static head or the hydrodynamic pressure drop ( $\Delta P$ ) through the upflow VTE tubes. If the typical pressure drop could be reduced by a significant factor, the minimal intereffect  $\Delta T$  could be reduced proportionately and the number of effects could then be increased correspondingly to thereby increase the performance or economy ratio of the upflow VTE. The interface enhancement method, or VTFE, provides the means of reducing the  $\Delta P$  quite substantially. This  $\Delta P$  reduction ranges from a factor of about 3 to a factor of about 10 over the usual evaporation temperature range which spans about 66°C (150°F), between the condenser temperature of about 32°C (90°F) and the steam-side temperature of the first effect at about 116°C (240°F). This  $\Delta P$  reduction effect is the most significant single consequence of interface enhancement applied to upflow VTE (VTFE). This reduction of the tube-side pressure drop ( $\Delta P$ ) by induced foamy flow (VTFE) also affects the operation of a single effect, vapor compression (VC) VTE to similarly reduce the minimal  $\Delta T$  that is required, and thereby to save power.

The second significant effect of imposed foamy evaporative flow is applicable to both upflow and downflow VTFE; it provides a substantial increase in the evaporation-side heat transfer coefficient. This effect, apparently the result of a thin-film, wiped-film mode of flow imposed over the heat transfer surface, has been observed to be consistently associated with continuous foamy layer flow of the evaporating liquid in VTFE (1-6). This enhancement is lost or reduced if an antifoaming agent is added to the feed or if the vapor flow becomes excessive, causing foam breaking (4). In the case of upflow VTFE, the combination of tube-side pressure drop reduction and foamy layer flow was shown to provide a 100 to 200 percent increase in the overall heat transfer coefficient,  $U$  (7); the downflow performance enhancement was not unequivocally established in comparison with the upflow enhancement at the outset of this work, but the effectiveness of VTFE had been demonstrated for both upflow and downflow modes individually.

One of the major objectives (and main results) of this work, was to establish, on a simultaneous, comparative basis the relative merits of upflow and downflow VTFE. For this purpose a downflow VTE pilot plant of 5000 gpd capacity was constructed under this project, and tested simultaneously with a pre-existing upflow VTE pilot plant of identical design and capacity. The significance of this work relative to industrial wastewater renovation-recycle follows from the need for concentration of such effluents before disposal, ponding or the recovery of useful solutes from the concentrate. The latter often utilizes crystallizing evaporation.

A second major objective of this work was to construct and test a 5000gpd crystallizing VTE, utilizing the foamy mode of enhancement; this unit was operated in the downflow mode of operation which is usually preferred for slurry-feed evaporators, to minimize scaling. The main

objective of this part of the work was to observe the effects of surfactant additives on the performance, and the fouling and scaling tendencies of a crystallizing evaporator operated in the slurry-feed mode. Since the latter is typically performed by the vapor compression (VC) mode of VTE operation (8), a VC-VTFE mode was used for some of this work. The usual preference for single effect operation is the consequence of the limited evaporation temperature range ideally suited for the crystallization of a particular species; the VC mode is preferred because of its economy over the steam-heated mode for single effect VTE.

Another objective of this work was to develop design data and criteria for a possible follow-on project that would demonstrate the technical and economic advantages of the interface-enhanced method of VTE operation for the renovation-recycle of power plant coolant blowdown. The need for developing an acceptable and economical means of cooling tower blowdown renovation was underlined by EPA guidelines for the steam electric power industries. This also increased industry interest in such a development. The earlier development work on interface-enhanced VTE for seawater desalination (9) indicated it to be a promising candidate for such applications. Not only is the use of surfactant additives quite compatible with most wastewater evaporation, but the antifouling and antiscaling properties of such additives should provide added benefits.

In response to the author's proposal to evaluate the use of interface enhancement for industrial wastewater renovation, the EPA supported an earlier project, the results of which have been published (5). These studies showed that, for three different industrial wastewaters examined, VTFE provides clear technical and economic advantages over existing evaporation-distillation technologies. The types of wastewater examined were industrial boiler blowdown, acid drainage from pyrites slag piles, and power plant cooling tower blowdown. The objective of the initial study was to obtain heat transfer performance data on these three types of wastewater with both typical, best state-of-the-art VTE and with the interface enhanced VTFE mode of operation, under otherwise identical process conditions. In each case, it was shown that VTFE provided clear and substantial advantages, especially in terms of increased heat transfer performance. Generally, these wastewaters were subjected to a 10-fold concentration; recovery of solids or a maximal recovery of distilled water was not an objective; both preconcentration by VTFE and the follow-on crystallizing evaporation of cooling tower blowdown in the presence of surfactant additives were to be examined here.



## SECTION IV

### BACKGROUND

Industrial cooling accounts for more industrial water use in the U.S. than all others combined; about 80 percent of industrial water use is for heat rejection. Much of this use is on a once-through basis, the slightly heated water being returned to the environment. Since the temperature of the rejected coolant is slightly above its previous equilibrium with the environment, proportionately more will evaporate to readjust to equilibrium. This in effect degrades the water quality by concentrating dissolved solids. To reduce the adverse impact of industrial heat rejection, the EPA has promoted the use of cooling towers operated with zero discharge; the objective in this case is to reject heat directly into the atmosphere by evaporative cooling, using a much smaller flow of coolant but concentrating it to a dry or semi-dry residue during the evaporative cooling process.

The concept of cooling tower operating with zero discharge is not new; it is however more expensive than both once-through cooling and partial evaporation followed by discharge of a somewhat concentrated warm effluent.

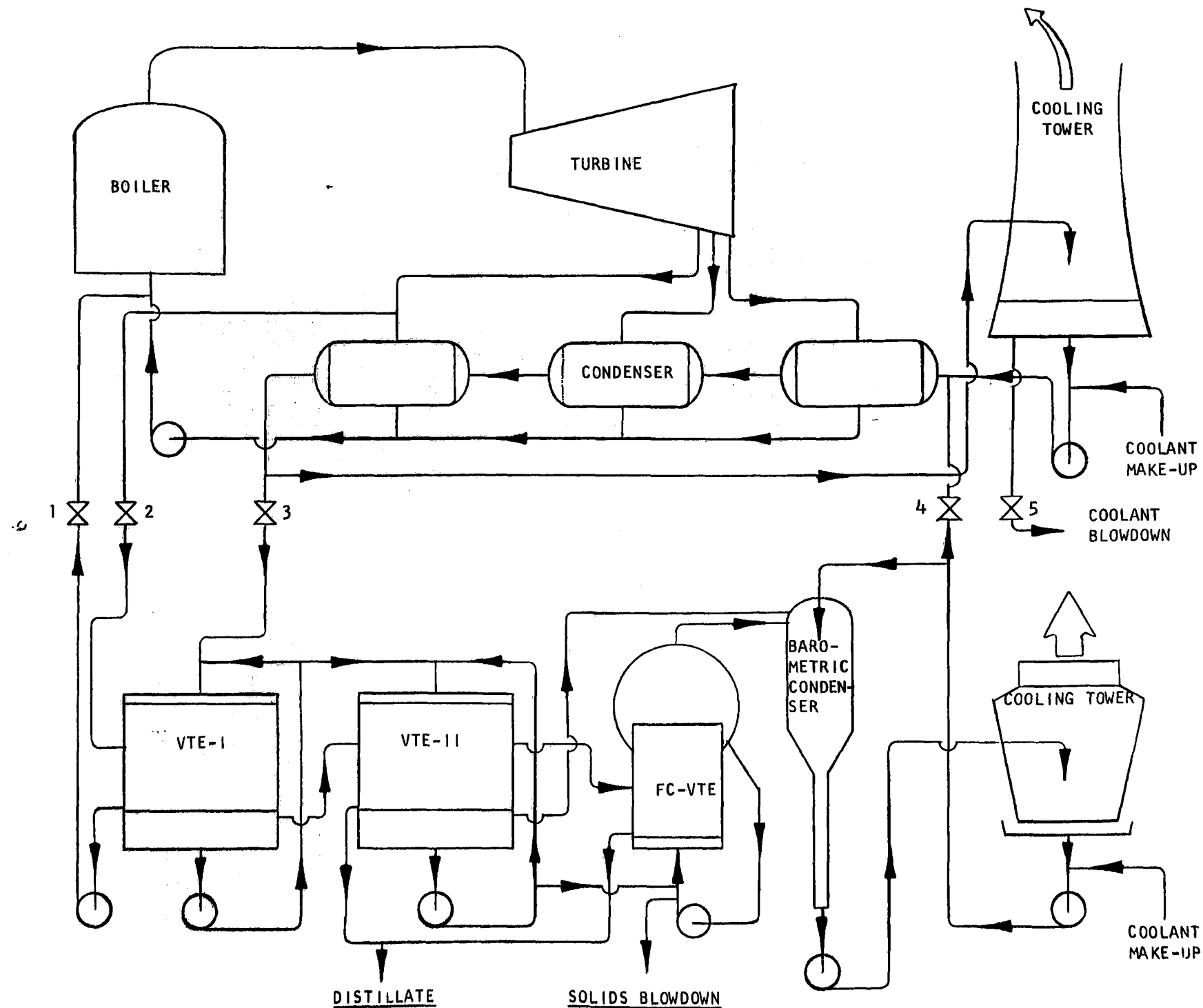
Development of VTFE promises economic improvements that should make adoption of cooling towers and zero discharge more acceptable to industry. Improved economy follows from the increased heat transfer performance of VTFE. This permits either a reduction in the heat transfer surface area required, or increased steam economy (increased performance ratio). Considering that heat transfer surface represents about 40 percent of the overall cost of evaporators, the approximately 100 percent improvement in heat transfer performance by VTFE provides up to 20 percent in capital cost savings. Alternatively, if the advantage of VTFE is realized in terms of an increased economy ratio, the cost of steam can be reduced significantly (by up to 30 percent). Ideally one would optimize for maximal overall cost savings by utilizing the increased design flexibility of VTFE. This increased flexibility permits improved trade-offs between capital cost reduction and fuel cost reduction, to best advantage for a particular industrial situation. In a situation where steam is available at a site at relatively low cost, one would design in favor of a smaller number of effects having an increased per-effect production. Where the cost of steam is relatively high, one would increase the number of effects to increase the ratio of product distillate to steam consumed. A similar situation pertains to the vapor compression VTE. When the cost of power is low, such as from a hydro-electric source, one would increase the size of a single-effect VTFE; on the other hand, a two-effect VTFE could prove advantageous where the cost of power to drive the compressor is high.

In addition the VTFE process permits the adoption of novel modes of operation previously only marginally feasible, or could add new capabilities to older VTE applications. One of these possible improvements addressed in this work is a novel power plant coolant flow diagram that permits the use of waste heat available within conventional coolant flow diagrams to renovate the cooling tower blowdown, producing distilled water for boiler feed, potable use or recycle as coolant, and a dry or slurried salt concentrate. Such an improved coolant flow diagram, presented in an earlier proposal to EPA for a scope of work germane to the work described here for VTFE applications development, is shown in Figure 1. The work reported here was planned to provide test data and design information needed as a preliminary to the earlier proposal. Work under that proposal is to proceed in the immediate future, building on the data and developments of this report. The overall objective of these two projects have been to develop, on an experimental basis, an advanced method of cooling tower blowdown renovation-recycle.

Figure 1 shows the proposed flow diagram that, if proven feasible by the work undertaken in this and the follow-on project, could be utilized by a pre-existing power plant. The main objectives of adopting such a flow diagram would be to renovate the cooling tower blowdown at low cost for recycle with zero blowdown, and to utilize a VTFE facility that would interface readily with normal power plant operation. Although at present still unproven, this flow diagram is presented here because it rationalizes much of the work undertaken in this as well as in the follow-on project.

One of the significant features of the flow diagram is that it permits almost instantaneous revision to the normal, pre-existing power plant mode of operation, should the need arise. This is done by simply closing four motorized valves (1 to 4) and opening valve number 5; the time required is only about 30 seconds. This responds to the concern of power plant operators about the possible adverse effects of a more complicated flow diagram in emergency situations. In actual fact this flow diagram reduces the potential for emergency situations by increasing the performance of the power plant. This occurs because of the additional heat dissipation capacity provided by a small cooling tower (forced draft), which reduces the normal heat load on the pre-existing cooling tower by about 5 percent.

The proposed add-on coolant blowdown renovation-recycle loop is shown in the lower half of Figure 1, with a pre-existing coolant flow diagram represented in the upper half. These two loops interface via four valves (1 to 5) shown across the middle of the diagram. The upper loop could be reverted to the pre-existing mode of operation at any time by closing valves 1 through 4 and opening valve 5 to resume the usual discharge of blowdown, for instance to a reservoir for later renovation. During the renovation-recycle mode of operation proposed, part of the turbine exhaust steam is diverted via valve number 2 and condensed in the first effect of the blowdown evaporation system; this condensate is returned to the boiler, through a pump and valve number 1. A portion of the heated coolant is diverted, as blowdown, to this vertical tube evaporator (VTE-1) via valve number 3. The vapor produced in the VTE-1 serves as heating steam for the VTE-2 and the forced circulation evaporator-crystallizer (FC-VTE). These



RENOVATION AND RECYCLE OF POWER PLANT COOLING TOWER BLOWDOWN

Figure 1

two units operate as a combination second effect, and the VTE-1 vapor condensed in these units provides distilled water available for a beneficial use such as boiler feed makeup, or potable quality fresh water, or for recycle to the cooling tower. Vapors produced in the VTE-2 and FC-VTE are returned to the main coolant loop as condensates from the barometric condenser, via valve number 4; dilution of this main coolant stream with condensates alleviates scale formation and reduces coolant makeup pretreatment requirements. The coolant makeup for the barometric condenser and small cooling tower need not be softened since it is continually diluted by the condensates in the barometric condenser followed by cooling through re-evaporation of essentially the same condensates in the small cooling tower. The nett effect of this blowdown renovation loop is to increase the capacities of the condenser and cooling tower, thereby increasing turbine generation capacity, and to produce some distilled water while reducing blowdown to a residue of solids, thus accomplishing zero liquid discharge by utilizing waste heat. The author presented this scheme in an earlier paper (10).

Several alternative subcycles are available within the generic coolant recycle system of Figure 1. All of those generic cycles are facilitated or rendered more economical by applying interface enhancement to the blowdown evaporation-concentration steps, by reducing the temperature difference ( $\Delta T$ ) required for cost-effective evaporation, or by increasing the rate or capacity of the evaporators. One alternative would be to use a backward feed mode, by condensing the turbine exhaust steam in the VTE-2 and FC-VTE where evaporation is subject to higher boiling point elevation penalties than in the VTE-1, and to use VTE-1 as the second, distillate-producing effect. Yet other alternatives available are combinations of VTE and FC-VTE in a single vessel or in a steam-side continuous vessel (11), and separate upflow and downflow effect arrangements. In the above flow diagram the evaporator and crystallizer of minimal capacity sufficient for zero discharge has been considered, especially for the case of a pre-existing power plant. An extension of this technique, if successful, is its application to future power plants, where the opportunity exists for designing the optimal coolant loop for zero liquid discharge. In such future designs, one of the three condensers shown in Figure 1 could be omitted and replaced with a vertical tube evaporator, to perform essentially the same condensation function while it simultaneously concentrates coolant blowdown (10, 11). If more than one conventional condenser could be replaced by evaporator-condensers (VTFE) of similar heat transfer duty, more useful distilled water can be produced. Considering that the heat transfer performance of a VTFE is several-fold higher than the conventional condenser heat transfer coefficient, the replacement of part of the condenser duty by a VTFE condenser can provide significant capital cost savings on copper-nickel heat transfer tubing. In addition, the need for coolant softening is reduced and may indeed be obviated if a sufficient portion of the coolant is diverted, as blowdown, to the VTFE and if the distilled water product is recycled to the coolant stream.

## SECTION V

### OBJECTIVES AND SCOPE OF WORK

The specific objectives of this work can be summarized as follows:

(a) To design, construct, install and instrument a second downflow VTE effect comparable to the pre-existing upflow effect of 5,000 gal. per day capacity.

(b) To obtain comparative upflow and downflow VTE data with the pilot plant facility using best state-of-the-art process conditions, to afford a basis for determining the relative merits of upflow and downflow VTE for wastewater renovation-recycle.

(c) To obtain comparative upflow and downflow VTFE performance data after the addition of a selected surface active agent to the feed, using process conditions consistent with those used under (b), to provide a basis for evaluating up- and downflow VTE and VTFE for industrial wastewater renovation.

(d) To design and assemble an evaporator-crystallizer (EC) facility capable of the seed-slurry feed mode of operation and to provide instrumentation of sufficient sensitivity for definitive test data.

(e) To obtain heat transfer performance data during crystallization by evaporation, under state-of-the-art process conditions.

(f) To obtain heat transfer performance data when a selected surfactant has been added to the seed-slurry feed.

(g) From the above series of tests, provide design criteria for a follow-on project that would have as its objective the design, construction and field test operation of a mobile pilot plant, for the renovation-recycle of cooling tower blowdown at an existing power plant site, utilizing VTFE. This mobile pilot plant would comprise an evaporator for preconcentration of the blowdown, an evaporator-crystallizer and means for separating crystalline or solid residues from the concentrated blowdown, during evaporation crystallization.

