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TUBELESS EVAPORATION PROCESS DEVELOPMENT
Final Report

December 1987

Work Performed Under Contract No. AC07-86ID12619

For
U. S. Department of Energy
Office of Industrial Programs
Washington, D.C.

By
Schroeder Process Corporation
Santa Ana, California

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**TUBELESS EVAPORATION PROCESS
DEVELOPMENT**

Final Report

December 1987

Work Performed Under Contract No. DE-AC07-86ID12619

**Prepared for
U.S. Department of Energy
Idaho Operations Office, Idaho Falls, Idaho
Sponsored by the Office of the Assistant Secretary for
Conservation and Renewable Energy
Office of Industrial Programs
Washington, DC**

**Prepared by
Schroeder Process Corporation
Santa Ana, California**

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ABSTRACT

Schroeder, P.J. + Schroeder, D.W.

TUBELESS EVAPORATION PROCESS DEVELOPMENT

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A tubeless evaporation process (TEP) which has the potential to combine the advantages of both evaporation and freezing processes, without their disadvantages is being developed. The TEP is capable of concentrating process solutions of such things as sugar, caustic soda, salt, sodium sulfate, black liquor from the pulp and paper industry, cooling tower blowdown, "spent" pickling liquor (sulfuric acid) from the steel industry, and nitric acid with potential energy savings of half to three-quarters of the energy required by conventional evaporators, with about half of the capital and maintenance cost. It has similar potential for the production of fresh water from seawater, which was the principal application considered in the previous work. The process uses working fluids (WF's) at their freezing point to effect direct contact heat exchange. The purpose of this project was to find additional and lower cost WF's in the laboratory, to obtain sizing information for the major equipment for an economic evaluation and a pilot plant design in a bench scale plant, and to perform the economic evaluation, and the pilot plant design and cost estimate.

The process is ready for the next step in its development - build and operate the pilot plant.

Distillation processes + vapor compression evaporation + vertical tube evaporation + freeze process + waste disposal + research results + bench scale + energy costs + fixed costs + DOE sponsored.

PART I

EXECUTIVE SUMMARY

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619

INTRODUCTION

BACKGROUND INFORMATION

Commercial evaporators have long been known for their high capital, operating, and maintenance costs. They have also been known for their high energy consumption, a fact which has become increasingly important in recent years. The Tubeless Evaporation Process (TEP) is being developed to alleviate these problems. This project was directed towards furthering the TEP development.

The TEP was originally envisioned as a more energy efficient and economic process than current or emerging processes to produce fresh water from seawater, and other saline waters. However, it has become apparent that the widest application of the process would be as a concentrator, because of the increasing interest in energy-efficient concentration processes. At first, it was thought of as a brine concentrator (a concentrator of saline waters) which could be applied effectively to such streams as cooling tower blowdown. Further analysis suggested that the TEP was applicable to numerous other evaporator applications.

The TEP is a direct contact heat exchange process which has the potential for saving half to three-quarters of the energy required by conventional evaporators in the concentration of aqueous streams. Since the process is tubeless, it does not require energy to drive heat through tubes, and their attendant films and scale. Heat is transferred directly from an insoluble, inert, non-toxic **working fluid** to the solution to be evaporated (see **PROCESS DESCRIPTION FOR DETAILS**). Because the equipment is simple (comprised, mostly, of empty vessels and pumps), the process can also save capital, operating, and maintenance costs.

The process is applicable to solutions of such things as sugar, caustic soda, salt, sodium sulfate, black liquor from the pulp and paper industry, cooling tower blowdown, "spent" pickling liquor (sulfuric acid) from the steel industry, and nitric acid. It has similar potential for the production of fresh water from seawater, which was the principal application considered in the previous work (see **RELATED WORK**, below).

RELATED WORK

Closely related work was performed under the U.S. Department of the Interior, Office of Water Research and Technology Contract No. 14-34-0001-0450. That project demonstrated the suitability of the TEP for the evaporation of various feeds. The process developed in the precursor project has changed but little. The

multieffect mode of operation has been improved, and the required characteristics of working fluids have been developed.

During the course of that work, it was shown that the TEP operates satisfactorily in the major steps which are required to boil the water and recycle the working fluid. A variety of materials which were candidate components of working fluids were tested in the laboratory for their suitability in the process (insoluble in water, quickly separated from water etc.). The chemical stability of these compounds was tested under conditions much more severe than expected in commercial operations. Several satisfactory materials were identified. Tests of mixtures of these materials made demonstrated that the multieffect operation could be run with the effects separated thermally by as little as 2°F. A few of these materials were tested in a bench scale plant.

ORGANIZATION OF THE REPORT

This report is divided into two principal parts:

PART I - EXECUTIVE SUMMARY

PART II - DISCUSSION

Every part of the **SUMMARY** (which follows) is elaborated on in the **DISCUSSION**. Cross referencing is provided in order to facilitate finding more detail on any subject of particular interest to readers of the **EXECUTIVE SUMMARY**. References are **bolded** or not **bolded** in accordance with their appearance in the **TABLE OF CONTENTS**. **PART I** concludes with **THE PROJECT THROUGH PICTURES** which illustrates key project activities.

SUMMARY

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-861D12619

SUMMARY

PROCESS DESCRIPTION

Process Applications

The TEP is a substitute for conventional evaporators in either multieffect evaporator or vapor recompression plants. The only real difference between the two applications of the process is the source of the steam and destination of the vapor produced by the evaporation. In the multieffect application, the steam comes from the vapor of the previous effect (except for the first effect, which uses steam from an outside source), just as it does in a conventional multieffect evaporator. The vapor from the last effect goes to the final condenser. In the vapor recompression application, once the substitution of the TEP is made the plant operates in the same manner - the vapor from the evaporator is compressed and used again as the heat source. The only significant difference is that the energy required to do the compression is less, because no energy is required to drive heat through tubes.

Operation

The heat for evaporation comes from direct contact heat exchange. The feed liquid, which could be virtually any feed which is used in commercial evaporators, comes into direct contact with a working fluid which is in the liquid state, in a vessel called a Flasher. The working fluid becomes partially frozen and gives up its heat of fusion, which is used to boil a portion of the feed (which is condensed and recovered). The partially frozen working fluid is remelted in another vessel called a Melter/Condenser, and is sent to a Flasher to boil more feed.

The two ways of operating the process (single effect and multieffect) are described in detail the **PROCESS DESCRIPTION** section in **PART II**.

LABORATORY PROGRAM

Objectives

The so-called working fluid is the heat transfer medium for the process. Hence, it is also the core of the process. The very earliest work on the TEP was based on single component working substances, since purity was then thought to be essential to satisfactory operation of the process (Quite the opposite is now known to be true!). However, it soon became apparent that the addition of another non-freezing constituent called a carrier

fluid to the working substance was desirable under certain circumstances. This combination of a working substance and a carrier fluid was called a working fluid. Subsequently, as will be explained below, it was learned that working fluids are always better than working substances.

At the conclusion of the previous work, only one working substance suitable for use above 212°F was found, was unstable in chlorox and nitric acid. It is probably also carcinogenic. All suitable working substances were very expensive (ca. \$45/lb). Only two carrier fluids had been evaluated, and one of these was rejected because of lack of sufficient stability.

Hence, the primary objectives of this program were to:

1. Identify additional working substances with melting points in the range of 100 - 250°F, with special emphasis on those above 212°F
2. Reduce the cost of working substances
3. Identify additional carrier fluids

Working Fluids

In the work done prior to the proposal for this contract, emphasis was placed on the purity of the working substance (WS). Carrier fluids (CF's) were valued primarily because they were essential to multieffect operation. It was also recognized that CF's mitigated freeze-up problems. Work performed since the proposal was made, made it clear that CF's are not merely desirable for this purpose, but essential to practical operation of a plant. In order to transfer material from one vessel in the process to another, it is essential that a portion of this material remain liquid at all times and in all places in the process. Thus, CF's are regarded as essential, even in single effect operation.

Selection Criteria

As mentioned above, working fluids must meet certain selection criteria in order to be usable in this process. They must meet the requirements of insolubility in the feed, solvent (usually water), or concentrate. They must be chemically inert with all of these materials under all operating conditions. They must differ significantly (by at least 10%) in density from the feed, product or concentrate streams. And, they must contain no harmful materials if either the product (solvent) is to be potable water, or the concentrate is to be edible. Certain classes of materials - the paraffins and polyolefins, for example - are known to meet these requirements. But, they must also melt at the proper temperature.

When a pure material or a mixture is heated until it is completely melted and then is allowed to cool, the temperature will drop rapidly at first, and then it will drop slowly, thereby causing a change in inflection, called a "thermal flat", in its curve, called a "cooling curve". For materials that meet all of the other requirements mentioned above, the major concerns are that the material does have a thermal flat and that it occurs at a desired temperature. The materials regarded as "satisfactory" in the SUMMARY OF RESULTS section (following) meet the required criteria. Their cooling curves are presented in the next section.

Cooling Curves

Cooling curves were made by the method presented in COOLING CURVE DETERMINATION FOR WORKING FLUIDS. A total of 37 cooling curves are presented in COOLING CURVES. This data are summarized in SUMMARY OF COOLING CURVE DATA, followed by a discussion of MEASUREMENT ACCURACY - SOURCES OF ERROR, and SIGNIFICANCE OF RESULTS.

Summary of Results

The following is a brief summary of the results of this work. A more detailed summary is presented in SUMMARY OF RESULTS:

The Successful Candidates - Polyethylene and other Paraffins. The entire range of WF's between 100°F and 135°F can be provided by a set of mixtures of household paraffin (Parawax), and hexadecane. The range between 135°F and 165°F can be covered by a combination of two commercially available paraffin mixtures (Chevron CW-155 and Parawax). The range to about 245°F can be covered by a combination of paraffin (Chevron CW-155) and commercially available polyethylene. Thus, the entire range from 100°F to 245°F is now satisfactorily covered.

The "Technical" Successes. Several materials were found to be satisfactory in every way except one - they just plain cost too much! Interest in these declined after the successes reported above were achieved. Some of them also suffered from other deficiencies that would limit their use. Details are provided in SUMMARY OF RESULTS.

The Unsatisfactory Candidates. Two materials were found to be unsatisfactory. Details are provided in SUMMARY OF RESULTS.

BENCH SCALE PLANT PROGRAM

Objective

The primary objective of the Bench Scale Plant (BSP) Program was to obtain sizing information for major equipment for the Pilot

Plant. Three major pieces of equipment the Flasher, the Boiler (the Boiler is an auxiliary, actually, not part of the TEP), and the Melter/Condenser were designed and built of pyrex to allow observation of the tests. The Flasher and Melter/Condenser were mounted on a board which stood on the floor and was fastened to a bench. The boiler and other auxiliaries were located on the bench. The various pieces of equipment were connected together as required for Flasher runs or Melter/Condenser runs. These hook-ups are illustrated on **FLASHER SIMULATION** in that section, and **MELTER/CONDENSER SIMULATION** in that section.

Flasher Runs

Technical Approach. The primary information desired from the operation of the Flasher was the heat transfer coefficient. It was also hoped, and proved possible, to observe the speed and completeness of phase separation. The heat transfer coefficient sought would be the heat flux divided by the ΔT , the temperature driving force.

The determination of heat flux required the measurement of three things:

1. Heat transferred
2. Time
3. Volume

The heat flux was then calculated by the equation:

$$\text{Heat flux (Btu/hr ft}^3\text{)} = \frac{\text{Btu transferred}}{\text{ft}^3 \text{ hr}}$$

The Measurement of Heat Transferred. Several methods of measuring the heat transferred are possible:

1. Measure the change in concentration of the initial aqueous phase.
2. Measure the amount of water condensed by the condenser.
3. Determine the amount of solid formed.

All of these were tried. Because of experimental difficulties with the other methods, the third method became the standard.

Time. Perhaps surprisingly, this was not easy to measure. Several problems occurred, but perhaps the most serious was that the heat transfer rate was so high (good, in itself) that the runs were extremely short (1 to 4 minutes). This made percent error in measurement much higher than would have been the case if it took 15 or 20 minutes for all of the working fluid to freeze.

Volume. Calculations are based on the volume of WF at the start of the run.

Delta-T. Reasonably accurate determination of ΔT was impossible with the apparatus employed, because it is so small. Delta-T was assumed to be 1°F . This assumption is regarded as conservative.

Flasher Simulation

The procedure for making a Flasher run, with specific sample calculations, and a discussion of sources of error, is presented under FLASHER SIMULATION in PART II. This is followed by a data summary of the Flasher runs, in which the results of runs with six feeds - sodium chloride, caustic soda, "spent" pickling liquor, and three different concentrations of sugar solutions - are presented. As a result of this work, an overall heat transfer coefficient (U_V) of 80,000 Btu/(hr-ft³[of WF]- $^{\circ}\text{F}$) was used in the TEP ECONOMIC EVALUATION.

Melter Condenser Runs

Just as with the Flasher, the primary information desired from the operation of the Melter/Condenser was the heat transfer coefficient. And, once again, the heat transfer coefficient sought would be the heat flux divided by the ΔT .

Melter Condenser Simulation

The procedure for making a Melter/Condenser run, with specific sample calculations, and a discussion of sources of error, is presented under MELTER/CONDENSER SIMULATION in PART II. This is followed by a data summary of the Melter/Condenser runs, in which the results of four runs - three runs were made on hexatriacontane/hexadecane (HTC/HD) mixtures, and one on Chevron CW 155. The results were the same for both materials within the range of accuracy of the experiment. As a result of this work, an overall heat transfer coefficient (U_V) of 320,000 Btu/(hr-ft³[of WF]- $^{\circ}\text{F}$) was used in the TEP ECONOMIC EVALUATION.

Phase Separation

In addition to achieving the primary goal of obtaining heat transfer data from the Flasher runs, it was also hoped to observe the speed and completeness of phase separation. But, Flasher runs are continued until all of the working fluid is solidified, thereby making it impossible to take a sample of the working fluid at the end of the Flasher run. Thus, it became necessary to make special Flasher runs in order to obtain samples for entrainment tests.