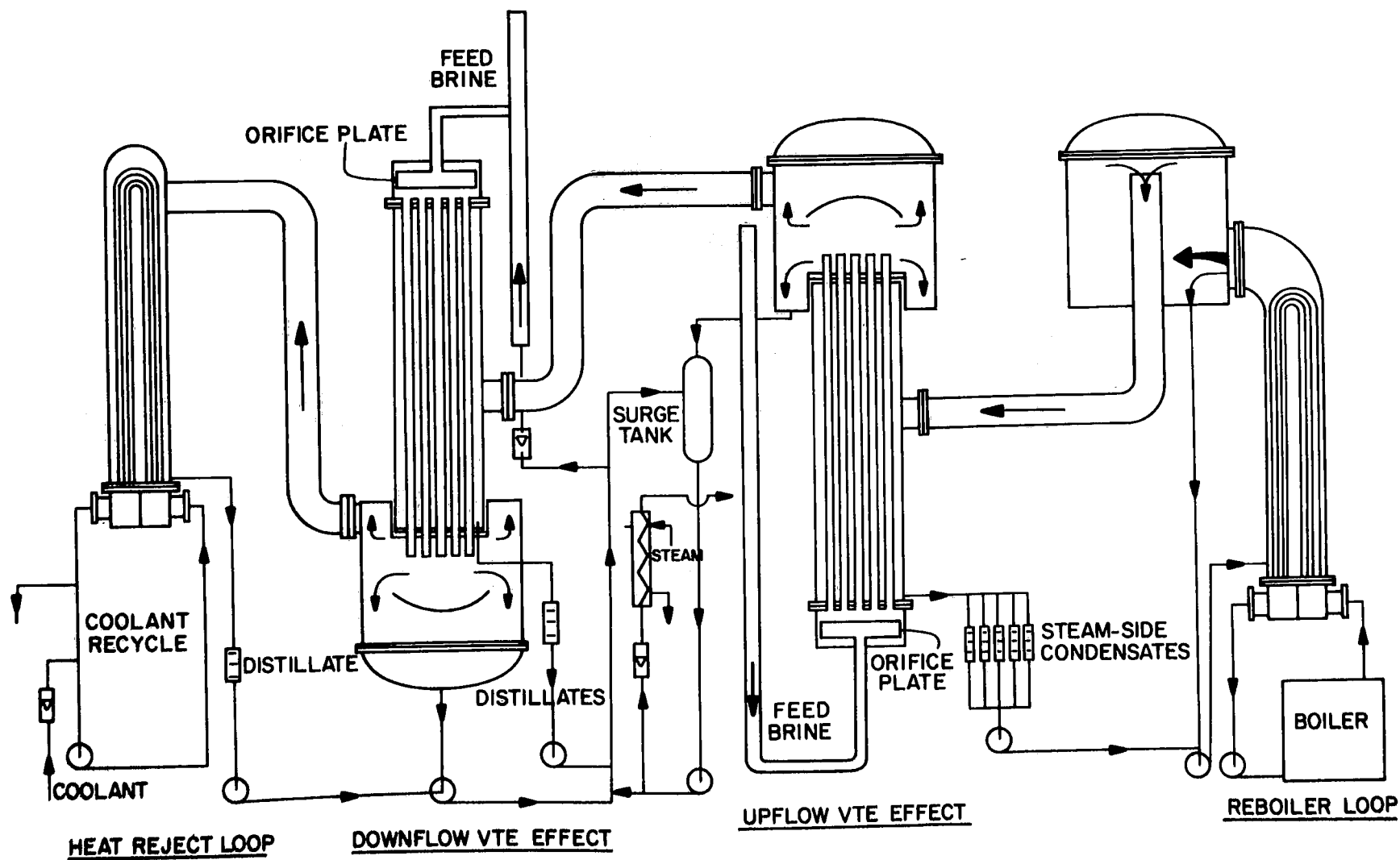
## SECTION VI

### UPFLOW-DOWNFLOW VTE TEST FACILITY

The two-effect upflow-downflow VTE pilot plant of 10,000 gpd capacity, assembled under this project is represented by the simplified flow diagram of Figure 2, and as a photographic print in Figure 3. It utilized full-sized 2-inch OD x 10-foot long commercial aluminum-brass distillation tubes of the double-fluted type supplied by Yorkshire Imperial Metals Ltd., England in 18-tube bundles, removably installed with 'O'-ring seals. Aluminum-brass tubes were selected over equally-acceptable 90-10 copper-nickel tubes on the basis of availability and cost. Materials used for constructing the brineside of the VTE effects were stainless steel, brass, bronze, copper and Pyrex glass, to reduce fouling by corrosion products. Two identical VTE effects were tested, in series connection with two end effects serving respectively as heat source and heat sink, one on either side of the two real effects. The second of the two real effects was rotatably mounted to provide the consecutive upflow-downflow test data reported here. Temperature and flow controls permitted steady-state operation of this 4-unit evaporator at any selected temperature level within the usual multieffect VTE temperature span of 100 to 240°F, to simulate the most significant VTE multieffect process conditions. Instrumentation allowed data and observations for defining the most significant process parameters and modes of two-phase flow for any effect of both upflow and downflow multieffect operation, on a comparative basis.

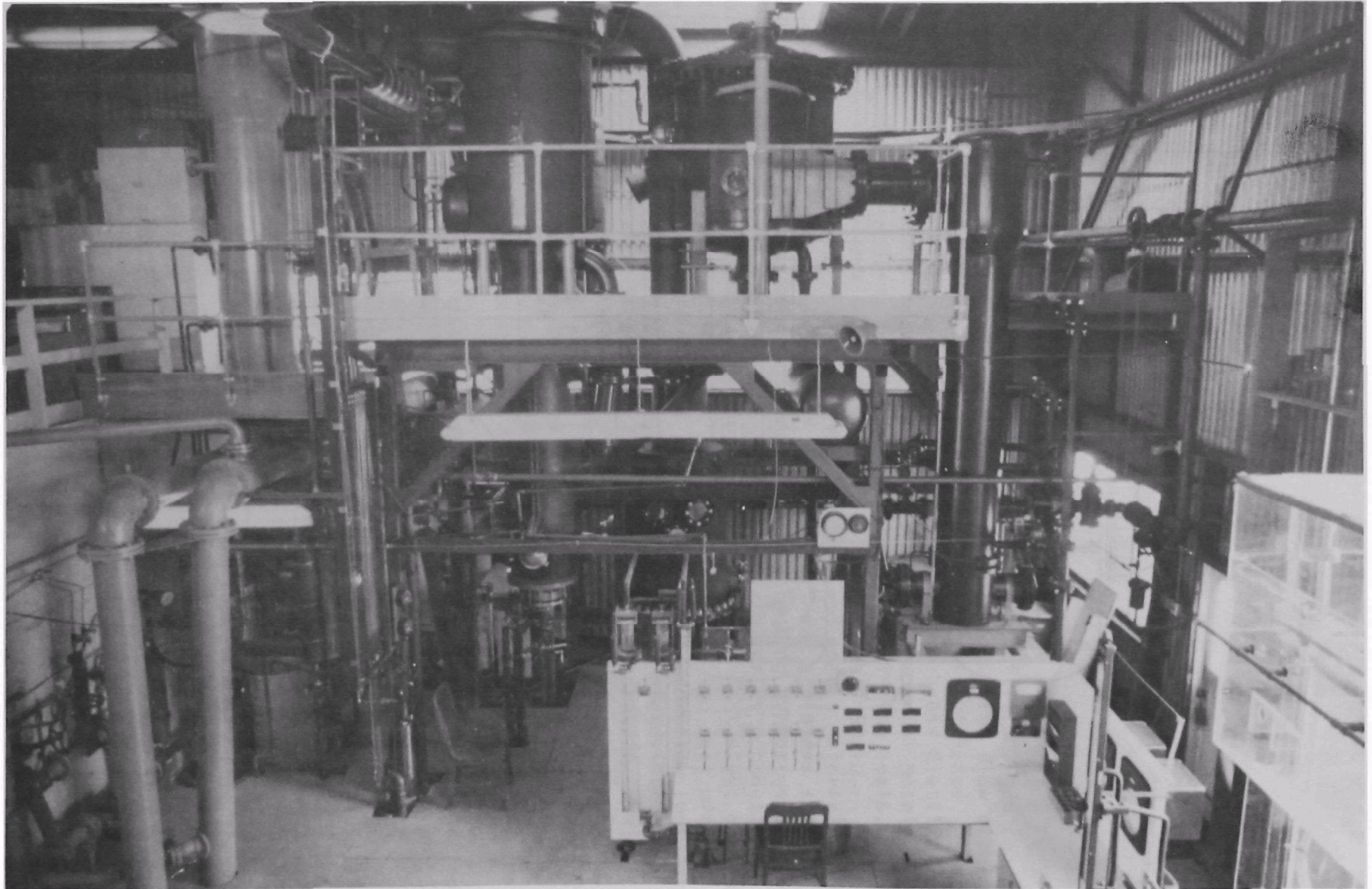
Clean steam was allowed to the upflow (first) effect at a selected temperature from the reboiler loop shown on the right of Figures 2 and 3. A pneumatic control valve passed 45 psi steam from a boiler into the tubeside of a vertical U-tube heat exchanger to maintain the reboiler steam, generated on the shell-side, at the selected temperature ( $\pm 0.1^\circ\text{F}$ ). Condensates from the first effect VTE were collected simultaneously from four individual distillation tubes and, collectively, from the remaining 14 tubes in this bundle. These condensate flow rates were determined by stopwatch, timing the intervals required to fill calibrated cylindrical collection vessels; and heat fluxes ( $Q$ ) were derived directly from these flow rates. Steamside design and tube spacings were as shown in Figure 4. Indicated are the positions of the four individually drained tubes, the remaining 14 tubes of the bundle, and dummy tubes and baffles to ensure proper steamside flow and venting into a 1.5-inch x 10-foot long vent collecting tube. All condensates, including the vent condensate, were collected, their flow rates measured and returned to their appropriate liquid cycles while being maintained under air-free (vacuum) conditions. Noncondensibles were continuously removed by bleeding some vapor to a separate condenser and vacuum pump.





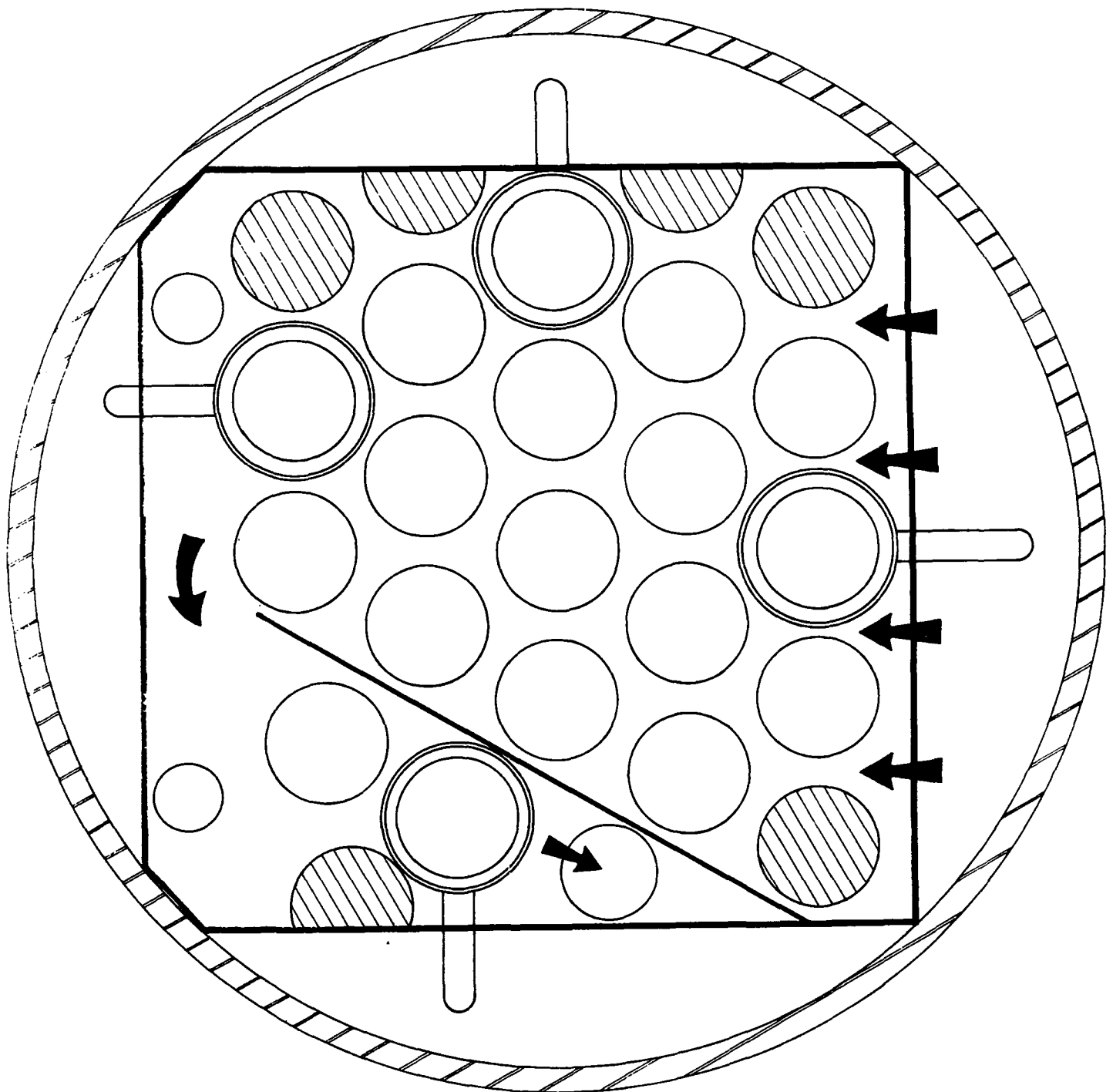
SIMPLIFIED FLOW DIAGRAM, TWO-EFFECT UPFLOW-DOWNFLOW PILOT PLANT

Figure 2



TWO-EFFECT UPFLOW-DOWNFLOW VTE PILOT PLANT

Figure 3



**STEAM SIDE FLOW PATTERN**

Distillation tubes were spaced 0.5-inch on a triangular pattern, with dummy tubes (shaded) and baffles to ensure continuous steamside flow from the plenum to the vent tube along the entire bundle length. The vent tube had 0.125-inch holes drilled at 6-inch intervals along its entire length for adequate vent collection.

Accurate temperature measurements were obtained for both effects, with calibrated platinum resistance probes that provided a digital display and direct printout. These probes were located in the steamsides ( $T_s$ ) within the tube bundles, in the brine ( $T_b$ ) flowing from the effect outlets, and in the feed ( $T_f$ ) liquid before flashdown and distribution to the tubes. These temperature data were checked by vapor pressure measurements with an absolute manometer (mercury-filled), corrected for boiling point elevation by reference to literature data, and by mercury-in-glass thermometers. Heat transfer performances were determined as the overall heat transfer coefficients:

$$U = \frac{Q}{A\Delta T} \quad \text{Where } A \text{ is the nominal tube outside surface area based on nominal OD, 2-inch; and } \Delta T = T_s - T_b.$$

Vapor produced in the first effect VTE served as heating steam for the second effect. Venting of the second effect was similar to the first effect, and all condensates (distillates) were collected and returned to the brine cycle under evacuated conditions after their flow rates were measured as above.

Vapor produced in the second effect was condensed in the heat reject loop, with continuous bleed-off of non-condensibles as above. Heat rejection at constant flux, was to a large volume of preheated coolant recycled at about 1,000 gpm through the vertical U-tube heat exchanger shown on the left of Figures 2 and 3, with a continuous addition of cold water to and simultaneous rejection of hot water from the recycled coolant. This coolant was maintained under 40 psi pressure, and the rate of addition of cold water and rejection of hot coolant were controlled to provide the desired temperature difference over both effects ( $2\Delta T$ ), with reference to the temperature selected for the reboiler cycle.

The evaporation of several types of distillands was examined with this VTE facility, including seawater and industrial wastewater, under both conventional and interface-enhanced process conditions. The feed was deaerated-decarbonated by recycling it under vacuum through both effects in series, and by maintaining the pH at 6.6-6.8 by the addition of sulphuric acid as required (50-100 ppm). Feed flow to the first effect was controlled at one gallon per minute per tube, passed through a heat exchanger to adjust its temperature to within  $1^\circ\text{F}$  of the steamside of the upflow effect, and discharged into a standing leg that was vented to the first effect vapor dome. Feed flow through the upflow effect orifice plate distributor was subject only to the level in the standing leg (measured with a sight glass), the effect-to-effect thermal heat and the 18 orifices of  $\frac{1}{4}$ -inch diameter, one per tube. Flashing of the feed occurred as it passed through the orifice plate in all tests; this condition was important for good upflow, two-phase flow stability and high heat transfer performance. Upflow of the feed through the distillation tubes was by vapor lift due to continuous further evaporation along the tube length. The orifice plate was adjustable in elevation, to provide an experimentally variable gap (plenum) between its upper surface and the lower tube sheet, and could also be rotated to direct feed either directly into the distillation tubes or to

impinge against the tube sheet between the tubes. The tubeside pressure drop for the bundle was obtained from a sight glass connected to the tube inlet plenum (post-orifice) and the vapor dome. Vapor-liquid disengagement in the first effect dome was by impingement against a saddle-shaped splash plate, and the vapor was passed through a stainless steel demister screen to the steamside of the second effect. Residual feed (brine) cascaded over the upper tube sheet and into a surge tank, and on to the second effect.