Determination of Entrainment in Working Fluids

The procedure for making a Determination of Entrainment in Working Fluids is presented in PART II. This is followed by a summary of two runs. No water was detected in either sample. As little as 0.01 cc of water in 100 cc of working fluid would have been detected.

TEP ECONOMIC EVALUATION

Overall

When compared to present commercial practice, estimated savings for the TEP are as follows:

Energy Savings 50 - 75%

Capital Savings 45%

Maintenance Cost Savings 45%

Energy Savings

The most significant saving is in energy. Because the TEP does not require any ΔT to drive heat through tubes, it can easily fit 20 effects (four effects of one-quarter the capacity for each conventional effect - as illustrated above) into the available temperature range now used up by 5 effects. This would mean a 75 percent savings in energy requirements!!!

Capital and Maintenance Cost Savings

Present Commercial Practice. The basis for comparison is information provided by the Amalgamated Sugar Company on one of their existing evaporators (see Exhibit ASC). The costs for auxiliaries, such as piping, instruments, and controls, should be somewhat less for the TEP, because of its simplicity, but the effect would be small.

The existing third effect consists of two vessels operating in parallel:

Effect 3A - 14 ft Dia x 32.5 ft - 9,352 ft²

4,120 - 1-1/4" x 7'-3" tubes

Effect 3B - 14 ft Dia x 32.5 ft - 9,928 ft²

4,903 - 1-1/4" x 8'-4" tubes

Current cost for replacement with 304 S.S. tubes and tubesheets (in a carbon steel shell) is @ \$15.00/ft² or about \$290,000.

The TEP Replacement. The TEP replacement for this would be four effects in series. Each effect would consist of:

Flasher - three 4 ft Dia x 24 ft vessels, containing about 3,500 # of WF (@ \$0.30/#)

Melter/Condenser - three 4 ft Dia x 24 ft vessels. containing about 1,550 # of WF

WF Pump - 3,000 gpm @ 10 ft

Estimated Cost per Effect:

6 - 4 ft Dia x 24 ft vessels @ \$5,000 = \$30,000

5,000 # of WF @ \$0.30/# 5,000

1 - 3,000 gpm x 10 ft pump 5,000

Subtotal \$40,000

Total Estimated Replacement Cost:

Four Effects @ \$40,000 \$160,000

Estimated Savings

The estimated savings would be \$(290,000 - 160,000) \$130,000 or 45 percent on purchased equipment cost. This 45 percent figure would also apply to installed cost and maintenance cost.

Other Benefits

The previous section focused primarily on the savings in capital (and, hence maintenance) costs ascribable to the comparatively simple TEP design. But being tubeless has other advantages. The TEP also compares favorably with other direct contact processes (freezing). These subjects are discussed in OTHER BENEFITS in PART II.

Operating costs

Quantitative determination of annual costs is a site and company specific task, because these costs are a function of accounting methods, local conditions, et cetera. But there can be no doubt that the large reductions in such important contributors to cost as energy consumption (now replacing beets as the largest cost factor for beet sugar), and capital and maintenance costs will result in proportionate reductions in annual costs.

APPARENT PROBLEMS CONSIDERED

Concern has been expressed about certain apparent problems with the process, particularly with regard to losses and degradation of the WF, and entrainment of the WF in the vapor from the Flasher. Also, the economic impact of heat-tracing, and the controllability of the process in the multieffect mode, have been questioned. An attempt has been made to consider and deal with all of the problems. This examination has revealed that these problems are more apparent than real. The latest information on these most frequently asked questions is presented in APPARENT PROBLEMS CONSIDERED in PART II.

PILOT PLANT DESIGN

The overall objective is a Pilot Plant with the capability of operating over the range of temperatures (and their corresponding pressures) for the various feeds as presented in ENERGY SAVINGS in PART II. A summary the current design basis for the Pilot Plant, followed by the project and equipment drawings, is presented in PART II.

PILOT PLANT COST ESTIMATE

This section presents a summary of the purchase, installation, and total costs.

<u>PROCESS EQUIPMENT</u>	<u>PURCHASE COST</u>	<u>INSTALLATION HOURS</u>	<u>COST</u>	<u>TOTAL COST</u>
Tanks	\$14,090	80	\$3,200	\$17,290
Vessels	12,990	130	5,200	18,190
Pumps	13,580	17	680	14,260
Heat Exchangers	2,800	8	320	3,120
Vacuum Pumps	<u>1,360</u>	<u>4</u>	<u>160</u>	<u>1,520</u>
TOTAL PROCESS EQUIPMENT	\$44,820	239	\$9,560	\$54,380
TOTAL INSTRUMENTS	103,760	537	16,110	119,870
TOTAL CONTROL VALVES	<u>13,490</u>	<u>34</u>	<u>1,020</u>	<u>14,510</u>
TOTAL PURCHASED EQUIPMENT	\$162,070	810	\$24,300	\$188,760
STRUCTURAL				12,740
PIPING				42,910
ELECTRICAL				2,000
HEAT TRACING AND INSULATION				<u>30,600</u>
SUBTOTAL				\$277,010
SALES TAX @ 4% OF MATERIALS				7,950
FREIGHT @ 4% OF MATERIALS				<u>7,950</u>
SUBTOTAL				\$292,910
CONTINGENCY @ 7%				<u>20,500</u>
GRAND TOTAL				\$313,410

CONCLUSIONS

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-861D12619

CONCLUSIONS

CURRENT STATUS OF THE PROGRAM

All of the primary, and many of the supplementary objectives of the current project have been met.

Laboratory Program

A complete spectrum of working fluids (equivalent to mixtures of a working substance and carrier fluid) is now available to cover the range from 100 - 245°F.

Bench Scale Program

This program provided the major equipment sizing and operating data necessary for the TEP ECONOMIC EVALUATION, and for the PILOT PLANT DESIGN and PILOT PLANT COST ESTIMATE.

TEP Economic Evaluation

Expected energy savings are still as predicted (50 - 75 percent). Potential capital and maintenance savings are nearly twice what was anticipated (25 percent), because of the higher than anticipated heat transfer coefficients.

Pilot Plant Design and Cost Estimate

This work has confirmed that, as stated in the Project Justification, on a total cost basis, one percent of the potential annual energy savings alone in any one of the major industries that the TEP could serve would easily justify the cost of building and operating the Pilot Plant.

PROGRAM NEEDS

1. Build and Operate the Pilot Plant

This need overwhelms all others!

Any activity which is not directed towards this goal, will not significantly advance the commercialization of the TEP. Not that there are not other worthwhile things to do. There are, and they will be discussed below. But, no additional theoretical, laboratory, or bench scale work should be regarded as a precondition to getting on with the Pilot Plant. The Authors have expressed their confidence that the "apparent problems considered" are more apparent than real. We are just

as certain that anything less than a pilot plant which runs continuously for several hours at a time, and, is capable of running continuously for days at a time, will not provide any more convincing answers to these apparent problems than we have already.

2. Other Activities

It is quite possible, although not certain, that during the course of the Pilot Plant work, it may become desirable to provide a modest amount of laboratory or bench scale plant support. All other activities should be directed toward process improvement. Their real benefit will be realized after the process is commercialized. Nevertheless, it could be advantageous to pursue some of these now, in order to prevent loss of momentum, available skills, et cetera.

Theoretical Work. As mentioned above, there are two ways, or modes, of operating the process - single effect (or vapor recompression), and multieffect. Although these have been studied extensively, there is still fertile ground to be plowed here. It would be easy to assume that multieffect operation would always be the most energy efficient mode, but this may not be true in special circumstances, such as with feeds which have a high boiling point rise, like caustic soda, sodium sulfate, and pickling liquor. For such situations, a "hybrid" plant, which combines vapor recompression with a multieffect process, might be the best answer. Such an approach has not yet been considered for the TEP, but the Principal Investigator's extensive prior experience with hybrid plants employing conventional evaporators suggests that this should at least be considered.

Laboratory Work. As previously stated, a complete spectrum of working fluids is now available to cover the range from 100-245°F. This is the most important range. It covers the largest number of today's commercial operations. But there are advantages in going higher. Equipment gets smaller at higher pressures. More effects can be accommodated above the cooling water temperature, thereby further reducing energy consumption. There are others. Higher temperatures have often been considered for conventional evaporators. They have been rejected for various reasons. In some instances, the problem was caused by tubes. The accumulation of scale on the tubes, with its attendant loss of heat transfer, is one. The tendency of the metal to catalyze undesired reactions is another. The TEP could avoid these problems. Polyethylenes have melting ranges up to 290°F; polypropylenes run even higher. It seems possible that the range of working fluids could be extended with advantage to above 300°F. On the other end of the scale (below 135°F) hexadecane is used to lower the melting range of Parawax to the desired levels. While this

material is very moderate in cost when compared to the working substances previously used, it is still more expensive than any other of the recommended materials. It is quite likely that a lower cost substitute could be found.

Bench Scale Plant Work. No need for any additional bench scale work is foreseen at this time. A need could develop during the Pilot Plant Program.

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RECOMMENDATIONS

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619

RECOMMENDATIONS

It is recommended that all of the activities discussed in PROGRAM NEEDS above be carried out. However, it cannot be overemphasized that if Build and Operate the Pilot Plant is not done, the other activities would be useless. If only Build and Operate the Pilot Plant is done, the process will be commercialized (the other activities will, most likely, be done then).

It is recognized that all of the necessary resources (manpower, expertise, and financial) must be marshalled together in order to make these things happen. These are proper subjects for future proposals.

DOE-AC7-861D12619

THE PROJECT THROUGH PICTURES

BENCH SCALE PLANT SETUP

FLASHER SIMULATION

MELTER/CONDENSER SIMULATION

PART II

DISCUSSION

 

PROCESS DESCRIPTION

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619

PROCESS DESCRIPTION

PROCESS APPLICATIONS

The TEP can be thought of as a substitute for conventional evaporators in either multieffect evaporators or vapor recompression plants. The only real difference between the two applications of the process is the source of the steam, and destination of the vapor produced by the evaporation. In the multieffect application, the steam comes from the vapor of the previous effect (except for the first effect, which uses steam from an outside source), just as it does in a conventional multieffect evaporator. And the vapor from the last effect goes to the final condenser, in both cases. In the vapor recompression application, once the substitution of the TEP is made the plant operates in the same manner - the vapor from the evaporator is compressed and used again as the heat source. The only significant difference is that the energy required to do the compression is much less, because no energy is required to drive heat through the tubes.

OPERATION OF A SINGLE EFFECT

Whether a given effect is one of many in a multieffect plant, or the evaporator of a multieffect plant, the principal of operation for a single effect is the same. The Process Flow Diagram (following) depicts such an effect. The Feed (stream 1) to be evaporated is mixed with a liquid working fluid (stream 2) at its melting point in a flasher (V-1) in which part of the water in the feed evaporates (stream 3). The heat of vaporization is supplied by the partial freezing of the working fluid. The temperature remains constant. The aqueous layer, or "concentrate", which contains the scale forming solids (if any), and any other solids which come out of the feed during concentration, exits (stream 5) to the next flasher (if multieffect) or elsewhere in the plant (if single effect or last effect).

The partially frozen WF separates from the aqueous layer in the flasher (The most important qualities of a WF are that it be insoluble, and non-emulsifiable in water, and stable in the presence of water, and its impurities).

The mixture of liquid and solid WF (stream 6) is routed to a melter/condenser (V-2) or other suitable contacting device, such as a venturi. In the melter/condenser, the solid is melted by the steam (stream 7) which has come from a flasher (as explained below). This steam is condensed, and becomes the fresh water

MATERIAL BALANCE

STREAM No.	1	2	3	4	5	6	7	8	9	10
STREAM	FEED	W	PRODUCT WATER	ELIMINATED	CONCENTRATE	W	STEAM	PRODUCT WATER	COND WATER IN	COND WATER OUT
FLOW - LB/HR										
AGGRESSIVE	577,500		25,600	15,600	245,500	595,500	25,500	25,500	550,500	500,500
W		595,500								
FLOW - GPM										
AS COND - WTA	48.2				48.3					
WT SOLIDS - WTA		0				89.3				
TEMP - F	220.5	220.5	220.5	217.0	220.5	221.0	225.0	225.0	78.0	157.6
PRESS - PSIG			5.7				4.5			
VAC - IN. Hg										
Sp. G.	1.31	0.75		1.39	1.39	0.78		1.00	1.00	1.50

DEPARTMENT OF ENERGY
ISLAND OPERATIONS OFFICE
ISLAND FALLS, IDAHO

TUMBLESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC07-80ID15019

SINGLE EFFECT PLANT

SCHROEDER PROCESS CORPORATION
SANTA ANA, CALIFORNIA

DEPARTMENT OF ENERGY

DESIGNED P.J.R. DATE 2-28-87

REVISED

DRAWN CAP DATE 2-28-87

APPROVED

CHECKED DATE

DATE

DRAWING TITLE

DATE

PROCESS FLOW DIAGRAM

DRAWING NUMBER

2-20-87 DOE-2-1

"product" (stream 8). The melted WF is recycled to the flasher.

The vapor from the flasher (stream 9) goes to the melter/condenser of the next effect (multieffect plant), or is compressed slightly (single effect vapor recompression plant), and sent to the melter/condenser, or is sent to the final condenser (last effect of a multieffect plant). In the last case the vapor is condensed by cooling water (stream 9), thus producing the condensate stream (4) and the return cooling water stream (10).