For the downflow VTE tests, the second effect was provided with a pump to recycle the feed at a measured rate (2 to 2.5 gpm per tube) and to discharge it into a standing leg, vented back to the surge tank. Feed to the downflow effect was thus mainly by recycle (55 to 65%), with first effect blowdown and recycled distillates as makeup. Feed flow to the downflow bundle was controlled by the level in the standing leg, the degree of superheat in the feed and the orifice diameters. The downflow orifices were interstitial to the distillation tube positions, comprising 27 drilled holes of 0.2-inch diameter through the orifice plate, for feed distribution at three locations peripheral to each distillation tube. Flashdown of the feed as it passed through the orifice plate occurred in all tests using this distributor. Vapor generated in the downflow effect was separated from the residual liquid phase in the vapor dome below, passed through a demister screen and condensed in the heat reject loop. The distillate flow rate, and the temperatures of the steamside ( $T_s$ ), blowdown brine ( $T_b$ ) and feed ( $T_f$ ) were determined as above, to provide heat transfer performance data. Residual brine and distillates were recycled to the downflow standing leg, with continuous removal of noncondensibles by bleed-off of some vapor to a condenser and vacuum pump as above.

## SECTION VII

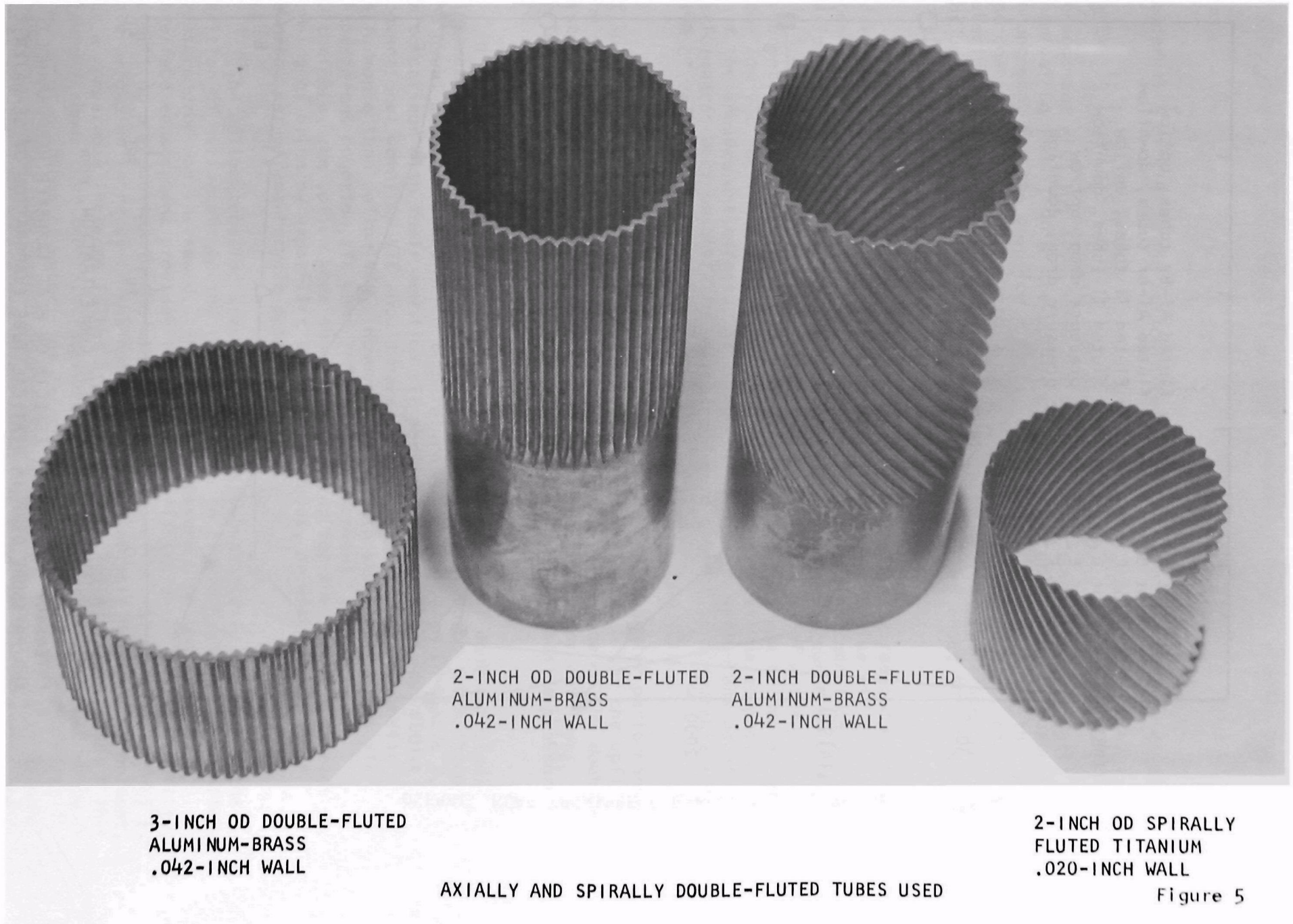
### COMPARATIVE UPFLOW-DOWNFLOW VTE AND VTFE TESTS

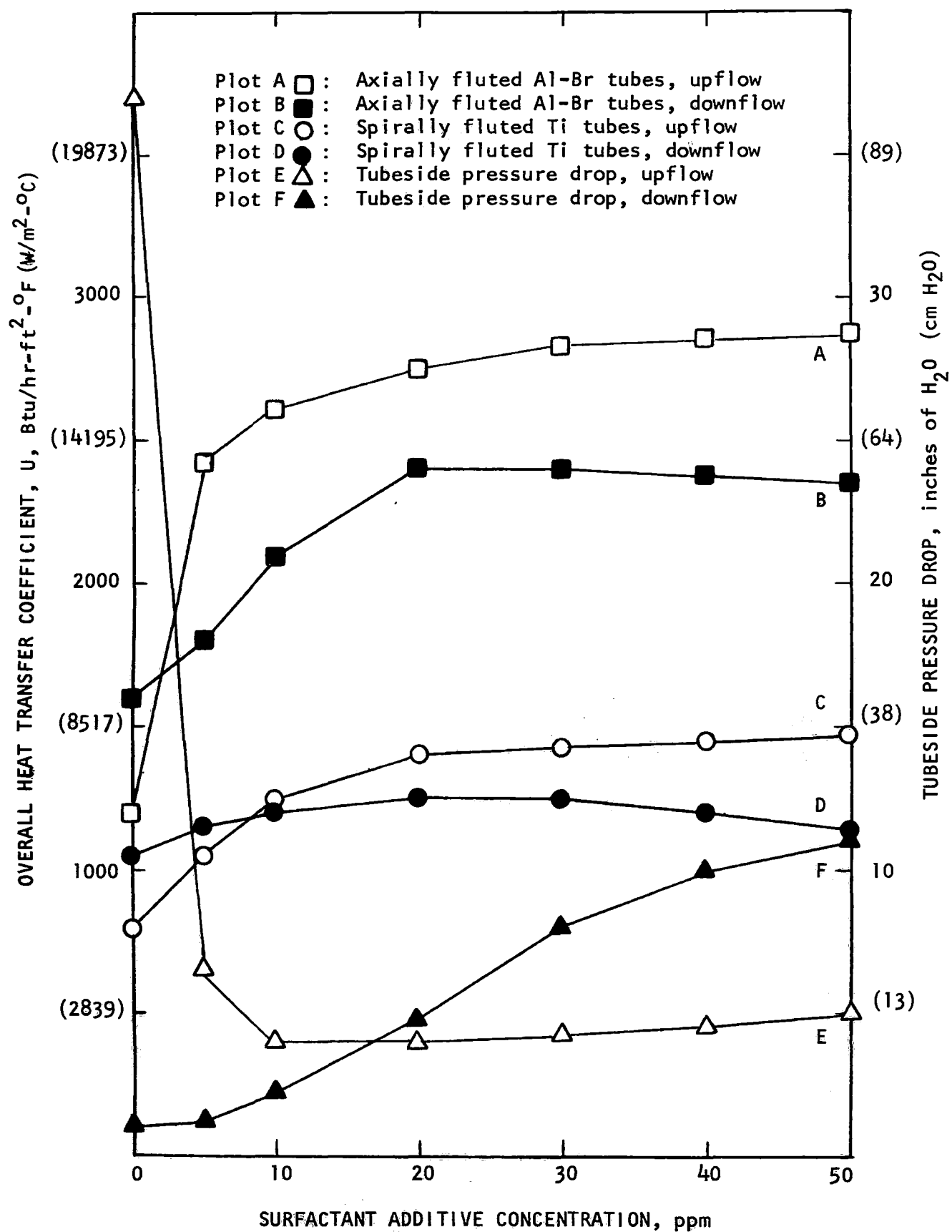
Earlier EPA-sponsored work (5) demonstrated that the use of interface enhancement provided a substantial increase in the heat transfer performance of upflow VTE of Mohave power plant cooling tower blowdown; increases in U of about 125 percent were obtained under realistic upflow VTFE process conditions, with double-fluted aluminum-brass tubes, and significantly enhanced heat transfer performance data during the 10-fold concentration of 800 gallons of this blowdown by upflow VTFE, was also obtained. In the present work the objective was to provide reliable comparative data on a basis of which one could elect when to use downflow VTFE in preference to upflow VTFE. These tests could be carried out with any aqueous feed water having evaporation properties similar to cooling tower blowdown. The most readily available source was clean seawater, collected off the California coast at the U.C. Bodega Marine Laboratories site; about 1,500 gallons<sup>a</sup> were pumped into two clean wooden wine barrels and transported to this Laboratory by truck, for the tests reported here. Seawater contains 35,000 ppm of total dissolved salts (TDS), about three times as high as the TDS in Mohave blowdown. Some VTE tests were also carried out with freshwater feed during preliminary, shake-down runs with this pilot plant, and served to also confirm the seawater data reported here.

Interface-enhanced performance data were obtained with two types of double-fluted tubes, with seawater feed. Tubes were axially double-fluted aluminum-brass (supplied by Yorkshire Imperial Metal Industries Limited, England) of 2-inch OD x 10-foot heated length, 0.042-inch wall, and spirally double-fluted titanium tubes (Timet, supplied by Oak Ridge National Laboratories and fluted by Yorkshire Imperial Metals) of 2-inch OD x 10-foot heated length x 0.020 wall. The titanium tubes were in an apparently clean but previously used condition, and were installed as received without cleaning; the aluminum-brass tubes were in an oxidized-annealed and stabilized condition after four years of intermittent testing in the upflow VTE effect without reconditioning. Their steamsides were in a dull, dark brown condition. Their brinesides were in a fairly clean condition because of the dispersant and flushing action of surfactant in seawater at a pH of 6.6-6.8. These double-fluted tube profiles are shown in Figure 5.

Heat transfer performance data were obtained after each steady-state operation had been reached and maintained for a sufficient time, usually requiring about 30 minutes for each set of data. Figure 6 shows typical interface enhancement effects on the overall heat transfer performance and the tubeside pressure drop for the upflow and downflow effects with seawater feed. Performance and pressure drop data were plotted as a function of the concentration of surfactant additive in the feed, at an evaporation







INTERFACE ENHANCEMENT EFFECTS ON HEAT TRANSFER PERFORMANCE AND PRESSURE DROP IN VERTICAL TUBE EVAPORATION OF SEAWATER, UPFLOW AND DOWNFLOW, WITH DOUBLE-FLUTED TITANIUM AND ALUMINUM-BRASS TUBES

temperature of 210<sup>a</sup> in the upflow effect and 200<sup>oFb</sup> in the downflow effect.

It is apparent that interface enhancement is more effective in upflow than in downflow VTE; it provided about a 100 percent heat transfer enhancement with the aluminum-brass tubes after 5 ppm of Neodol (Shell Chemical Co.) addition in the upflow effect versus only about 13 percent enhancement for the downflow effect. At 20 ppm of foaming agent the upflow and downflow performances were within 15 percent of one another, and their tubeside pressure drops were nearly equal. The maximal interface enhancement for upflow VTE was in this case about 130 percent, and about 50 percent for downflow.

The upflow tubeside pressure drop was dramatically reduced by the addition of 5 to 10 ppm surfactant, reaching a minimal value 11% of its unenhanced value, and rising only marginally with further addition of surfactant. The downflow pressure drop was gradually increased by surfactant addition to the feed, rising from 0.5 to about 12 inches of water. It is apparent that upflow VTE is substantially improved by 5 ppm of Neodol while downflow VTE required about four times higher concentrations to provide half the enhancement effect. At 20 ppm of Neodol the upflow and downflow performances were essentially maximal and were subject to comparable effective  $\Delta T$  values.

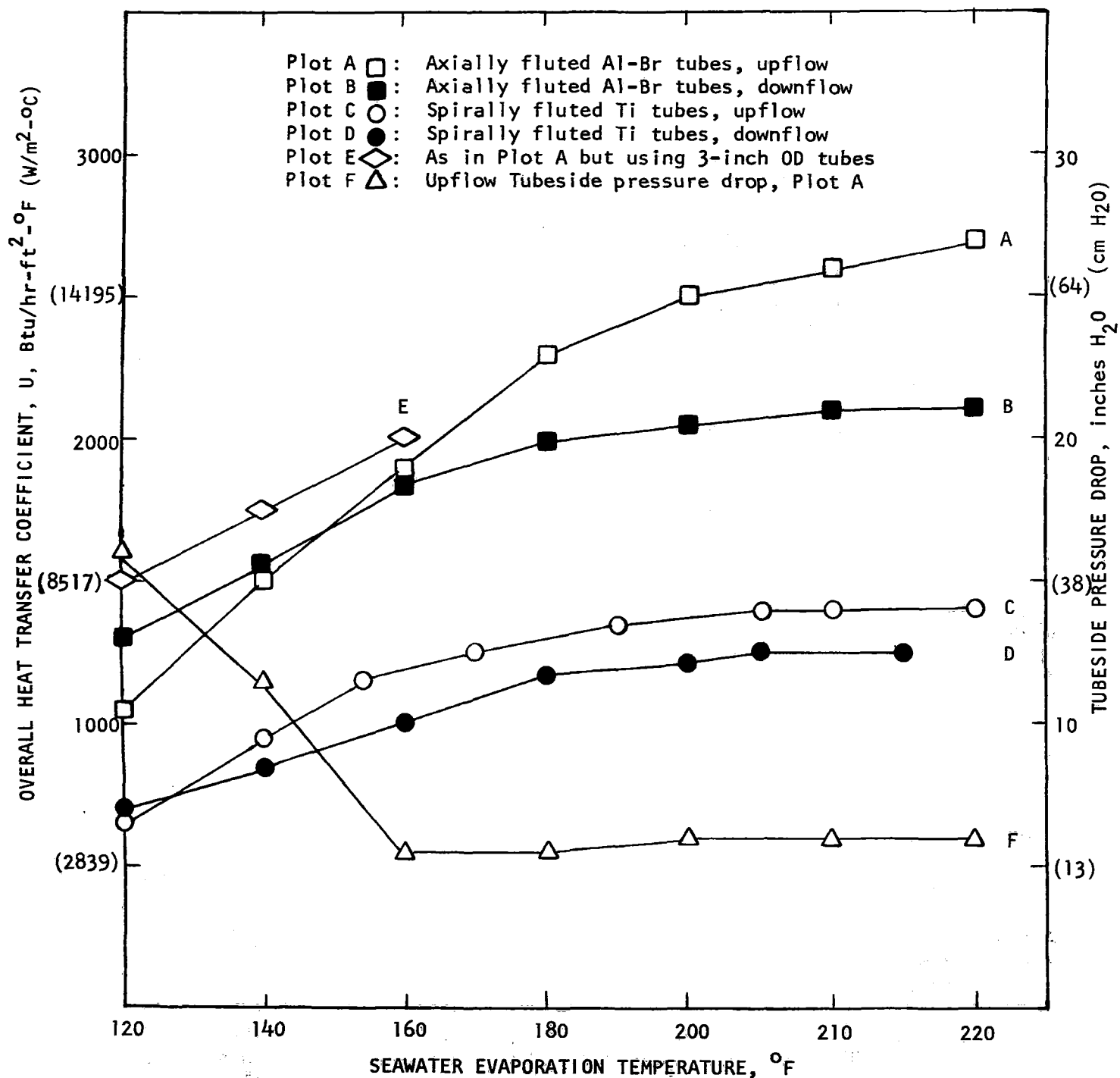
The interface enhancement effects on the heat transfer performance of spirally double-fluted titanium tubes shown in Figure 6 for the upflow and downflow VTE modes caused responses parallel with those of the double-fluted aluminum-brass tubes, but at an approximately 40 percent lower performance level, in both upflow and downflow modes. Tubeside pressure drops were not determined for these tubes individually.

The interface-enhanced upflow VTE performances of double-fluted aluminum-brass tubes and of spirally double-fluted titanium tubes through the usual seawater evaporation temperature range are shown in Figure 7, in comparison with their downflow VTE performances. It was known that conventional downflow VTE should have a slightly higher heat transfer performance than upflow VTE. However, these data show that upflow VTE outperforms downflow VTE when interface enhancement is applied. Furthermore, the upflow mode should be credited for the effect-to-effect pumping of brine by vapor-lift through the tubes, or with the costs of downflow per-effect recycle pumping.

The upflow tubeside pressure drop was substantially reduced by the addition of surfactant to the feed, as shown in Figure 6 while the downflow pressure drop was increased. This upflow pressure drop reduction by interface enhancement provides for substantially increased upflow stability, permitting a reduction in the minimal effect-to-effect  $\Delta T$  by about 4<sup>c</sup> to 5<sup>oF.d</sup>. This allows an increase in the number of upflow effects by about 50 percent, for a comparable increase in the steam to product (gain) ratio. Plot F in Figure 7 shows that the interface-enhanced upflow pressure drop was quite low, about 6 inches of water, for 2-inch tubes between 160<sup>e</sup> and 200<sup>oFb</sup>. It was shown earlier that 3-inch double-fluted tubes are preferable to 2-inch tubes for the low temperature effects, both in terms of pressure drop and

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<sup>a</sup>98.9<sup>oC</sup>; <sup>b</sup>93.3<sup>oC</sup>; <sup>c</sup>2.2<sup>oC</sup>; <sup>d</sup>2.8<sup>oC</sup>      21      <sup>e</sup>71.1<sup>oC</sup>



INTERFACE-ENHANCED UPFLOW AND DOWNFLOW VTE PERFORMANCE AT SEAWATER EVAPORATION TEMPERATURES 120<sup>a</sup> TO 220<sup>b</sup>  $^\circ\text{F}$  WITH DOUBLE-FLUTED ALUMINUM-BRASS AND TITANIUM TUBES AND USING 10 PPM OF SURFACTANT ADDITIVE

<sup>a</sup>48.9 $^\circ\text{C}$ ; <sup>b</sup>104.4 $^\circ\text{C}$

Figure 7

interface-enhanced heat transfer performance. Plot E of Figure 7 indicates that 3-inch double-fluted tubes should be used for effect temperatures up to about 170°F.<sup>a</sup> For effect temperatures above 170°F<sup>a</sup> the 2-inch tubes would be more advantageous, as shown by plot A.

The mode of two-phase flow in the upflow tubes changed significantly upon the addition of a surfactant, as also noted earlier (9). A rather wet foamy condition was imposed immediately after the orifice, and the outflow conditions for both upflow and downflow modes were essentially as foamy annular layers rather than liquid films. The vapor core flow from the tubes appeared to entrain fewer liquid droplets after surfactant addition than before it, for equivalent VTE process conditions. Flow from the upflow tubes was more continuous with than without surfactant; the latter is normally a distinctly gushing flow (intermittent spurts). The dramatic reduction in the upflow pressure drop is very significant; it caused a substantial improvement in the upflow hydrodynamic stability; it also increased the effective  $\Delta T$  available for vaporization (13). This increase in effective  $\Delta T$  is, however, not the only cause of increased heat transfer performance, as evidenced by the fact that in Figure 6, Plots A & E, the performance was increased further by the addition of surfactant above the 20 ppm level while the pressure drop also increased slightly.

The significance of pressure drop in interface-enhanced upflow VTE for distillation tubes of different diameters was extensively examined and discussed in recent work done in this Laboratory (14).

The pressure drop response to surfactant addition, observed for the downflow mode, has not been reported earlier, and further work is required to characterize it. In this case it included the pressure drop due to channeling the two-phase foamy layer from the top tube sheet upper surface into the downflow tubes at 2.5 times the rate of feed flow for the upflow tubes. A slight reduction in the downflow enhancement effect at surfactant concentrations above 20 ppm was observed in each downflow case.

The mechanism of heat transfer performance enhancement after the addition of a surfactant was postulated as being the result of causing a foamy layer to flow over the heat transfer surface, providing a thin-film, wiped-film mode of flow over this surface (1, 7, 14). This liquid film was seen as being continually renewed by liquid traveling in the interstices between vapor bubbles being sheared over the surface as the annular foamy layer or network sweeps over it. This mechanism has recently been defined more precisely, based on definitive work in this Laboratory on the upflow, two-phase flow morphology under typical VTE process conditions (15).

The interface-enhanced performance data for 3-inch and 2-inch double-fluted aluminum-brass tubes (Figure 7, Plots A & E) provide a basis for a multieffect plant design study. Such data, modified by the appropriate fouling factors, were utilized in the joint study with Kaiser Engineers of Oakland, California, to arrive at the preliminary technical feasibility evaluation of VTFE, part of which was published (6).

The heat transfer performance of titanium tubes was somewhat lower

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<sup>a</sup>76.7°C

than anticipated from their wall conductance, and was about equal to the comparable performance obtained with axially double-fluted titanium tubes, to be reported elsewhere (12). This rather low performance may be attributed to the less sharply defined, less angular ridge profile obtained when fluting a titanium tube as compared with an aluminum-brass tube. It is apparent that titanium tubes may be less economical than aluminum-brass tubes for VTE use except in cases where the corrosion-erosion characteristics of the feed are of compelling significance, for instance in the case of an acidic wastewater feed.

It is clear, from the above test series, that vertical tube foam evaporation (VTFE) offers a promising new approach to wastewater renovation-recycle. Applied to upflow VTE, it improves two-phase flow stability substantially, reducing the tubeside pressure drop by a factor of 3 to 10 and increasing the heat transfer performance by about 100 percent. It also provides the basis for increasing the number of effects normally permissible within the usual available temperature span, in the interest of fuel economy; this also improves design flexibility. Interface enhancement eliminates the main question that has impeded the application of multi-effect upflow VTE in preference to the downflow mode for distillation. When applied to downflow VTE, interface enhancement provides a relatively lower but significant heat transfer performance increase, sufficient to merit an attempt at improving the performance of existing downflow VTE plants. Existing upflow VTE plants could also benefit from VTFE after installation of orifice plates.

The relatively low performance of double-fluted titanium tubes, compared with aluminum-brass tubes, indicates that the selection of titanium must be justified on a basis of erosion-corrosion considerations and cost. This will be the case for many industrial wastewaters, especially those derived from metal industries, acidic mine effluents, and other corrosive effluents such as those relatively high in sulfides, ammonia or amines.

It is apparent that, for evaporation to concentrate relatively dilute wastewaters within a concentration range where precipitation and fouling are not serious hazards, upflow VTFE is to be preferred over downflow VTFE and VTE. This preference of upflow VTFE is supported by both its relatively high heat transfer performance and process simplicity.



## SECTION VIII

### FACILITY FOR EVAPORATION-CRYSTALLIZATION WITH INTERFACE ENHANCEMENT

A vertical tube evaporator-crystallizer, capable of 5,000 to 7,000 gallon per day capacity was used to obtain comparative performance data for the conventional slurry-feed mode of operation and for the interface-enhanced method of operation. For this purpose, a pre-existing evaporator (16) was adapted by providing the necessary pumps, flow controls and instrumentation. A basket type centrifuge was also provided to separate crystalline product from the blowdown slurries. The main objectives were to determine the feasibility of interface-enhanced evaporation-concentration into the crystallizing concentration range, to determine the heat transfer performance effects of the enhancement additive and to observe its effects on crystallization, fouling and scaling of the heat transfer surfaces. These data and observations were obtained with both synthetic salt solutions and with real cooling tower blowdowns obtained from several industrial sources.

The vertical tube evaporator (VTE) used for this work is shown in a flow diagram sketch in Figure 8 and as a photographic print in Figure 9. The VTE contained a 49-tube bundle of double-fluted aluminum-brass distillation tubes (Yorkshire Imperial Metals Ltd.) of 1.5-inch diameter by 6-foot heated length. Feed distribution to this tube bundle was in the downflow mode, using an orifice plate distributor for dispensing the feed through 0.20-inch holes on to the top tube sheet at three positions adjacent to each tube inlet end. The feed was thus bounced off the top tube sheet, and directed downward into the tubes by means of short cylindrical deflectors attached to the orifice plate and entering the distillation tube inlet ends by about 0.5-inch to provide an annular feed distribution for each tube. The orifice plate with cylindrical deflectors attached is shown (upside down) in Figure 10. Provision was made for preheating the feed with two electrical immersion heaters of 3,000 watt each, located in the inlet distributor vessel, immediately above the orifice plate. The 49-tube bundle discharged from its open-ended outlet ends directly into a large stainless steel vessel for separating the vapor produced from the feed slurry. This vessel was provided with three windows to permit observation of the modes of flow at the tube outlet ends. A pair of rectangular windows (visible in Figures 11 and 12) located in the inlet distributor vessel similarly permitted observation of the mode of flow between the orifice plate and the tube inlet ends. The vapor-liquid separation vessel was provided with an annular stainless steel mesh mist eliminator located in the upper part of the vessel as a collar surrounding the tube bundle, for the purpose of removing entrained droplets of liquid phase from the vapor produced. Vapor was conducted from the mist eliminator to a condenser cooled with water coolant of controlled temperature, to provide steady-state

SCHEMATIC FLOW DIAGRAM  
DOWNFLOW EVAPORATOR-CRYSTALLIZER

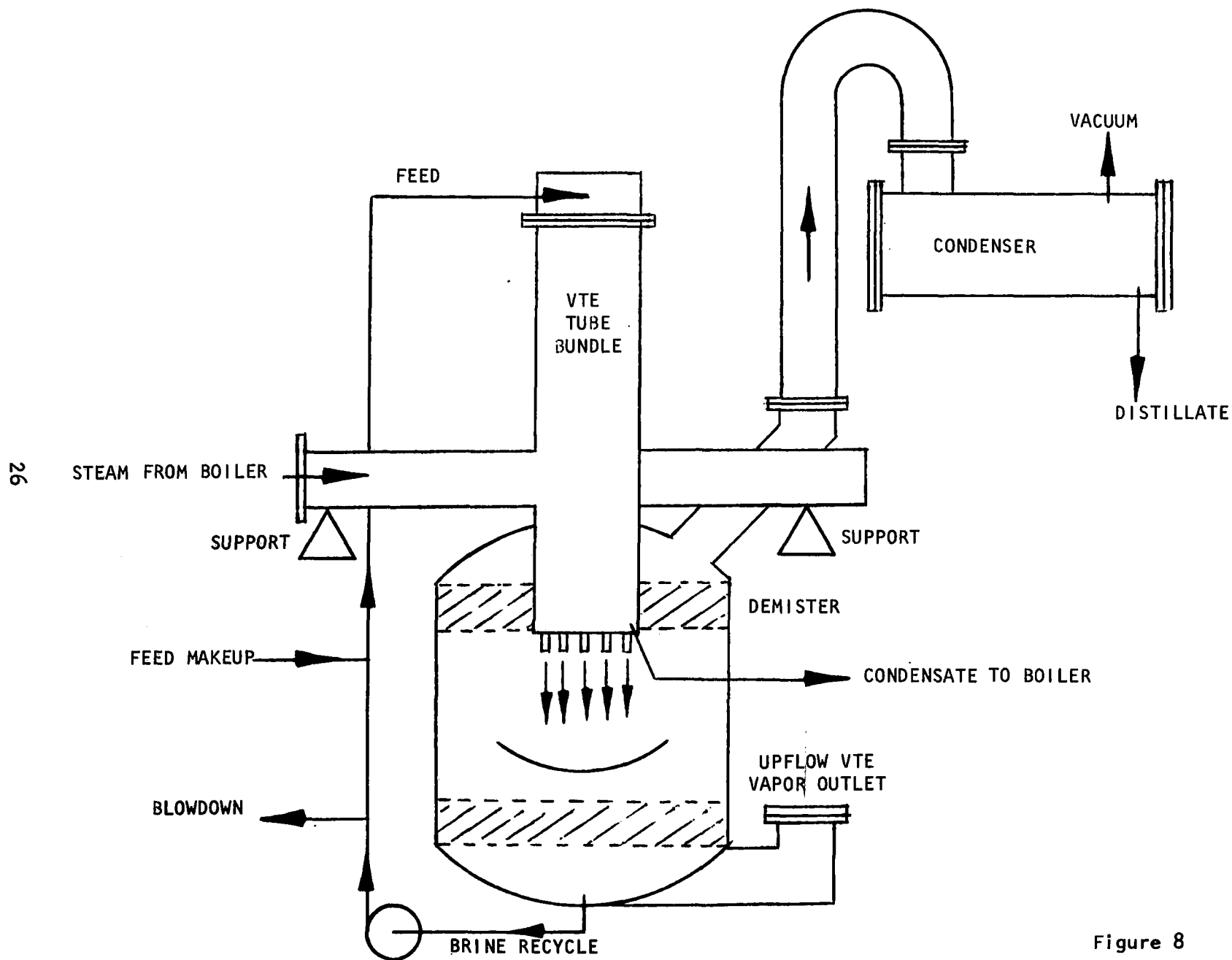
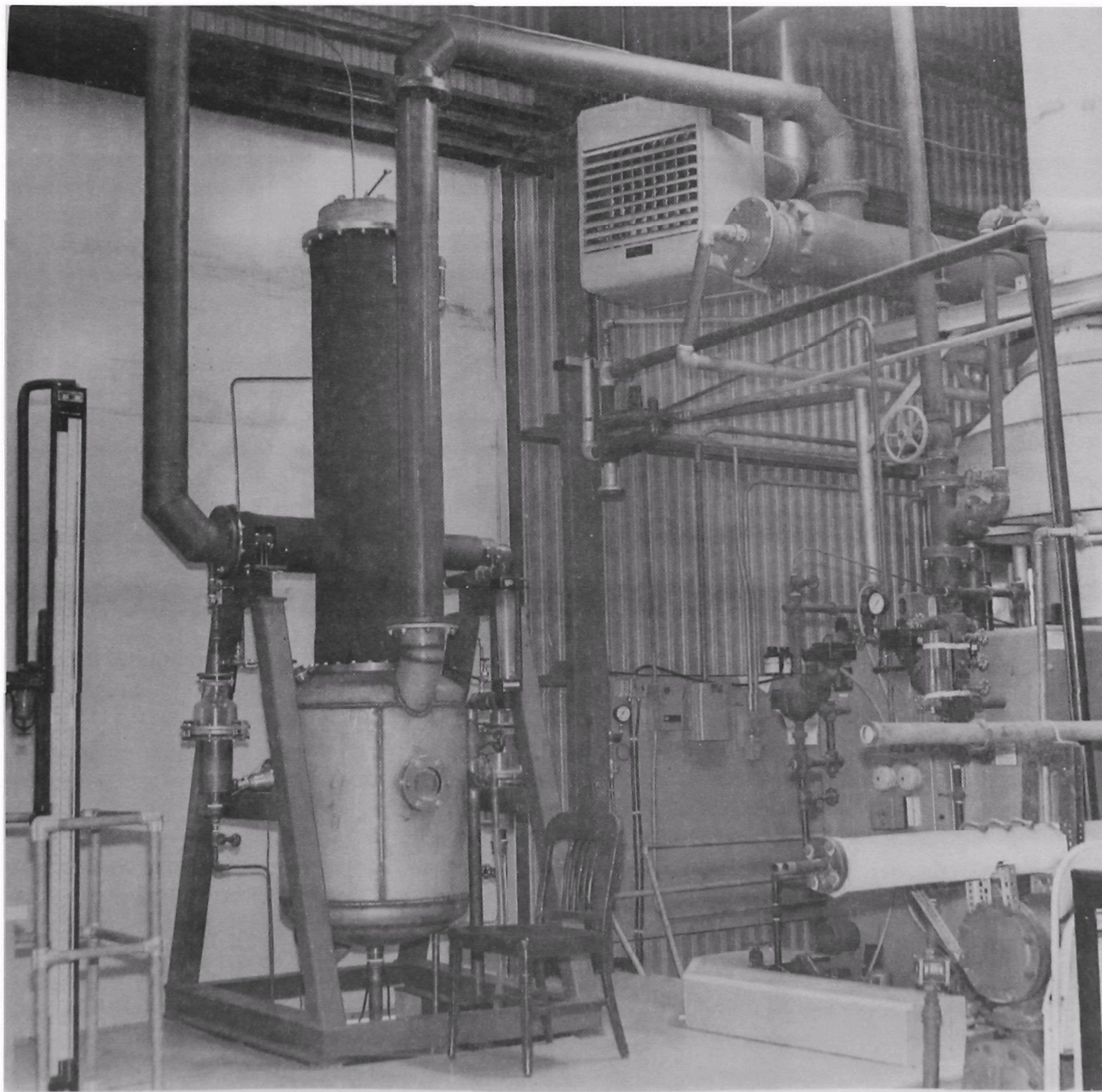
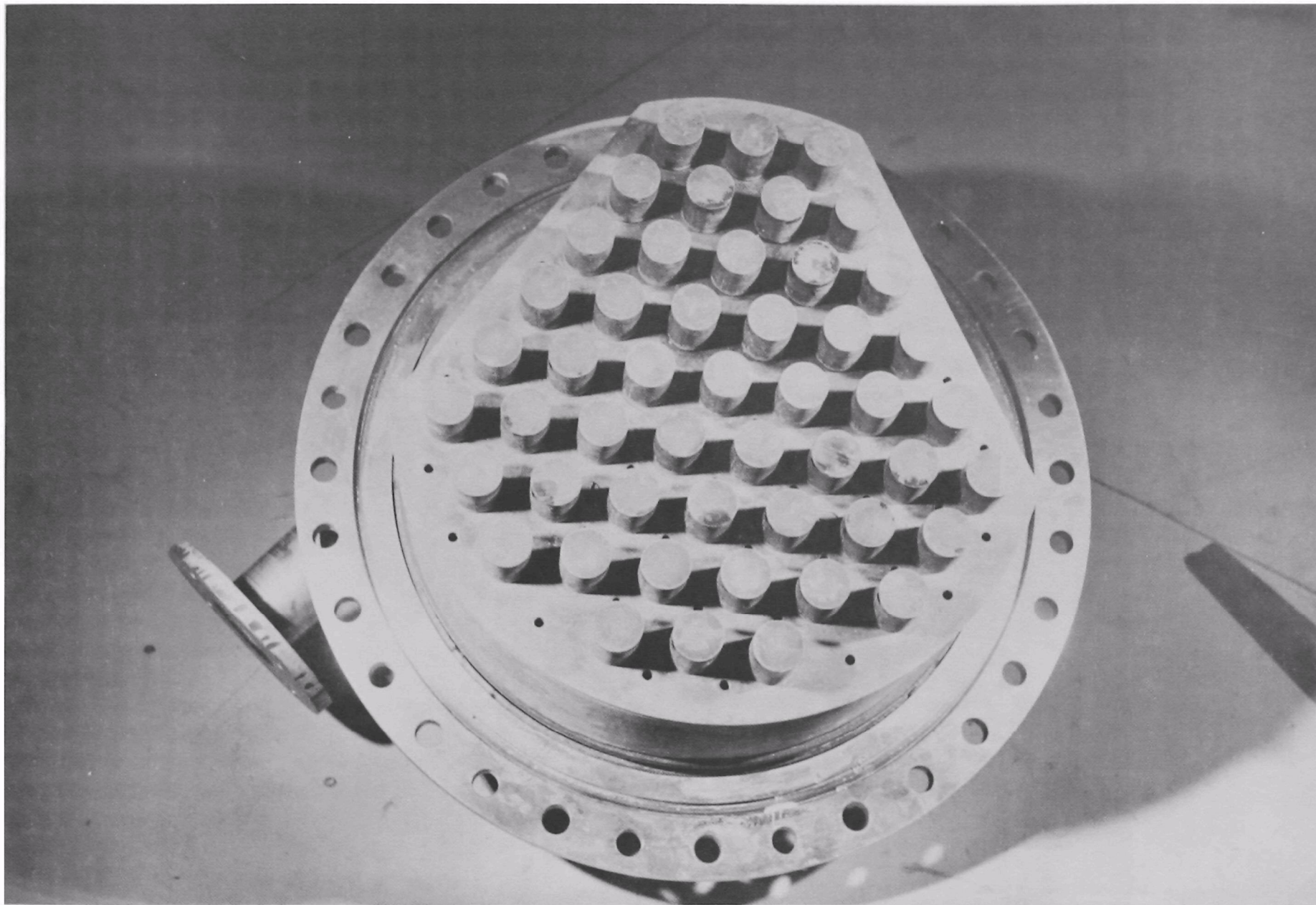