THE MULTIEFFECT PROCESS

The Process Flow Diagram (following) depicts two effects of a multieffect plant. These effects may be the first and second effects of a multieffect system, intermediate effects, or the last and next to last effects, or, the multieffect system may comprise only two stages. Feed-I, which may be virgin feed stock or feed derived from a preceding effect, is fed into a flasher F-I, which also received molten working fluid from melter/condenser M-I. The flasher F-I, and the melter/condenser M-I, which receives Steam-I from any convenient steam source, or from a preceding effect, operate as previously described. Steam-I condenses in M-I, and is removed as Fresh Water-I. The steam from flasher F-I, Steam-II, provides energy input to melter/condenser M-II in the second effect, and the concentrate from flasher F-I constitutes Feed-II to flasher F-II. The flasher and melter/condenser operate as previously described wherein Steam-II condenses to become Fresh Water-II. Similarly, the outputs of flasher F-II constitute Steam-III and Feed-III which may go to a succeeding effect. If effect II is the last of a series of effects, then the Feed-III would be the final concentrate and would be removed from the system for further handling. The steam from this last effect would be condensed and constitute the final product water of the multieffect system. Normally, Fresh Water-I would be returned to the steam source as condensate. The remaining fresh water streams would be the product water of the plant.

PROCESS FLOW DIAGRAM

TWO EFFECT PLANT

LABORATORY PROGRAM

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-86LD12619

LABORATORY PROGRAM

WORKING FLUIDS

In the work done prior to the proposal for this contract, emphasis was placed on the purity of the working substance (WS). Carrier fluids (CF's) were valued primarily because they were essential to multieffect operation. Sufficient working substances do not exist to permit spacing effects close enough together, thermally, to make multieffect operation possible, unless the WS's are "stretched" by combination with CF's. It was also recognized that CF's mitigated freeze-up problems. Work performed since the proposal was made, made it clear that CF's are not merely desirable for this purpose, but essential to practical operation of a plant. In order to transfer material from one vessel in the process to another, it is essential that a portion of this material remain liquid at all times and in all places in the process. The transfer of a totally frozen solid mass from the flasher to the melter/condenser would be prohibitively expensive. This would be extremely difficult to do if a single pure compound were employed since an incremental drop in temperature would cause all of the material to freeze. This event is a near certainty during shutdown of the system, an expected event which must be considered in process design. It is also a highly likely occurrence at stagnant places in the system during normal operation. Considerable expense for both equipment and energy for heat tracing would be needed in order to make such a plant operable, and even then, operation would be difficult. Thus, CF's are regarded as essential, even in single effect operation.

The combination of a WS and a CF is called a working fluid (WF). Generally speaking, in such a combination, the WS is regarded as the "phase change constituent" since most or all of what freezes is the WS. However, in at least some of the WS/CF combinations or WF's proposed, solid solutions occur, so a small portion of the CF also freezes. Furthermore, a material that would be an acceptable WS in a low temperature effect, might be an acceptable CF in a high temperature effect. Hence, distinguishing between the search for WS's (Task 1), and CF's (Task 2), is somewhat arbitrary. Both must comply with the same requirements of insolubility in the feed, solvent (usually water), or concentrate. They must be chemically inert with all of these materials under all operating conditions. They must differ significantly (by at least 10%) in density from the feed, product or concentrate streams. And, they must contain no harmful materials if either the product (solvent) is to be potable water, or the concentrate is to be edible.

For the purposes of this project, this arbitrary distinction is made as follows: To be regarded as a WS, the material must have the capability of being the primary phase change constituent at a practical operating temperature (This does not rule out the possibility, or the consideration, that this same material might be a perfectly acceptable material as a CF with another WS at a higher temperature). Carrier fluids are being sought with the primary intention of serving in the role (as CF's). They may not be capable of serving as WS's because their melting point may be too low (e.g. hexane). This does not rule out the possibility that some of the CF's considered may also be usable as WS's, with lower temperature CF's. Another possibility exists. Certain materials, such as Fischer-Tropsch waxes (described below), may have the proper melting range to be used as WF's, without the addition of a CF.

Task WF - New Working Fluids

As early as Report No. 3, the third month of the project, it was reported that the minimum requirement for tasks 1 and 2 had been exceeded, but that additional working fluids (WF's) which melt above 100°C would be sought. The following is taken from that report:

TASK 1 - IDENTIFY AND TEST (AT LEAST TWO) WS's

This task requires that at least two working substances (WS's) be chosen and tested for their application to the TEP for the feed solutions of task 4, and that at least one of these will be selected for potential use above 100°C. This has been done. Two paraffins have been tested and found suitable for use below 100°C, and triphenylbenzene has been tested and found suitable for use above 100°C.

Although the minimum requirements for this task have already been exceeded, further work with other materials is planned.

TASK 2 - IDENTIFY AND TEST (AT LEAST TWO) CF's

This task requires that at least two carrier fluids (CF's) be identified and tested for their application to the TEP in combination with WS's identified in task 1, or used previously. This has been done. Hexadecane was found to be acceptable with hexatriacontane and unsatisfactory with triphenylbenzene. Also, xylene and biphenyl were found to be acceptable with triphenylbenzene, and hexane was found to be unacceptable with triphenylbenzene. In summary, four CF's have been tested with two WS's producing three successful WS/CF combinations.

Although the minimum requirements for this task have already been exceeded, additional WS/CF tests will be made.

Subsequent to that time, all further work was reported under TASK WF.

Selection Criteria

As mentioned above, working fluids must meet certain selection criteria in order to be usable in this process. They must meet the requirements of insolubility in the feed, solvent (usually water), or concentrate. They must be chemically inert with all of these materials under all operating conditions. They must differ significantly (by at least 10%) in density from the feed, product or concentrate streams. And, they must contain no harmful materials if either the product (solvent) is to be potable water, or the concentrate is to be edible. Certain classes of materials - the paraffins and polyolefins, for example - are known to meet these requirements. But, they must also melt at the proper temperature.

When a pure material or a mixture is heated until it is completely melted and then is allowed to cool, the temperature will drop rapidly at first (until its liquidus temperature is reached) and then will drop slowly, thereby causing a change in inflection (called a "thermal flat") in its curve (called a "cooling curve"). This phenomenon is known as "thermal arrest". The reason that it occurs is that, while heat is being removed from the mixture at a nearly constant rate, it is only sensible heat (ca. 0.5 Btu/#) until the liquidus temperature is reached. Then it is heat of fusion (ca. 63 Btu/#) that is being removed. Of course, when the material is completely solidified, it is sensible heat that is being removed, once more. The temperature would tend to drop more rapidly again, but this change in inflection is much harder to detect. When the mixture is completely solidified, it is no longer stirred. This causes a significant drop in the heat transfer rate, which partially offsets the effect of removing sensible heat instead of heat of fusion. Fortunately, it is the "thermal arrest" that is of primary interest in the work at hand. For materials that meet all of the other requirements mentioned above, the major concerns are that the material does have a thermal flat and that it occurs at a desired temperature. The materials regarded as "satisfactory" in the SUMMARY OF RESULTS section (following) meet the required criteria. Their cooling curves are presented in the next section.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-86ID12619
LABORATORY PROCEDURES

COOLING CURVE DETERMINATION FOR WORKING FLUIDS

SCOPE

This describes a procedure for determining the cooling curve for working fluids (WF's).

Summary

A sample of the WF is placed in a beaker about half full of water (caustic soda if the bath is to operate above 95°C). The mixture is heated and stirred until all of the WF is melted. The heat is shut off, but stirring is continued. As the mixture cools, periodic readings of its temperature are taken and appearance of solids noted. The temperature range for the mixture is from the temperature at which a minimum rate of temperature change and the appearance of solids first occurs to the temperature at which the mixture is completely solidified.