Figure 8



DOWNFLOW EVAPORATOR-CRYSTALLIZER, 5,000 GPD LOW PRESSURE STEAM HEATED

Figure 9



VTFE FEED ORIFICE PLATE

Figure 10



evaporation at selected temperatures and temperature differences ( $\Delta T$ ) in the evaporator tubes. The rate of flow of the distillate was measured during its continuous removal from the system, under evacuated, air-free conditions.

Heating steam, maintained at a preset pressure and temperature, was provided from the reboiler loop of the VTE pilot plant, and distributed to the shell-side of the evaporator-crystallizer through a 6-inch pipe-line with provision for withdrawal under vacuum of line condensates. Steam condensation in the tube bundle was under air-free conditions and with positive flow across the bundle diameter. This was arranged by the wedge-shaped form of the tube bundle and by continuous withdrawal, under vacuum, of a part of the steam flow and noncondensable gases to a condenser. The steam condensed on the shell-side of the tube bundle was continuously removed, under vacuum, by a pump and its rate of flow measured. This condensate flow rate provided the heat flux data ( $Q$ ) needed to measure evaporator performance. Additional data required were the steam temperature ( $T_s$ ) and the brine temperature ( $T_b$ ), measured with platinum resistance probes and converted electronically into degrees Fahrenheit in a digital printout and display. From these data the overall heat transfer performance of the evaporator-crystallizer ( $U$ ) was determined according to the usual formulation:

$$Q = U A \Delta T$$

where  $A$  is the shell-side surface area of the tubes based on their nominal OD (1.5-inch) and length (6-foot), and the  $\Delta T$  is the temperature difference between the steam in the shell-side ( $T_s$ ) and the brine ( $T_b$ ) as measured above.

Makeup feed to the evaporator-crystallizer (EC) was preheated to within a few degrees of the evaporation temperature before introduction into the EC. Residual brine or brine-crystal slurry, was recirculated from the bottom of the EC vessel to the feed distributor and orifice plate with a stainless steel pump at a rate of flow of one gallon per tube per minute. Provision was made for continuous or intermittent blowdown of brine-crystal slurry from the recirculation pump. This slurry discharge was introduced into the bowl of the centrifuge for separation of the crystalline phase.

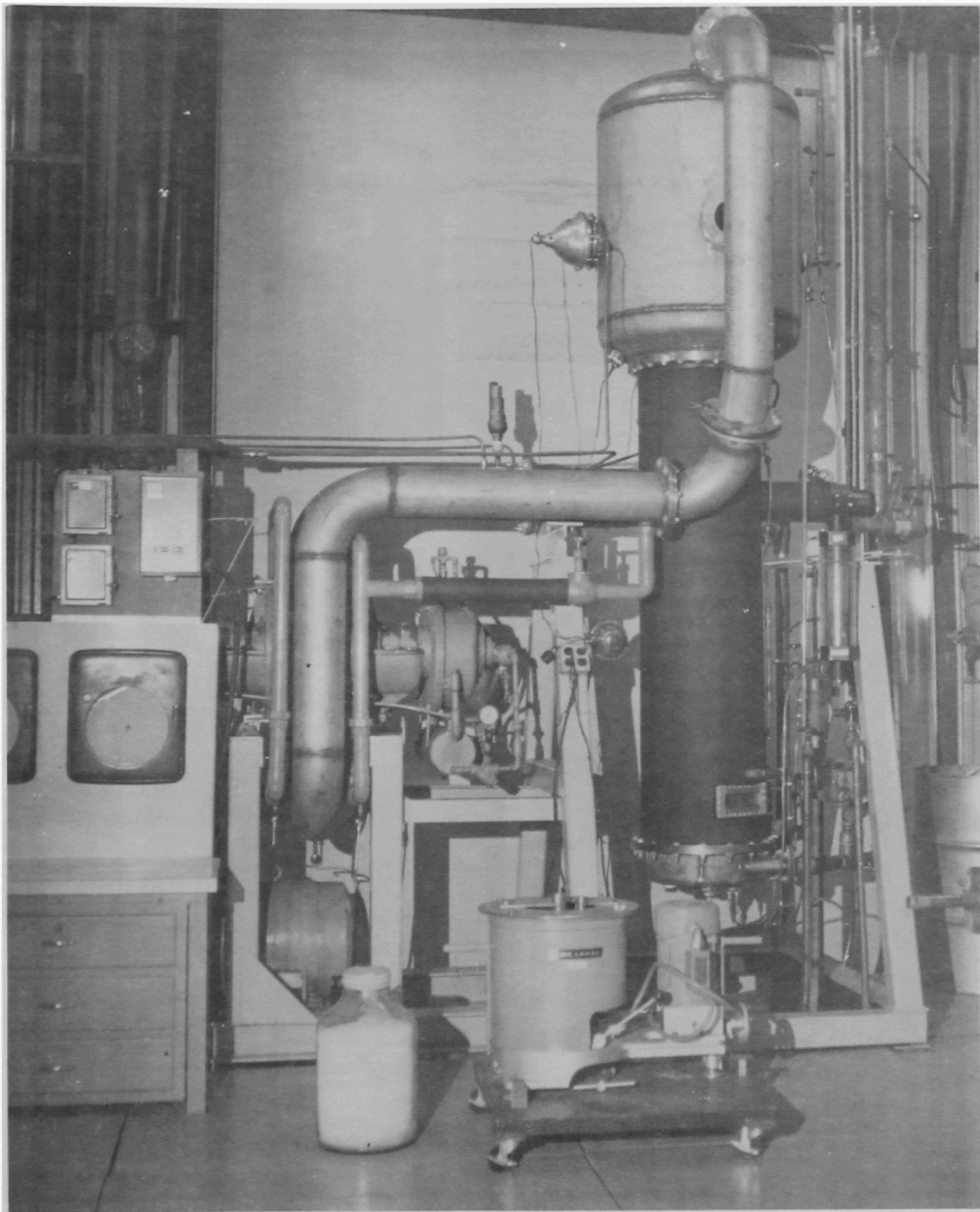
After several preliminary runs with aqueous solutions containing sodium sulphate and calcium sulphate, cooling tower blowdown was examined in this EC, increasing the concentration of the blowdown into a two-phase crystal-brine slurry stream, in recirculated flow at different evaporation temperature conditions.

After operating this EC with reboiler steam as the source of heat, generally at low evaporation temperatures to favor the crystallization of sodium sulphate into a brine slurry, this pilot plant was converted to the vapor compression mode of operation at evaporation temperatures above 210°F.<sup>a</sup> For this purpose a Sutorbilt, Roots type compressor constructed of bronze was acquired having a 920 CFM displacement, capable of

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<sup>a</sup> 98.9°C

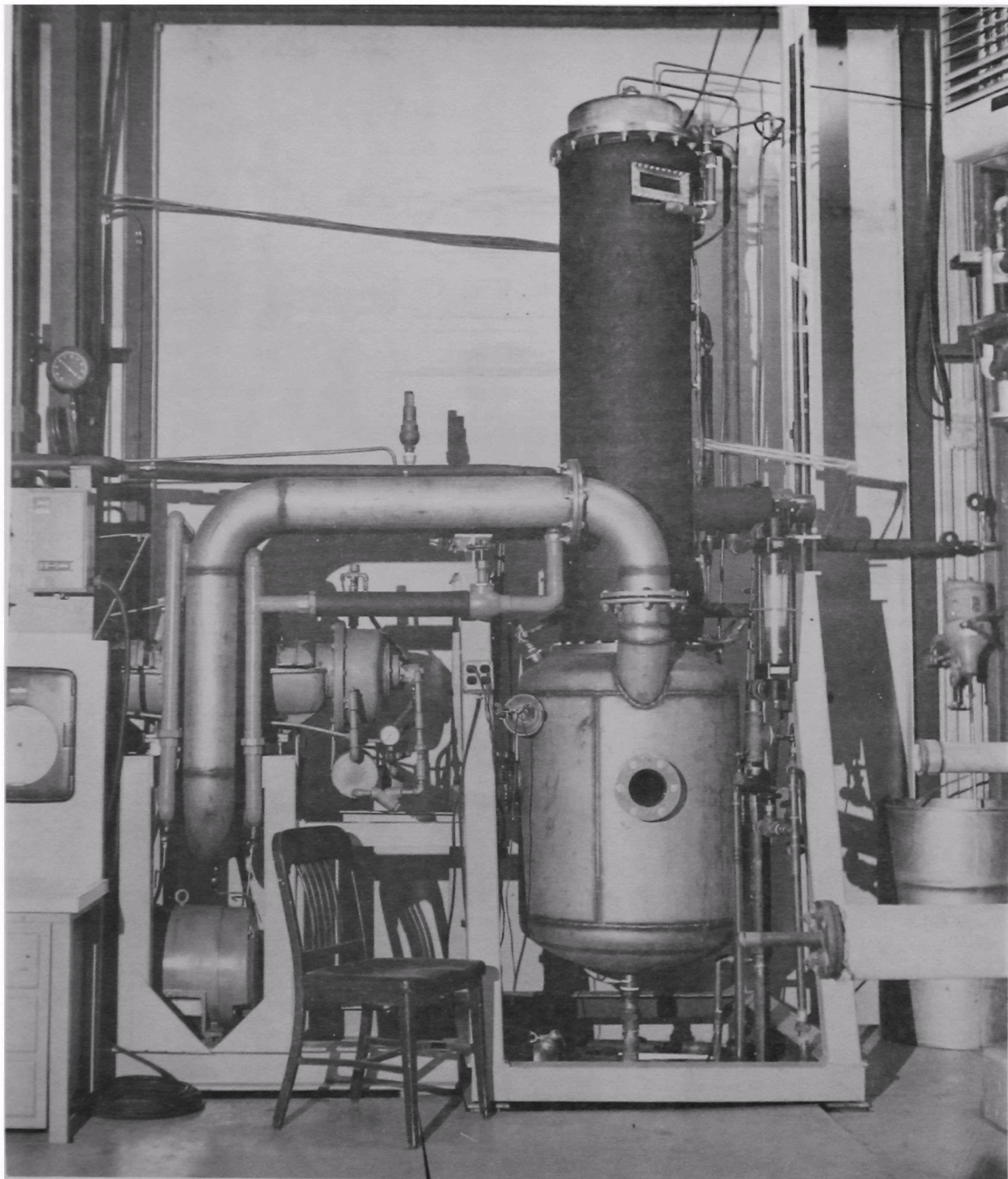
producing about 300 gallons of distillate per hour. A 25 horsepower electric motor was used to drive the compressor at 918 RPM. This provided for about a 5 psi compression of the vapor from the EC vessel; the compressed vapor was then conveyed to the shell-side of the EC through stainless steel pipes of 6-inch diameter. Two bypass lines of 2-inch diameter were provided, for the optional bypassing of some compressed vapor to the suction end of the compressor, to provide an experimental variation of its capacity. This VC-EC is shown in Figure 11, in the upflow mounting with the De Laval basket type centrifuge used for recovering crystalline products from the slurried blowdown in the foreground, and in Figure 12, as used for the downflow concentration and crystallization data on cooling tower blowdown.



UPFLOW VAPOR COMPRESSION EVAPORATOR WITH CENTRIFUGE

Figure 11





DOWNFLOW VAPOR COMPRESSION EVAPORATOR-CRYSTALLIZER

Figure 12



## SECTION IX

### EVAPORATION-CRYSTALLIZATION TESTS

All evaporation-crystallization (EC) tests were carried out with the downflow mode of brine flow. Two methods of heating were used, low pressure reboiler steam for EC of sodium sulphate at about 120°F,<sup>a</sup> and vapor compression heat for EC at about 220°F.<sup>b</sup> These methods were applied to the evaporation of cooling tower blowdown and several other saline waters. Evaporator heat transfer performance data were obtained under conventional EC process conditions and after the addition of a selected surfactant to the feed. This work was divided into three sections:

- (a) Low temperature evaporation with low pressure reboiler steam as the source of heat for evaporation-crystallization,
- (b) Vapor compression evaporation-concentration at elevated temperature,
- (c) Vapor compression evaporation-crystallization at elevated temperature.

#### (a) Low Temperature Evaporation-Crystallization, Downflow

Evaporation-crystallization (EC) at about 120°F<sup>a</sup> was preferred for the separation of sodium sulphate from brine also containing sodium chloride, because of the favorable relative solubilities at low temperature. Several initial experiments were carried out to obtain heat transfer performance data and to gain experience with the pilot plant facility. These runs utilized low pressure reboiler steam, drawn from the VTE pilot plant discussed in Chapter VI, at preset temperatures held at steady state ( $\pm 0.5^\circ\text{F}$ )<sup>c</sup> during these experiments. Condensates from the shell-side of the distillation tubes were continuously removed under vacuum (air free) conditions, their combined rates of flow measured, and returned to the VTE pilot plant.

The heat flux (Q) was calculated directly from these condensate flow measurements in the usual manner. Brine and slurried feeds were recirculated, under vacuum (air free) conditions, for downflow through the tubes at 1 gallon per minute per tube. Vapors produced by evaporation were passed through a stainless steel mesh Demister and conveyed to a water-cooled condenser; these condensates (distillates) were withdrawn under vacuum conditions, their rates of flow were measured and their total dissolved solids (TDS) content determined. Precise temperature measurements were obtained on the steam in the shell-side (Ts) and the brine flowing from the vessel (Tb) to determine the temperature difference ( $\Delta T = T_s - T_b$ ) imposed for evaporation. Steady-state conditions were maintained on Tb and the vapor flow to the condenser by rapidly recirculating the coolant water through the tube-side of the condenser under moderate pressure (25 psi) while continuously adding fresh coolant to that cycle at a sufficient, steady rate

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<sup>a</sup>48.9°C; <sup>b</sup>104.4°C; <sup>c</sup> $\pm 0.3^\circ\text{C}$

with simultaneous reject of hot coolant from this cycle at an equal rate. Measurements were also made on the steam ( $P_s$ ) and vapor ( $P_v$ ) pressures, with an absolute manometer.