Application

This method is applicable to mixtures. Other methods for test of pure materials are not suitable because they do not employ mixing. These methods (which do not employ mixing) are satisfactory for pure materials because once solids appear, the remaining liquid is of the same composition. But for mixtures, an opportunity must be provided for the solids that form to equilibrate with the remaining liquid in order to establish the solidus and liquidus lines correctly.

Definition

The melting range begins with the temperature at which a minimum rate of temperature change and solids first appear to the temperature at which the mixture is completely solid, when the mixture is allowed to cool as specified herein.

Apparatus

The apparatus consists of the following:

1000 ml beaker

-20 to 150°C partial immersion thermometer with 1° subdivisions, calibrated to meet tolerance limits set by National Bureau of Standards Monograph 150.

hot plate stirrer with stirrer bar

ring stand with clamp and rubber stopper (for holding thermometer)

Procedure

1. During this entire procedure, record any unusual occurrences which are observed.
2. Approximately one-half fill the beaker with water (for operation above 95° use a caustic soda solution of sufficient strength to prevent boiling at the intended operating temperature.
3. Add stirrer bar and place the beaker on the hot plate stirrer.
4. Add sufficient WF to bring the level in the beaker to about three-quarters full.
5. Turn on stirrer.
6. Turn on heater.
7. Insert thermometer so that the bulb is immersed in the WF as soon as sufficient liquid forms.
8. Continue heating to a temperature at least 10°C above the temperature at which the WF is all melted.
9. Shut off heat (continue stirring).
10. Read the thermometer at one minute intervals, and record the temperature to the nearest estimated 0.1°C .
11. Read the thermometer when solids first begin to appear, and record the temperature to the nearest estimated 0.1°C .
12. Continue until WF is completely solidified.

Report

Plot temperature versus time.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-86ID12619
LABORATORY PROGRAM

COOLING CURVES

ORDER OF PRESENTATION

In an attempt to aid the reader in "walking in the footsteps of the researcher", the cooling curves are presented in chronological order. However, care must be taken in inferring that the results of one test lead to the next. The planned course was clear at the time of the first monthly report. Availability of material played a major role in the order in which the work was done. Polyethylene, which was mentioned as a promising candidate in that first report, was not available until several weeks later.

In the summary that follows, the data from the cooling curves is grouped by type of material, in order to facilitate comparison.

SYSTEM
PARAWAX (FROM LOCAL SUPERMARKET)

FIGURE CC-1

SYSTEM
10% TRIPHENYLBENZENE IN XYLENE

FIGURE CC-2

SYSTEM

10% TRIPHENYLBENZENE IN BIPHENYL

FIGURE CC-3

SYSTEM
50% TRIPHENYLBENZENE IN XYLENE

FIGURE CC-4

SYSTEM
50% TRIPHENYLBENZENE IN BIPHENYL

FIGURE CC-5

SYSTEM
PARAWAX (REPEAT OF CC-1)

FIGURE CC-6

SYSTEM
PARAWAX (COOLED IN WATER)

FIGURE CC-7

SYSTEM
CHEVRON CW-155

FIGURE CC-8

SYSTEM
75% PARAWAX IN HEXANE

FIGURE CC-9

SYSTEM
50% PARAWAX IN HEXANE

FIGURE CC-10

SYSTEM

75% PARAWAX IN HEXADECANE

FIGURE CC-11

SYSTEM
50% PARAWAX IN HEXADECANE

FIGURE CC-12

SYSTEM
25% PARAWAX IN HEXADECANE

FIGURE CC-13

SYSTEM
25% CHEVRON CW-155 IN PARAWAX

FIGURE CC-14

SYSTEM
50% CW-155 IN PARAWAX

FIGURE CC-15

SYSTEM
75% CHEVRON CW-155 IN PARAWAX

FIGURE CC-16

SYSTEM
90% CHEVRON CW-155 IN PARAWAX

FIGURE CC-17

SYSTEM
CHEVRON CW-155 (REPEAT OF CC-8)

FIGURE CC-18

SYSTEM

UCC POLYETHYLENE

FIGURE CC-19

SYSTEM
UCC POLYETHYLENE (REPEAT OF CC-19)

FIGURE CC-20

SYSTEM
EPOLENE N-10

FIGURE CC-21

SYSTEM
EPOLENE N-10 (REPEAT OF CC-21)

FIGURE CC-22

SYSTEM
EPOLENE N-10 (REPEAT OF CC-22)

FIGURE CC-23

SYSTEM
EPOLENE N-14

FIGURE CC-24

SYSTEM
EPOLENE N-14 (REPEAT OF CC24)

FIGURE CC-25

SYSTEM

50% EPOLENE N-10 IN CHEVRON CW-155

FIGURE CC-26

SYSTEM

33% EPOLENE N-10 IN CHEVRON CW-155

FIGURE CC-27

SYSTEM

33% EPOLENE N-10 IN CHEVRON CW-155 (REPEAT OF CC-27)

FIGURE CC-28

SYSTEM

50% EPOLENE N-10 IN CHEVRON CW-155 (REPEAT OF CC-26)

FIGURE CC-29

SYSTEM

25% EPOLENE N-10 IN CHEVRON CW-155

FIGURE CC-30

SYSTEM

50% EPOLENE N-14 IN CHEVRON CW-155

FIGURE CC-31

SYSTEM

33% EPOLENE N-14 IN CHEVRON CW-155

FIGURE CC-32

SYSTEM

25% EPOLENE N-14 IN CHEVRON CW-155

FIGURE CC-33

SYSTEM

33% EPOLINE N-15 IN CHEVRON CW-155

FIGURE CC-34

SYSTEM

25% EPOLENE N-15 IN CHEVRON CW-155

FIGURE CC-35

SYSTEM

50% EPOLENE N-15 IN CHEVRON CW-155

FIGURE CC-36

SYSTEM

33% HEXATRIACONTANE IN HEXADECANE

FIGURE CC-37

SUMMARY OF COOLING CURVE DATA

<u>SYSTEM</u>	<u>Cooling Curve</u>	<u>Thermal Arrest Temperature °C</u>
<u>Triphenylbenzene</u>		
10% in Xylene	2	47
50% in Xylene	4	--*
10% in Biphenyl	3	66
50% in Biphenyl	5	105
* This curve was plotted in order to show what the cooling curve looks like for a typical case with no thermal flat.		
<u>Parawax (from Supermarket)</u>		
As received	1	59
Repeat of CC-1	6	58
Cooled in water	7	60
75% in Hexane	9	50
50% in Hexane	10	43
75% in Hexadecane	11	50
50% in Hexadecane	12	47
25% in Hexadecane	13	40
<u>Chevron CW-155</u>		
As received	8	71
Repeat of CC-8	18	72
25% in Parawax	14	60
<u>Chevron CW-155 (Continued)</u>		
50% in Parawax	15	63
75% in Parawax	16	65
90% in Parawax	17	67
<u>UCC Polyethylene</u>		
As received	19	119
Repeat of CC-19	20	118
<u>Epolene N-10</u>		
As received	21	104
Repeat of CC-21	22	102
Repeat of CC-22	23	103
25% in Chevron CW-155	30	91
50% in Chevron CW-155	31	92
33% in Chevron CW-155	32	86

Epolene N-14

As received	24	99
Repeat of CC-24	25	101
50% in Chevron CW-155	26	95
Repeat of CC-26	29	96
33% in Chevron CW-155	27	94
Repeat of CC-27	28	93
25% in Chevron CW-155	33	74

Epolene N-15

33% in Chevron CW-155	34	70
25% in Chevron CW-155	35	69
50% in Chevron CW-155	36	119

Hexatriacontane

33% in Hexadecane	37	60
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MEASUREMENT ACCURACY - SOURCES OF UNCERTAINTIES

The most obvious sources of error (not in order of importance) are errors in the thermometer (minuscule), errors in reading the thermometer (can be read to within about 0.1°C), and errors resulting from non-uniform mixing of the sample (the observed temperature may or may not be the average temperature of the mixture). Of these sources of error, the last one is the only one with the apparent potential to be significant (see SELECTION CRITERIA, above). For this reason, the tests were repeated on some of the samples, in order to get an indication of reproducibility. Six tests were repeated once; one test was repeated twice. Of those that were repeated once, the worst case showed a difference of 1°C or about 0.25% from the mean; the rest of the cases showed only about half of that difference, as did the case that was repeated twice. These differences cannot be regarded as significant for the purposes of the project at hand. An inspection of the cooling curves themselves will reveal that the rate of cooling was different on the repeated tests; hence the curves have different slopes. This is also of no consequence. The tests were run to determine the temperature at which thermal arrest occurs. This temperature is the proper basis for comparison.

SIGNIFICANCE OF RESULTS

These tests were run in order to demonstrate that sufficient materials, which meet all of the selection criteria, exist to cover a wide temperature range. The SUMMARY OF RESULTS shows that this is the case. The entire temperature range of $100\text{--}245^{\circ}\text{F}$ can be covered by using materials of the type tested. It

is quite likely that this range can be expanded to 290°F. It could also go lower, but operation below 100°F tends to become impractical, because the low vapor density requires very large equipment.

SUMMARY OF RESULTS

A summary of the results of this work follows:

The Successful Candidates

Polyethylene and Other Paraffins. Real progress began when the concept of WS's and CF's were merged into the single concept of WF's. These WF's were to be made of combinations of paraffins because paraffins are completely insoluble in water, are regularly used in food, (coating milk cartons, paper cups, fruit, etc.), and cost less than \$0.40/lb. This new approach has produced very good results. The entire range of WF's between 100°F and 135°F can be provided by a set of mixtures of household paraffin (Parawax) and hexadecane. The range between 135°F and 165°F can be covered by a combination of two commercially available paraffin mixtures (Chevron CW-155 and Parawax). And the range to about 245°F can be covered by a combination of paraffin (Chevron CW-155) and commercially available polyethylene. Thus, the entire range from 100°F to 245°F is now satisfactorily covered.

The "Technical" Successes

Several materials were found to be satisfactory in every way except one - they cost too much! Interest in these declined after the great successes reported above were achieved. Some of them also suffered from other deficiencies that would limit their use.

Fischer-Tropsch Waxes. Previous work with waxes, produced by the Fischer-Tropsch process indicated that such materials (as manufactured by Thornstensen Laboratories) had the potential to be WF's. The product of the reaction is fractionated into various cuts. Some have melting ranges of 158-167°F, another of 185-194°F, et cetera. These products provoked continuing interest. However, additional study revealed problems that caused postponement of further consideration, until alternative prospects could be considered. The combining of various cuts to produce the desired melting range for each effect would be difficult. The highest melting range available is just barely above 212°F. Also, the one product with a melting range above 212°F is available (according to the manufacturer) in quantities so limited that it would be difficult to produce sufficient quantities for a commercial plant. Of course, more of this cut could be produced, but then a market would have to be found for the lower melting cuts, or all of the cost would have to be borne by the desired cut, which would be prohibitively expensive.

Hexatriacontane and Hexadecane. A mixture of hexatriacontane (WS) and hexadecane (CF) was extensively tested. Hexatriacontane, identified in previous work, is technically

highly satisfactory, but very expensive, above \$40/lb. This high cost provided the incentive to look for other materials.

Hexadecane is also satisfactory from every technical viewpoint as a CF. The only problem is that, at above \$1 per pound, it is somewhat expensive for the intended purpose.

Triphenylbenzene and Biphenyl. A viable, but not optimum system employing triphenylbenzene, melting point 338°F, and biphenyl (CF) was also found. This last combination might be worthwhile to use in a case where a very high temperature was desirable, and where the water produced would be recycled, not diverted to potable use (e.g. caustic soda). Triphenylbenzene is probably toxic although it is not mentioned in any current list (most likely because it is not in general use). It is now expensive, although it could probably be manufactured very cheaply from acetophenone. It is heavier than water. Biphenyl (not a chlorinated compound) is cheap, stable, and available. It is also toxic and heavier than water.

Triphenylbenzene and Xylene. Ten percent triphenylbenzene in xylene was found to have a thermal arrest at 113°F.

Parawax and Hexadecane. Cooling curves were determined for "Parawax" with 25, 50, and 75 percent hexadecane. Thermal arrests at about 126, 116 and 109°F were observed.

The Unsatisfactory Candidates

Chevron 225. A cooling curve was run on "Chevron 225". No real thermal flat appeared. Further work on this sample was abandoned.

Triphenylbenzene and Xylene. Fifty percent triphenylbenzene in xylene produced lots of solids but no thermal flat. Hence, this combination was not regarded as a satisfactory WF.

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BENCH SCALE PLANT PROGRAM

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619

BENCH SCALE PLANT PROGRAM

The primary purpose of the Bench Scale Plant (BSP) Program was to obtain sizing information for major equipment for the Pilot Plant. Three major pieces of equipment the Flasher, the Boiler (the Boiler is an auxiliary, actually, not part of the TEP), and the Melter/Condenser were designed and built of pyrex to allow observation of the tests. The Flasher and Melter/Condenser were mounted on a board which stood on the floor and was fastened to a bench. The boiler and other auxiliaries were located on the bench. The various pieces of equipment were connected together as required for Flasher runs or Melter/Condenser runs. These hook-ups are illustrated on **FLASHER SIMULATION** in that section, and **MELTER/CONDENSER SIMULATION** in that section.

In the pages that follow, the information sought from the various runs (Flasher, Melter/Condenser, and Entrainment in Working Fluid), the procedure used for making them, with specific sample calculations, and a discussion of sources of error, are presented. This is followed by a data summary.