Test data are reported in tabular form below. The steam-side surface area of the tube bundle was 115.45 square foot; the condensate and distillate flow rates are given as the time, in seconds, required to collect 16 liters of each; the calculated heat transfer performance ( $U$ ) is reported in Watts per square meter per  $^{\circ}\text{C}$  (and in Btu per  $\text{hr}\cdot\text{ft}^2\cdot^{\circ}\text{F}$ ).

#### (a-1) Evaporation-Crystallization of Sodium Sulphate

Anhydrous sodium sulphate (commercial c.p. grade) dissolved in tap water was recrystallized by EC at  $120^{\circ}\text{F}^a$ , to gain experience with the pilot plant and to determine its heat transfer performance with, and without surfactant additives. Several runs were performed, two of which are reported in Tables I and II. (After these preliminary runs, a batch of cooling tower blowdown obtained from the Mohave power plant in Arizona was examined in the EC.)

Table I shows the change in overall heat transfer coefficient  $U$  during the gradual concentration into the crystallizing regime, of a 27.3%  $\text{Na}_2\text{SO}_4$  aqueous solution at an evaporation temperature of  $121^{\circ}\text{F}^b$ . The steamside temperature was maintained close to  $127.6^{\circ}\text{F}^c$ , and the condenser was maintained at constant heat reject capacity. As a consequence, the temperature difference ( $\Delta T$ ) adjusted in response to the effect of the brine boiling point elevation, due to the gradual increase in salt content followed by a gradual decrease after the start of crystallization; this of course is derived from the fact that the evaporation temperature and the saturation vapor pressure of the brine adjust downward with increased boiling point elevation and vice versa. The difference in absolute pressures of the steamside and the vapor-side also reflect this variation in  $\Delta T$ . Consequently, the overall heat transfer performance, under conditions of constant heat flux, is first gradually reduced until this trend is overcome by rapid crystallization and a downward reversion of the boiling point elevation due to the reduction in total dissolved solids content of the brine (liquid phase). Thus the observed data shown in Table I can be rationalized, excepting the minor fluctuations outside of these general trends, which are due to the usual experimental errors.

Table II reports data obtained under process conditions close to those of Table I, except that 100 ppm of Neodol 25-3A (Shell Chemical Co.) were added after steady-state conditions comparable to the start of the previous run were established. The data that follow this addition of surfactant are quite interesting and revealing of the dispersant properties of the additive. Also of interest was the observation that only a very slight foaminess was induced in the brine flow by the addition of the surfactant, which is consistent with earlier work done in this Laboratory (4); the latter showed induced foaminess to be a prerequisite for interface enhancement rather than merely adding a surfactant or lowering the surface tension. In this case, the addition of surfactant at 100 ppm had no significant effect on the heat transfer performance, consistent with the observed absence of significant foaminess. In fact, when  $U$ -values

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<sup>a</sup>48.9 $^{\circ}\text{C}$ ; <sup>b</sup>49.4 $^{\circ}\text{C}$ ; <sup>c</sup>53.1 $^{\circ}\text{C}$

TABLE I

## CRYSTALLIZING EVAPORATION OF SODIUM SULPHATE

Feed: 56.7 Kg (125 lb) of Anhydrous Sodium Sulphate in 208 Liters (55 Gallons) of Water

Evaporation Temperature (Tb) 50°C (121°F)

Time	Ts		Tb		$\Delta T$		Rejected Distillate		Ps-Pv		Dist. Flow	Cond. Flow	U		Conc. Fact.
	°C	(°F)	°C	(°F)	°C	(°F)	Liter	(Gal.)	CmHg	(InHg)	Secs. /16L	Secs. /16L	W/m <sup>2</sup> -°C	(Btu/hr-ft <sup>2</sup> -°F)	
2.15	53.1	(127.6)	49.9	(121.8)	3.2	(5.8)	0	(0)	26.0	(10.25)	304	296	3708	(653)	1
2.25	53.1	(127.6)	49.9	(121.8)	3.2	(5.8)	0	(0)	26.0	(10.25)	300	296	3708	(653)	1
2.30	53.05	(127.5)	49.85	(121.7)	3.2	(5.8)	0	(0)	26.7	(10.5)	304	300	3657	(644)	1
2.35	53.0	(127.4)	49.65	(121.4)	3.35	(6.0)	18.9	(5)	27.9	(11.0)	306	302	3515	(619)	1.1
2.40	53.2	(127.8)	49.65	(121.4)	3.55	(6.4)	37.85	(10)	28.4	(11.2)	310	305	3259	(574)	1.2
2.47	53.2	(127.8)	49.4	(120.9)	3.8	(6.9)	56.8	(15)	23.0	(13.0)	316	316	2918	(514)	1.4
2.55	53.0	(127.4)	49.25	(120.6)	3.75	(6.8)	75.7	(20)	33.5	(13.2)	320	314	2981	(525)	1.8

## START OF CRYSTALLIZATION

2.59	53.2	(127.8)	49.05	(120.3)	4.15	(7.5)	94.6	(25)	38.1	(15.0)	370	322	3043	(536)	1.8
3.07	53.1	(127.6)	48.95	(120.1)	4.15	(7.5)	113.6	(30)	36.8	(14.5)	330	324	2618	(461)	2.2
3.12	53.1	(127.6)	49.1	(120.4)	4.0	(7.2)	132.5	(35)	36.8	(14.5)	326	325	2725	(480)	2.8
3.25	53.2	(127.8)	49.35	(120.8)	3.85	(7.0)	132.5	(35)	36.8	(14.5)	329	325	2799	(493)	2.8
3.35	53.2	(127.8)	49.35	(120.8)	3.85	(7.0)	132.5	(35)	36.1	(14.2)	329	328	2742	(483)	2.8
3.55	53.2	(127.8)	49.35	(120.8)	3.85	(7.0)	132.5	(35)	36.2	(14.25)	328	325	2799	(493)	2.8

TABLE II

## CRYSTALLIZING EVAPORATION OF SODIUM SULPHATE

Feed: 56.7 Kg (125 lb) of Anhydrous Sodium Sulphate in 208 Liters (55 Gallons) of Water

Evaporation Temperature (Tb) 50°C (120°F)

Effect of Surfactant Addition on Crystallization of Sodium Sulphate

36

Time pm	Ts		Tb		$\Delta T$		Rejected Distillate		Ps-Pv		Dist. Flow	Cond. Flow	U		Conc. Fact.
	°C	(°F)	°C	(°F)	°C	(°F)	Liter (Gal.)		CmHg (InHg)		Secs. /16L	Secs. /16L	W/m <sup>2</sup> -°C	(Btu/hr-ft <sup>2</sup> -°F)	
10.30	52.7	(126.9)	49.6	(121.3)	3.1	(5.6)	(0)		23.75 (9.5)		302	296	3838	(676)	1
10.42	52.7	(126.9)	49.55	(121.2)	3.15	(5.7)	(0)		23.75 (9.5)		300	292	3821	(673)	1
100 ppm NEODOL 25-3A ADDED (40 ml); VERY SLIGHT FOAMINESS OBSERVED															
11.15	52.55	(126.6)	49.35	(120.8)	3.2	(5.7)	(0)		25.5 (10.2)		300	298	3747	(660)	1
11.25	52.5	(126.5)	49.2	(120.6)	3.3	(5.9)	18.9 (5)		26.3 (10.5)		300	297	3634	(640)	1.1
11.30	52.55	(126.6)	49.1	(120.4)	3.45	(6.2)	37.85 (10)		27.3 (10.9)		304	297	3458	(609)	1.2
11.35	52.55	(126.6)	49.0	(120.2)	3.55	(6.4)	56.8 (15)		28.8 (11.5)		310	304	3276	(577)	1.4
11.43	52.6	(126.7)	48.6	(119.5)	4.0	(7.2)	75.7 (20)		33.8 (13.5)		315	312	2839	(500)	1.6
11.50	52.65	(126.8)	48.5	(119.3)	4.15	(7.5)	94.6 (25)		36.8 (14.7)		324	318	2674	(471)	1.8
START OF CRYSTALLIZATION															
11.57	52.6	(126.7)	48.45	(119.2)	4.15	(7.5)	113.6 (30)		40.0 (16.0)		332	328	2589	(456)	2.2
12.05	52.5	(126.5)	48.0	(118.3)	4.5	(8.2)	132.5 (35)		40.0 (16.0)		326	320	2430	(428)	2.8
12.15	52.5	(126.5)	47.6	(117.5)	4.9	(9.0)	141.9 (37.5)		40.6 (16.25)		-	324	2192	(386)	3.1
12.25	52.3	(126.2)	48.2	(118.7)	4.1	(7.5)	141.9 (37.5)		38.8 (15.5)		-	317	2527	(445)	3.1
12.35	52.6	(126.7)	48.5	(119.3)	4.1	(7.4)	141.9 (37.5)		38.8 (15.5)		-	318	2549	(449)	3.1

are compared with the previous run at equal brine concentrations, it is apparent that the additive had a negative effect on heat transfer performance from the point where crystallization ensued for the first run. This is rationalized as being the consequence of a retardation effect of the additive on crystallization; such a threshold-inhibition of crystallization was also observed in earlier work in this Laboratory (5). Retarding or inhibiting crystallization is clearly indicated by the data of Table II; crystallization was first observed at a concentration factor of 1.57 for the first and 1.83 for the second run. Also, recovery of heat transfer performance due to the reduction of the boiling point elevation of the brine, by rapid crystallization of sodium sulphate from the solution, was clearly retarded in the second run by comparison with the first run. (See Tables I and II.)

In summary, the above data indicate that the addition of a surfactant during crystallizing evaporation of highly concentrated salt solutions (27.3% and higher) provides no significant heat transfer advantage and retards the onset and rate of crystallization. The latter in turn reduces the evaporator heat transfer performance by the increase in boiling point elevation due to the increased dissolved salt content (supersaturation) of the brine in recirculation through the evaporator.

#### (a-2) Cooling Tower Blowdown Evaporation-Crystallization

Table III reports heat transfer performance data obtained during the crystallization-evaporation of 6.4-fold preconcentrated cooling tower blowdown obtained from the Mohave power plant. Preconcentration of blowdown having a TDS of 10,000 ppm by a factor of 10 was performed by upflow VTFE (5) at low temperature (130°F)<sup>a</sup> after the addition of 10 ppm of Neodol 25-3A. This 10-fold preconcentrated blowdown (45 gallons) was reconstituted to a 6.43-fold concentrate by dilution to 70 gallons with distilled water; this dilution was done to facilitate the quantitative transfer of the 10-fold concentrate, having considerable crystallized solids in suspension, into the evaporator-crystallizer (EC). This suspension was recirculated through the evaporator under evacuation for about one hour to remove non-condensibles (deaeration) and to bring the solids in equilibrium with the brine. The temperature of the recirculated slurry was adjusted to 120°F<sup>b</sup> and recirculation under vacuum was continued for another hour to equilibrate suspended solids with the liquid phase (brine). Evaporation-crystallization was then performed as reported in Table III. In this operation the steam-side temperature was held steady at about 125.5°F<sup>c</sup> and the coolant flow into the condenser loop was adjusted to provide a  $\Delta T$  of about 5.5°F.<sup>d</sup> The EC feed was recirculated at close to 1 gallon per minute per tube; concentration of the feed was completed in 1 hour and 35 minutes, evaporating it from a 6.43-fold to a 30-fold concentrate, 3.33% of its original volume. The distillate obtained, representing 96.66% of the original cooling tower blowdown, had a total dissolved solids content of 19 ppm. Figure 13 shows a sample of the final brine slurry obtained from the Mohave cooling tower after 30-fold concentration.

The evaporator heat transfer performance during crystallization fluctuated somewhat more than usual, generally between 750 and 670 Btu per hr-ft<sup>2</sup>-°F, but did not decrease significantly during this final

TABLE III

## CRYSTALLIZING EVAPORATION OF COOLING TOWER BLOWDOWN

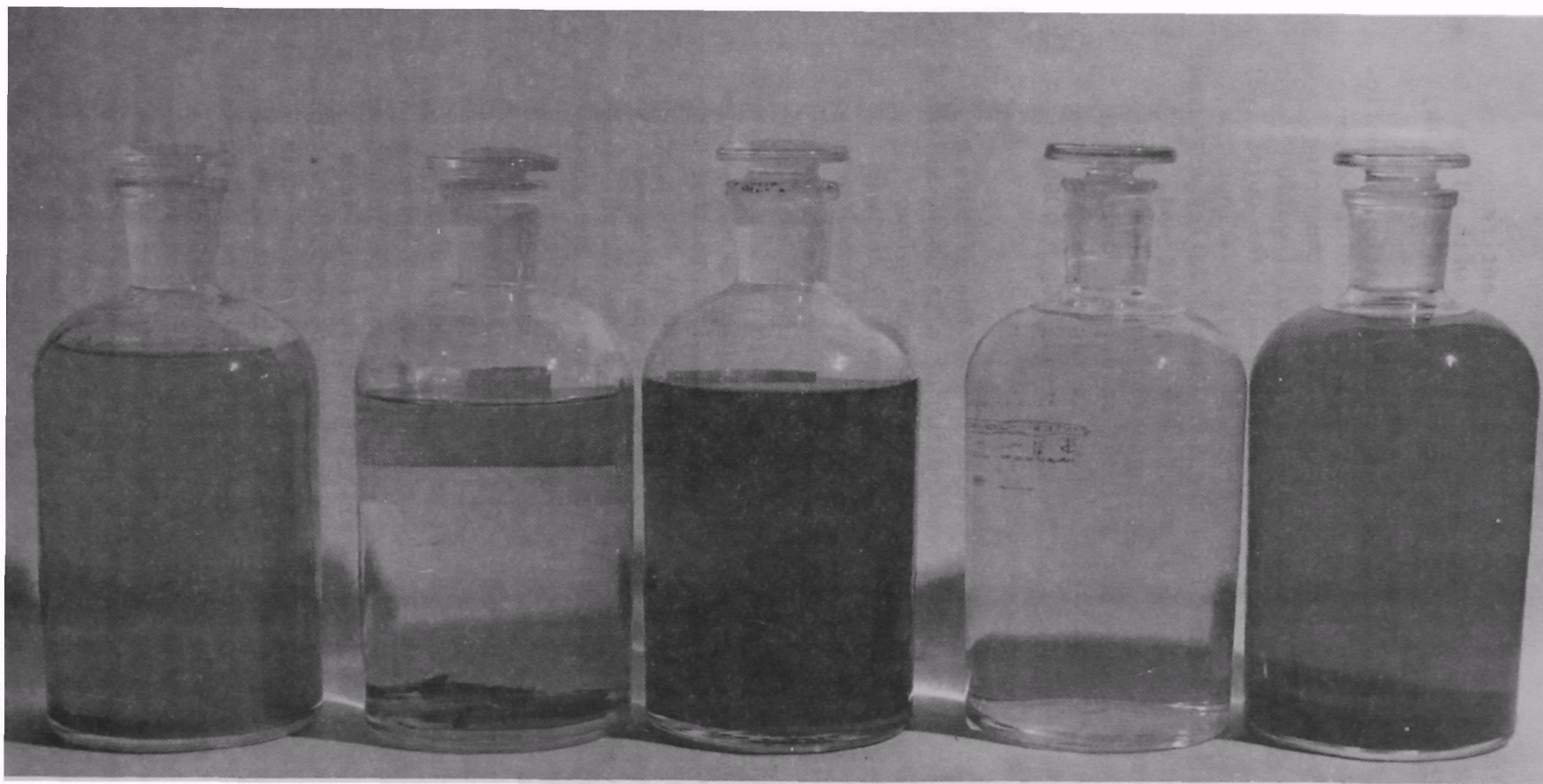
Feed: Cooling Tower Blowdown from Mohave Power Plant Having 10 ppm of Surfactant Added and Concentrated 6-fold by VTFE, 265 Liters (70 Gal.).

Evaporation Temperature 50°C (120°F)

Time pm	Ts		Tb		$\Delta T$		Rejected Distil.	Ps-Pv		Dist. Flow	Dist. Flow	U		Conc. Fact.
	°C	(°F)	°C	(°F)	°C	(°F)	Liter (Gal.)	CmHg	(InHg)	Secs. /16L	Secs. /16L	W/m <sup>2</sup> -°C	(Btu/hr-ft <sup>2</sup> -°F)	
3.00	52.1	(125.8)	49.35	(120.8)	2.75	(5.0)	0 (0)	15.2	(6.0)	287	288	4423	(779)	6.43
3.30	51.9	(125.3)	48.9	(120.0)	3.0	(5.3)	37.9 (10)	16.1	(6.35)	290	304	3952	(696)	7.50
3.40	52.0	(125.6)	49.0	(120.2)	3.0	(5.4)	56.8 (15)	16.5	(6.5)	291	306	3855	(679)	8.18
3.45	51.9	(125.3)	48.9	(120.0)	3.0	(5.3)	75.7 (20)	19.1	(7.5)	296	318	3787	(667)	9.0
3.52	51.5	(124.7)	48.4	(119.1)	3.1	(5.6)	94.6 (25)	17.8	(7.0)	294	290	3929	(692)	10.0
3.56	51.65	(125.0)	48.15	(119.6)	3.0	(5.4)	134 (30)	17.8	(7.0)	294	290	4077	(718)	11.26
4.05	52.15	(125.9)	49.15	(120.5)	3.0	(5.4)	132 (35)	17.8	(7.0)	290	288	4088	(720)	12.85
4.08	52.05	(125.7)	49.1	(120.4)	2.95	(5.3)	151 (40)	18.3	(7.2)	291	282	4259	(750)	15.00
4.15	52.05	(125.7)	49.05	(120.3)	3.0	(5.4)	170 (45)	18.4	(7.25)	294	294	4014	(707)	18.00
4.22	51.8	(125.2)	48.7	(119.7)	3.1	(5.5)	189 (50)	19.1	(7.5)	298	288	4037	(711)	22.5
4.30	51.7	(125.0)	48.55	(119.4)	3.15	(5.6)	199 (52.5)	19.6	(7.7)	300	301	3782	(666)	25.72
4.35	51.95	(125.5)	48.85	(119.9)	3.1	(5.6)	208 (55)	19.8	(7.8)	feed	302	3765	(663)	30.01
4.40	51.95	(125.5)	48.9	(120.0)	3.05	(5.5)	208 (55)	19.8	(7.8)	too low	292	3970	(699)	30.01

BRINE RESIDUE COLLECTED: 14 gall

DISTILLATE QUALITY: 19 ppm TDS



CONCENTRATED  
MOHAVE POWER PLANT  
COOLING TOWER  
BLOWDOWN  
30-FOLD CONCENTRATE

BLOWDOWN FROM  
PETROLEUM REFINERY  
COOLING TOWER

CONCENTRATED  
PETROLEUM REFINERY  
COOLING TOWER  
BLOWDOWN  
50-FOLD CONCENTRATE

BLOWDOWN FROM  
CHEMICAL INDUSTRY  
COOLING TOWER

CONCENTRATED  
CHEMICAL INDUSTRY  
COOLING TOWER  
BLOWDOWN  
27-FOLD CONCENTRATE

SAMPLES OF COOLING TOWER BLOWDOWN AND THEIR CONCENTRATES

Figure 13

concentration stage. The heat transfer performance for the entire concentration operation, including preconcentration by VTFE, with 10 ppm of surfactant added to the original Mohave blowdown is shown in Figure 14.

(b) High Temperature, Vapor Compression Evaporation-Concentration, Downflow

Vapor compression evaporation was carried out at brine-side temperatures of 212-224°F<sup>a</sup> with and without surfactant addition to the brine. The downflow mode was used, with brine recirculation at about 1 gallon per tube per minute. This series of runs were performed with the objective of gaining experience with VC-VTE operation. Crystallization was not the objective here; agricultural wastewater from the San Joaquin Valley was used. This irrigation drainage water had a TDS of about 7000 ppm but was presoftened by cation exchange of calcium and magnesium for sodium ions. It had a pH of about 8 and was used as such without acidification. This type of wastewater has a typical composition, after softening, as follows in parts per million: Na = 2200; K = 2; Ca = 12; Mg = 10; SO<sub>4</sub> = 4000; Cl = 500; HCO<sub>3</sub> = 300; NO<sub>3</sub> = 50.

In this test series the objective was to determine the effects of surfactant addition to the downflow VTE concentration of a strongly saline wastewater by a factor of 20. Since the maximum and minimum holdup volumes of the evaporator was 55 and 15 gallons, respectively, it was necessary to add the feed batchwise, after consecutive concentration steps. The test data are shown in Table IV.

The first line of Table IV reports the steady-state process conditions applied and the VC-VTE heat transfer performance observed with ion exchange softened agricultural wastewater having a typical wintertime high TDS of 7000, without any additions to it. The next line shows the comparative VC-VTFE performance after the addition of 10 ppm of Neodol 25-3A (Shell Chemical Co.) to the same feed. The  $\Delta T$  adjusted downward, while the distillation rate increased; simultaneously (and consistently) the vapor compressor ran noticeably smoother under the reduced differential pressure ( $\Delta T$ ) (Power consumption by the 25 horsepower VC motor could not be measured for these tests.) A 31 percent increase in performance was obtained with 10 ppm of surfactant. The third line of data shows the comparative overall heat transfer coefficient after the addition of a further 5 ppm of surfactant. The heat transfer performance was enhanced to 2188 Btu per hr-ft<sup>2</sup>-°F, 37.4 percent above the conventional performance of line 1. The distillates were recombined with the recirculated feed for each of these above sets of data, maintaining the concentration factor of the brine at 1, or 7000 TDS. The fourth through tenth lines of data summarize the procedure of batchwise additions of softened wastewater to the recirculated feed and the batchwise rejections of distillates, to obtain a stepwise increase in the concentration factor of the recirculated feed. The eleventh line reports the VC-VTFE process conditions applied and the heat transfer performance obtained on 20-fold concentrated wastewater having a TDS of 140,000 ppm and about 15 ppm of surfactant. The overall heat transfer coefficient at these concentrations was 33 percent above the initial performance with unconcentrated wastewater. These data are plotted in Figure 15.

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<sup>a</sup> 100°C-106.7°C



CONCENTRATION OF MOHAVE POWER PLANT COOLING TOWER BLOWDOWN  
BY INTERFACE-ENHANCED VERTICAL TUBE EVAPORATION AT 130°F

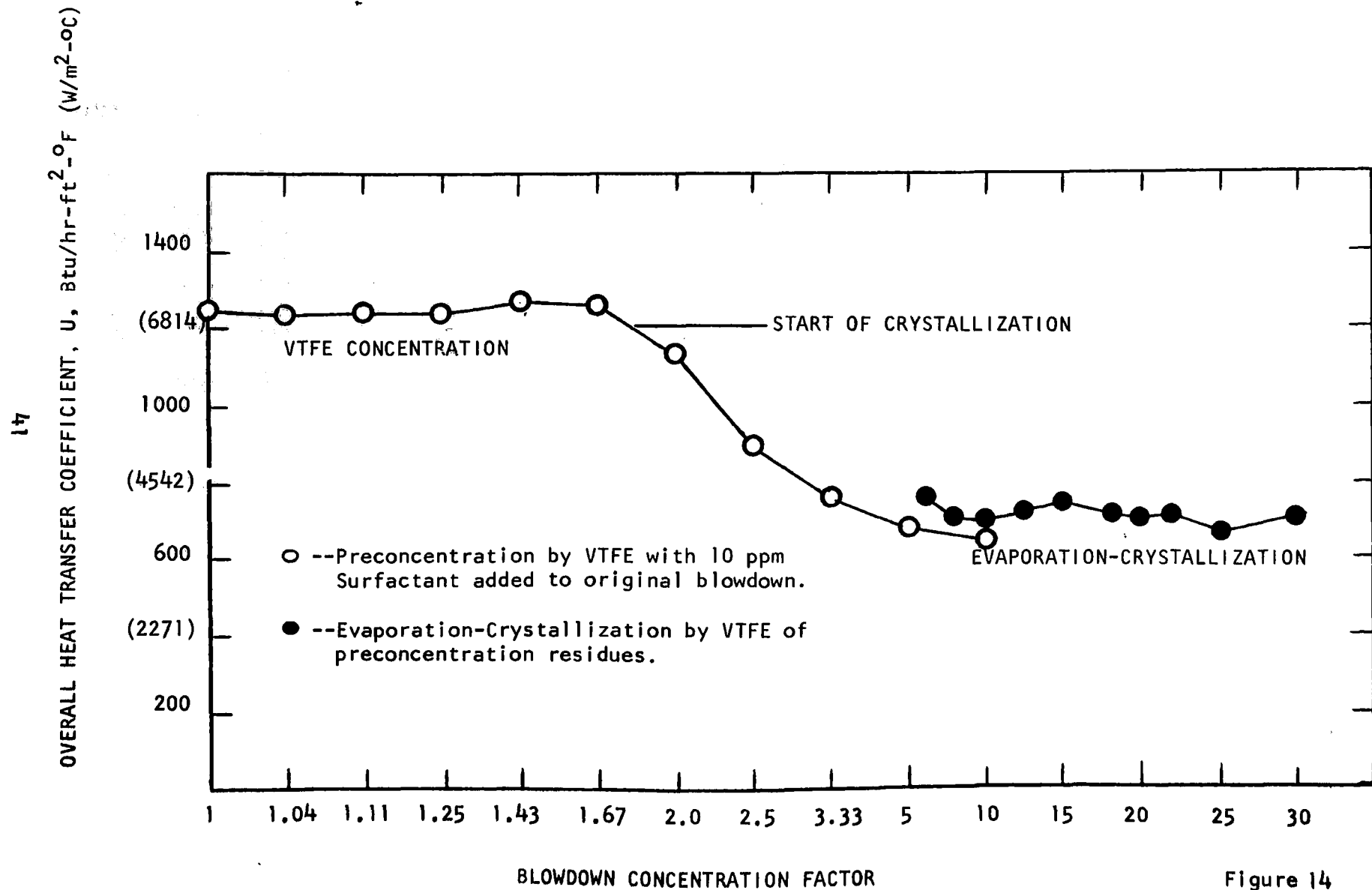


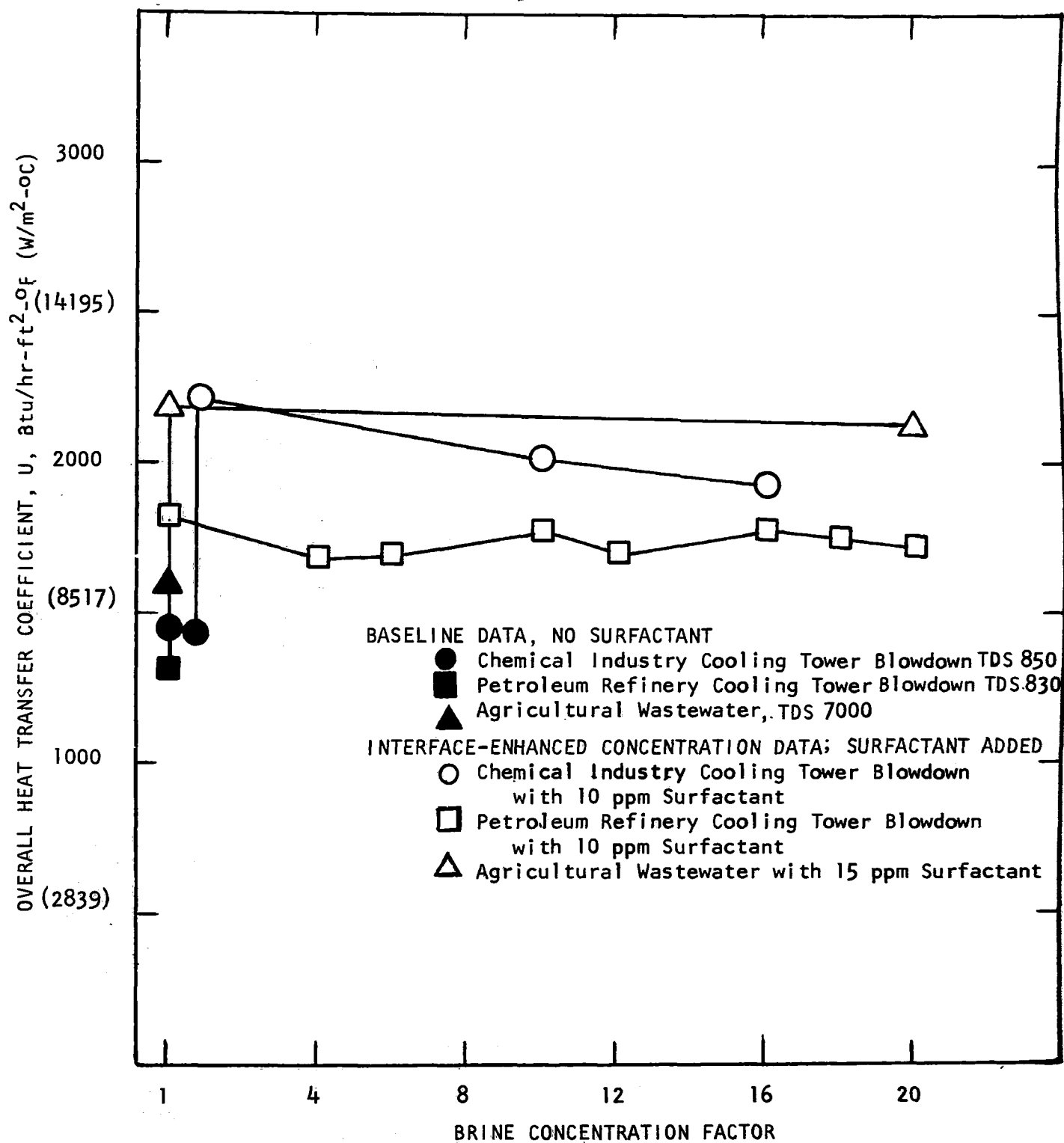
Figure 14

TABLE IV

VAPOR COMPRESSION EVAPORATION CONCENTRATION

Feed: Agricultural Wastewater, Softened by Ion Exchange, TDS of 7000 ppm

Additions to feed Liter (Gal.)	Ts		Tb		$\Delta T$		Rejected Distil. Liter (Gal.)	Dist. Flow Secs. /16L	U		Conc. Fact.
	°C	(°F)	°C	(°F)	°C	(°F)			W/m <sup>2</sup> -°C (Btu/hr-ft <sup>2</sup> -°F)		
189 (50)	109.3	(228.7)	104	(219.3)	5.3	(9.4)	0 (0)	68	9045	(1593)	1
Neodol added to Feed, to 10 ppm.											
0 (0)	110.5	(230.8)	105.8	(222.4)	4.7	(8.4)	0 (0)	58	11,838	(2085)	1
Neodol added to Feed, to 15 ppm.											
0 (0)	111.5	(232.5)	106.6	(223.9)	4.9	(8.6)	0 (0)	54	12,423	(2188)	1
0 (0)	-	-	-	-	-	-	95 (25)	-	-	-	2
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	4
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	6
190 (50)	-	-	-	-	-	-	189 (50)	-	-	-	8
189 (50)	-	-	-	-	-	-	190 (50)	-	-	-	10
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	12
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	14
190 (50)	108.2	(226.8)	103.8	(218.8)	4.4	(8.0)	208 (55)	60	12,054	(2123)	20
189 (0)							1438 (380)				
1514 (400)	No crystallization occurred during this 20-fold concentration.										



EVAPORATION-CRYSTALLIZATION OF WASTEWATER BRINES  
HEAT TRANSFER ENHANCEMENT BY ADDITION OF SURFACTANT

(c) Vapor Compression Evaporation-Crystallization of Cooling Tower  
Blowdown at 215-220°F<sup>a</sup>

Two different industrial cooling tower blowdowns were examined by downflow VC-VTFE in this section. The first of these, reported in Table V, was obtained from a chemical manufacturing complex. The second came from a large petroleum refinery and is reported in Table VI. The evaporation-crystallization temperatures, and the vapor compression mode of operation were chosen to represent realistic present wastewater renovation practice (8).

The chemical industry cooling tower blowdown had a TDS of 850 and a pH of 7.95; it was used as received, without pH adjustment. Evaporation-concentration was done on a batch-wise-continuous basis as in the previous case, with surfactant addition to 10 ppm in the residual, recirculated feed. A 27-fold concentration level was obtained, with some calcium carbonate being precipitated into the recirculated feed towards the end of the run.

The first two lines of data in Table V report the VC-VTE process conditions used, with a heat transfer performance ( $U$ ) of 8233 W/m<sup>2</sup>-°C (1450 Btu/hr-ft<sup>2</sup>-°F) on the wastewater as received. The second line shows a slight decline in performance at a wastewater concentration factor of 1.33. Distilled water produced at this condition had a TDS of 5 ppm. Line 3 shows the shift in process conditions resulting from the addition of 10 ppm of Neodol 25-3A (Shell Chemical Co.). The steam-side temperature ( $T_s$ ) and pressure dropped, the brine temperature ( $T_b$ ) increased adjusting the  $\Delta T$  downward, and the evaporation or distillation rate increased. This combination of interface enhancement effects provided a 53 percent increase (to 12,548 W/m<sup>2</sup>-°C) in the VC-VTFE performance over the conventional VC-VTE conditions. In addition to increasing the distillate productivity rate, the vapor compressor ran noticeably smoother because of the reduction in  $\Delta T$  and pressure after surfactant addition to the feed; this advantage should be reflected in a reduced electric power consumption by the 25 horsepower motor; it could however not be measured because of a lack of the necessary equipment (watt-hour meter). A further minor advantage of the induced foamy vapor-liquid flow through the tubes was that the vapor and distillate produced had less entrained brine droplets; the distillate quality was improved to a range of 1.5 to 2.5 ppm of TDS compared to 5 ppm before surfactant addition. This simultaneously provides a measure of the surfactant transfer (in brine droplets) into the distillate, at about 18 parts per billion.

A 27-fold concentration of this wastewater was readily obtained with VC-VTFE, with a heat transfer performance enhancement ranging from 53 to 34 percent through the 16-fold concentration range, renovating 96 percent of this wastewater into pure distilled water, and reducing the wastewater to a slurry of 4 percent of its original volume. See Figure 15 for a plot of these performance data.

Another batch of cooling tower blowdown examined was obtained from a large petroleum refinery; it had a TDS content of 380 ppm, a pH of 6.8

TABLE V

## VAPOR COMPRESSION EVAPORATION-CRYSTALLIZATION

Feed: Cooling Tower Blowdown from Chemical-Industry: TDS-850 ppm, pH-7.75.

Addition to Feed Liter (Gal.)	Ts		Tb		$\Delta T$		Rejected Distillate Liter (Gal.)	Dist. Flow Secs. /16L	U		Conc. Fact.
	°C	(°F)	°C	(°F)	°C	(°F)			W/m <sup>2</sup> -°C (Btu/hr-ft <sup>2</sup> -°F)		
189 (50)	107.8	(226.0)	102	(215.5)	5.8	(10.5)	0 (0)	66	8233	(1450)	1
189 (50)	108.9	(228.0)	103.3	(217.9)	5.6	(10.1)	95 (25)	70	8193	(1443)	1.33

Neodol added to 10 ppm

0 (0)	108.6	(227.5)	103.8	(218.8)	4.8	(8.7)	0 (0)	53	12,548	(2210)	1.33
0 (0)	-	-	-	-	-	-	189 (50)	-	-	-	4
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	6
190 (50)	-	-	-	-	-	-	189 (50)	-	-	-	8
189 (50)	107.5	(225.5)	102.9	(217.2)	4.6	(8.3)	190 (50)	61	11,458	(2018)	10
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	12
189 (50)	-	-	-	-	-	-	189 (50)	-	-	-	14
190 (50)	107.1	(224.8)	102.7	(216.8)	4.4	(8.0)	189 (50)	66	10,987	(1935)	16
0 (0)	-	-	-	-	-	-	38 (10)	-	-	-	26.7
1514 (400)							1457 (385)				

Distillate quality varied between 1.5 and 5 ppm TDS.

These data were plotted in Figure 15.

Blowdown precipitated off-white layer shown in Figure 13.

TABLE VI

## VAPOR COMPRESSION EVAPORATION-CRYSTALLIZATION

Feed: Cooling Tower Blowdown for Petroleum Refinery; TDS-380 ppm, pH-6.8, chromate-14 ppm.

Additions to Feed		Ts		Tb		ΔT		Rejected Distillate		Dist. Flow		U		Conc. Fact.
Liter (Gal.)		°C	(°F)	°C	(°F)	°C	(°F)	Liter (Gal.)		Secs. /16L		W/m <sup>2</sup> -°C (Btu/hr-ft <sup>2</sup> -°F)		
189	(50)	108.6	(227.5)	102.5	(216.5)	6.1	(11.0)	0	(0)	70		7540	(1328)	1
Neodol was added to 10 ppm.														
0	(0)	107.0	(224.5)	102.3	(216.1)	4.7	(8.4)	95	(25)	67		10,323	(1818)	1
189	(50)	109.2	(228.5)	104.0	(219.1)	5.2	(9.4)	189	(50)	62		9937	(1750)	4
189	(50)	107.9	(226.0)	102.6	(216.6)	5.3	(9.4)	189	(50)	64		9641	(1698)	6
190	(50)	-	-	-	-	-	-	189	(50)	-		-	-	8
189	(50)	109.5	(229.0)	104.5	(220.1)	5.0	(8.9)	190	(50)	64		1947	(1787)	10
189	(50)	107.6	(225.7)	102.6	(216.6)	5.0	(9.1)	189	(50)	66		9619	(1694)	12
189	(50)	-	-	-	-	-	-	189	(50)	-		-	-	14
190	(50)	107.4	(225.3)	102.4	(216.3)	6.0	(9.0)	189	(50)	64		10,084	(1776)	16
189	(50)	106.1	(223.0)	101.3	(214.4)	4.8	(8.6)	190	(50)	68		9948	(1752)	18
189	(50)	108.1	(226.5)	103.1	(217.5)	5.0	(9.0)	189	(50)	66		9778	(1722)	20
	(0)	-	-	-	-	-	-	57	(15)	-		-	-	50
1892	(500)							1855	(490)					

Distillate Quality -- 1 to 2.5 ppm TDS  
Blowdown is shown in Figure 13.

Data could not be managed for the terminal concentration steps  
because of time constraints.

and contained 15 ppm of chromate. This wastewater was renovated by down-flow VC-VTFE, similar to the previous case reported; test data are reported in Table VI and plotted in Figure 15.

Initial, baseline performance data are shown in the first line of Table VI, using the wastewater (as received) as feed and recycling the distillate into the feed. Neodol 25-3A was then added to 10 ppm, and the interface-enhanced data of the second line were obtained. This provided a heat transfer enhancement of 37 percent; less foaminess was observed than in the previous run in which an enhancement of 53 percent was obtained with 10 ppm of Neodol addition. This reduced foaminess may have been caused by the presence of 15 ppm of chromate in the wastewater used in this run. This reduced level of interface enhancement was observed through the entire concentration range. An increased level of surfactant additive should overcome this adverse effect. Further stepwise concentration of the feed, added batchwise as before, is reported in Table VI showing that the heat transfer enhancement remained at about 33 percent through the remainder of the run, somewhat below the previous run but consistently above the baseline performance. These data are plotted in Figure 15, on a comparative basis.

Final concentration of this wastewater by a factor of 50 was readily obtained, producing 98 percent of high quality distilled water (TDS ranging from 1 to 2.5 ppm) and reducing the wastewater to a slurry only 2 percent of its original volume. The final concentration from 20- to 50-fold was obtained in a very short time (4 minutes), by the rejection of the final 15 gallons of distillate, and heat transfer data collection could not be managed for this terminal step, because of time constraints and the shutdown procedure required.

Figure 13 shows samples of wastewater feeds and corresponding concentrates obtained during evaporation-crystallization with VTFE in this work.

## SECTION X

### DISCUSSION OF RESULTS

The objective of this work was to obtain sufficient vertical tube evaporation (VTE) heat transfer data on the preconcentration, followed by the evaporation-crystallization of industrial cooling tower blowdown, to provide a basis for the design of a mobile pilot plant facility for field testing. This pilot plant would also provide definitive data on the merit of the interface-enhanced mode of vertical tube foam evaporation (VTFE) for the complete renovation of power plant cooling tower blowdown, and for using waste heat available within the usual power plant cycle to accomplish this evaporation at low temperature (120°F)<sup>a</sup>. For this objective, we needed to construct a pilot plant to provide both comparative upflow and downflow data with VTE and VTFE used for the preconcentration of cooling tower blowdown; we also had to provide a separate pilot plant for comparative downflow VTE and VTFE evaporation-crystallization data with the preconcentrated blowdown. This second pilot plant needed to be operated both at low evaporation temperatures, and under vapor compression conditions at elevated temperature. Much of the time and effort under this study was therefore devoted to process and hardware design, procurement of equipment, and construction of these facilities.

A two-effect, upflow-downflow VTE pilot plant of 10,000 gallons per day (gpd) capacity, shown in Figures 2 and 3, was constructed, and comparative test data were obtained to ascertain the heat transfer performance with brine of 35,000 ppm TDS (seawater). These data, when compared with earlier concentration data obtained under EPA sponsorship (5) with cooling tower blowdown from the Mohave power plant using the single-effect upflow VTE pilot plant section of the present two-effect pilot plant, provided information required under the objectives of the present study. Those earlier data (10) are in part reproduced in Figure 14, and the additional comparative upflow-downflow data of this study are reported in Figures 6 and 7 of this report.

It is apparent from Figure 6 that 20 ppm of Neodol 25-3A provides about the optimal foaming agent concentration for downflow VTFE enhancement, while 10 ppm should be adequate for upflow VTFE. This applies for both aluminum-brass and titanium tubes of the most advanced, double-fluted types. It is clear that the use of interface enhancement is well justified for both upflow and downflow VTE; the cost of the surfactant additive used would amount to about 22 cents per pound for a 60 percent solution supplied in bulk deliveries. A significant reduction of this relatively low cost should result from the use of foam fractionation for recovery and recycle of the additive (6). The tube-side pressure drop data

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<sup>a</sup>48.90C



plotted in Figure 6 show that, in the case of upflow VTFE, a significant further advantage is gained by this foamy mode of flow. The approximately 10-fold reduction in tube-side upflow pressure drop improves the upflow hydrodynamic stability substantially, thereby permitting an increase in the number of effects acceptable for a multieffect VTFE plant. Alternatively, in the case of a vapor compression (VC) VTE, this pressure drop reduction reduces the load on the compressor, which should reduce its power consumption. Also significant, is the possibility of driving a second effect VTFE with the same vapor compressor that would normally manage only one effect VTE. This could provide a substantial increase in the usual gain ratio, to reduce power cost by about half, for upflow VTE.

Figure 7 shows the conventional VTE and VTFE performance for upflow and downflow operation with the two different types of enhanced tubes, on a comparative basis. Also shown are the upflow VTE tube-side pressure drop profiles confirming that, for 2-inch diameter distillation tubes, one can increase the number of effects in the temperature range above 160°F.<sup>a</sup> Also apparent, is the advantage of 3-inch diameter tubes over 2-inch tubes in the evaporation range below 160°F<sup>a</sup> on a basis of their comparative VTFE performances. On this basis, it should be advantageous to select 3-inch by 10-foot double-fluted tubes for the field test pilot plant (preconcentrator) planned for the follow-on EPA-sponsored project. These data generally confirm the above conclusions regarding the advantage of upflow VTFE over downflow VTE and VTFE, with both the aluminum-brass and titanium tubes used in these tests, especially in the higher temperature region.

Comparing the upflow VTFE data on Figure 7 with the Mohave cooling tower blowdown data of Figure 14, it is apparent that the addition of 10 ppm of surfactant had about equal effects on the heat transfer performance at 130°F<sup>b</sup> for these two different saline waters. In addition the response in upflow VTE heat transfer performance in Figure 6 corresponds closely with similar data obtained under the earlier EPA-sponsored project (5) with Mohave blowdown. Taken together with other reports from this Laboratory, these data provide confidence in upflow and downflow VTFE and VC-VTFE for the concentration of cooling tower blowdown, and for producing high quality distillate for recycle, boiler feed or potable use.

The second part of the objective of this work, the evaporation-crystallization of cooling tower blowdown, or of its preconcentrate obtained by VTFE, was carried out by using the second new pilot plant. This pilot plant, a 5,000 gpd rotatably mounted, upflow-downflow VTE was operated both with low temperature, reboiler steam as the source of heat, and with a newly acquired vapor compressor. This VTE was designed to provide reliable comparative heat transfer performance data for upflow versus downflow VTE, and VTFE data for vapor compression evaporation and for evaporation-crystallization with the downflow crystal-slurry mode of operation. The work reported here was obtained with the latter mode and was performed at two temperature levels: 120°F<sup>c</sup> with low pressure, reboiler steam, and 220°F<sup>d</sup> with compressed vapor as the source of heat energy. Two types of slurry-feed crystallization of relatively insoluble salts from

dilute solution (cooling tower blowdown and sodium sulphate) were examined at high temperature. The effects of surfactant addition on the heat transfer performance was investigated in each case.

In the crystallization of sodium sulphate from highly concentrated solution at low temperature, it was found that the addition of a surfactant did not enhance heat transfer but tended to reduce it, apparently because of the inhibition or retardation of crystallization which increases the boiling point elevation. Also, foamy vapor-liquid flow through the distillation tubes, previously shown to be required for good heat transfer enhancement, was not obtained in these low temperature, highly saline runs. It is therefore apparent that removal of surfactant from wastewaters preconcentrated by VTFE should be advantageous, if one wants to subsequently recover sodium sulphate by evaporation-crystallization (EC) with the slurry-feed mode of operation at low temperature (as proposed to EPA for the follow-on project). Such removal of the surfactant additives should have the beneficial effect of clarifying the VTFE preconcentrate, in the interest of a cleaner EC product. In cases where the slurried feed mode is applied in EC at high temperature, the more readily imposed foamy vapor-liquid flow with surfactant additives may overcome the boiling point elevation effect and the retardation effects of the additive. In such cases the presence of surfactant could be beneficial for heat transfer performance, but the crystalline product would contain adsorbed surfactant.

The crystallizing evaporation (EC) of Mohave cooling tower blowdown concentrate was performed at low temperature by the slurry-feed mode in the presence of a surfactant. The precipitated solutes (mostly calcium sulphate) reduced the heat transfer performance enhancement effects of the surfactant, as could be anticipated because of the apparent loss of surfactant due to adsorption on the precipitate. Some enhancement effect remained however, and removal of the surfactant from the VTFE preconcentrate does not appear advisable in such cases. Adsorption of the fully biodegradable surfactant on to solid residues to be ponded, does not appear objectionable. The removal of the surfactant additives by foam fractionation before final EC, and their recycle to the VTFE preconcentrator appear advisable when the final solid product is to be utilized subsequently. A photograph of this concentrate is shown in Figure 13.

Vapor compression (VC) downflow evaporation-concentration of agricultural wastewater, presoftened by ion exchange, was carried out at about 220°F<sup>a</sup> in the presence of 15 ppm of surfactant. The addition provided a 37 percent heat transfer enhancement effect at a TDS of 7000 ppm. This heat transfer enhancement remained effective through a 20-fold concentration, to 140,000 ppm; no salt crystallization was noticed during concentration, but some flocculent precipitate was noticed in the concentrate after cooling to room temperature. This concentration is reported in Table IV and the data are plotted in Figure 15.

Cooling tower blowdown from a large chemical-industrial complex was examined with VC-EC, to determine the effects of surfactant addition; data are reported in Table V and plotted in Figure 15. At an EC temperature of 220°F<sup>a</sup>, it was found that the addition of 10 ppm of surfactant

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<sup>a</sup>104.4°C

provided a 53 percent heat transfer performance increase. The evaporation or distillate productivity rate was increased while the  $\Delta T$  or power consumption was simultaneously reduced. A 27-fold concentration was obtained with a good interface enhancement (minimal of 34 percent) being maintained throughout in spite of the crystallization of some solutes. Distillate quality was good and represented 96 percent of this wastewater. The final concentrate, shown in Figure 13, had a volume of 4 percent of the original wastewater and contained the surfactant additive calculated to be in approximately 270 ppm concentration. Excessive foaminess was not a problem at any time, and removal of the additive would be feasible by foam fractionation if the solid residues are to be used as land fill or as chemicals.

A third cooling tower blowdown, obtained from a large petroleum refinery, was examined by VC-EC at 220°F<sup>a</sup>, to determine the effects of surfactant addition during its reduction to a slurry of only 2 percent by volume and 98 percent distilled water of high purity. Data are reported in Table VI and Figure 15. This wastewater contained about 14 ppm of chromate; this or another ingredient apparently inhibited foaminess with the 10 ppm of surfactant added. This adverse effect could have been overcome by adding more surfactant. The interface enhancement effect of 10 ppm Neodol 25-3A (Shell Chemical Co.) was however substantial (at 37 percent) and remained effective through 20 factors of concentration. As in the previous run, the removal of the additive by foam fractionation would not be necessary unless the residual solids are to be used subsequently, for instance as a source of chemicals. For the more usual case of ponding such residues, the presence of the surfactant additive should not be objectionable. This surfactant is claimed to be fully biodegradable, and non-toxic, by its manufacturer.

It appears, from these and previous VTFE test data obtained on a comparative basis with conventional test data, that the use of surfactant additives during the renovation of cooling tower blowdown to produce concentrated saline-crystalline slurries and pure distilled water for recycle, can be recommended.

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<sup>a</sup>104.4°C

## SECTION XI

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16. ABSTRACT <b>The report confirms the effectiveness of a novel evaporation method which reduces the energy and capital cost requirements for the renovation/recycle of industrial wastewaters. Interface enhancement (U.S. Patent 3,846,254) depends on foamy two-phase vapor/liquid flow induced during the evaporation of a liquid flowing over a heat transfer surface; this flow mode substantially increases the liquid's evaporation rate, after adding a surfactant. Two new vertical tube evaporation (VTE) pilot plants were used; a two-effect upflow/downflow VTE of 10,000 gpd capacity was produced by adding an identical downflow second effect to an existing single-effect upflow VTE pilot plant. This pilot plant was used to obtain comparative data on the concentration of saline water by upflow and downflow VTE, and by interface-enhanced upflow and downflow VTE (the interface-enhanced upflow VTE operation provides a higher performance than both the interface-enhanced and conventional downflow operations). A second pilot plant assembled for this work was a 5,000 gpd downflow VT evaporator-crystallizer. The heat transfer performance of this plant was increased and its energy requirements reduced, by applying interface enhancement to the concentration of several cooling tower blowdowns.</b>			
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