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-861D12619

BSP PROGRAM

FLASHER RUNS

TECHNICAL APPROACH

The primary information desired from the operation of the Flasher was the heat transfer coefficient. It was also hoped, and proved possible, to observe the speed and completeness of phase separation. Before the experimental work started, it was possible that the coefficient should be based upon the interface between the organic layer and the layer of concentrate (with respect to the feed) below. Such data would Btu/hr-FT²-°F. A few experiments settled this problem. When the volume of WF was doubled, while the gross interface between WF and undisturbed concentrated brine remained the same the rate of heat transfer within the unit doubled. This made confirmed that the heat transfer coefficient must be expressed per unit volume rather than unit area. The heat transfer coefficient sought would be the heat flux divided by the ΔT , the temperature difference which is the driving force.

The determination of heat flux required the measurement of three things:

1. Heat transferred
2. Time
3. Volume

The heat flux was then calculated by the equation:

$$\text{Heat flux (Btu/hr ft}^3\text{)} = \frac{\text{Btu transferred}}{\text{ft}^3\text{-hr}}$$

The approach to this problem was necessarily a matter of trial and error. No one, to our knowledge, had previously confronted this problem, at least in the exact circumstances that we face. The only pertinent previous work that might have given guidance was the work on the freezing process and that of Wilkie (ref. 1) on use of direct contact for boiling water. However, Wilkie did not freeze his working fluid (a mixture of PCB's), and the behavior of the butane-ice-brine system at low temperature provided no exact parallel.

THE MEASUREMENT OF HEAT TRANSFERRED

To make these measurements, it was first of all necessary to establish the system in a very nearly adiabatic condition. Since the WF has to be melted, this requires that the system be at an elevated temperature (e.g. 160°F) compared to that of the room.

Even when insulation was employed, some small heat input was required to keep the system at high temperature and the WF melted. Electrical heating tapes, controlled by a powerstat, were used for this purpose. High power was used to melt the WF and heat the concentrate. The power was then turned down to the point that a predetermined temperature could be maintained in the apparatus. There was sometimes a difference in temperature between the WF layer and the concentrate layer, since they were on different sets of heaters and powerstats. However, each temperature was constant. When the run began, the pressure was lowered and the WF layer dropped to its freezing temperature. It then started to solidify, providing, from its heat of fusion, the heat required to boil the water. At this lower temperature, there was surely a heat leak into the system, but it was very small. If only the heat resistance of the glass is considered, the heat transfer rate was only about 30 Btu/hr-ft²-°F. This compares to an internal heat exchange between the WF and the aqueous phase of about 100,000 Btu/hr-ft³-°F.

The problem of measuring the heat transferred must still be addressed. Several methods seemed possible:

1. Measure the change in concentration of the initial aqueous phase.
2. Measure the amount of water condensed by the condenser.
3. Determine the amount of solid formed.

Measurement of Change in Initial Concentration

This alternative seemed unpromising due to the difficulty in determining the amount of aqueous phase that was present at the beginning and end of a run; a difficulty made even worse during early unsuccessful attempts to provide continuous feed and take out product continuously. The change in concentration was also difficult to measure since it was very small. Still, the few attempts to measure things this way, did confirm, within the expected degree of accuracy, the measurements made by other methods.

Measurement of Water Condensed

In all flasher runs, measurements were made of the amount of condensate produced. Serious experimental difficulties, which caused the apparent quantity of heat transfer to be low, soon became apparent. As the experimental difficulties were gradually mitigated, it was this method improved sufficiently to bring it into agreement with the third method, the amount of solid formed. The third method proved to be the most reliable, and became the standard.

It is now appropriate to mention the difficulties that attend attempts to measure heat transferred by measuring the amount of condensate collected during a run. The measurement of the amount of condensate collected was easy. It could be measured within a cubic centimeter from a typical total of about 100 cubic centimeters. The difficulty was in relating this measurement to the real quantity of heat transferred in the flasher. A "run" was usually done by starting with a closely known amount of liquid WF in the flasher, at a known temperature, above a less closely known amount of aqueous phase, also at a known temperature. Pressure was lowered sufficiently to induce boiling. The temperature of the WF/aqueous phase section rapidly dropped to the freezing temperature of the WF and thereafter gradually fell. When about half of the expected solid was produced the run was discontinued by releasing the vacuum. The net condensate collected was measured. However, the boiling action was so vigorous that solid often carried into the exit tubing plugging the tubes and aborting the run. Even when this did not happen, some solid was deposited on the wall of the vapor space. This solid was originally deposited as a liquid, and became solid only by heat loss from the vapor space. The WF so deposited could not contribute to the boiling of water. The result was that less than the expected amount of condensate was collected.

Some attempts were made to correct these problems. First, attempts were made to bring the vacuum up more slowly so as not to have too violent an action at the start. These attempts were supplemented by trying to control the vacuum more accurately by use of a cartesian diver pressure control. None of these efforts were more than marginally successful. When the vacuum reaches the point where boiling begins, action is violent. To mitigate this problem, the flasher's 2" column was replaced by a 4" column. This involves the additional cost of material and the additional potential for breakage. The results seemed to justify the change. Deposition of solid in the vapor space and carry-over to the melter/condenser decreased to where they lowered the measured amount of heat transferred by only a few percent. After these changes, the measurements of heat transferred appeared more reasonable, but were still uncertain. This was caused by water refluxing from the vapor space of the flasher. Any water that refluxed had to be evaporated again or was retained in the aqueous phase after the run had stopped. Refluxing caused a direct loss in the measured quantity of heat transferred. Heating of the vapor space could offset this factor. Such heating of the vapor space would contributed nothing to the water evaporated from the flasher; only to the correct measurement of it. Increased quantities of heat transferred were observed (Unfortunately, this was accompanied by increased glass breakage), but it became obvious that these problems could not be completely overcome in the existing experimental apparatus.

Recourse was then taken to method three of measuring the quantity of heat transferred - measuring the amount of solid formed.

Measurement of the Amount of Solid Formed

This procedure, which differed from the original test protocol for the project, resulted in reproducible results. Errors, of course, existed, but they were too small to measure in the BSP. Such data will be forthcoming from the Pilot Plant. Continued attention to the gathering of condensate eventually brought this set of observations into line with the observations made by determining the amount of solid formation. Never, however, was the measurement of condensate formation totally reliable.

The reason that observation of solid formation was against the previously perceived protocol for the project is that attempting to feed the flasher and remove condensate from the melter condenser, became useless exercises. They could be carried on, but had no effect on the final result. However, it is contended that the experiments were valid. There was a diminution in the amount of concentrated aqueous phase in the flasher and accumulation of condensate in the melter condenser. Continuous feed to the flasher and withdrawal from the melter/condenser would have preserved, at a superficial glance, an apparent appearance of continuous operation, but it would have really not changed things, and would have made more difficult an already intricate series of operations during the runs. In using the solid formation as measure of the quantity of heat transferred, it became necessary to totally solidify it. The amount of working substance to be frozen was determined by weighing the charge of WF put in the flasher.

TIME

Perhaps surprisingly, this was not easy to measure. The problem of determining time becomes apparent when the course of a run is considered. At the start of a run, it is necessary that the WF be above its liquidus temperature (As experience was gained, the temperature at the start was only very little above the liquidus temperature). The concentrated brine layer was at, or slightly above, the liquidus temperature of the WF. If it was not, a layer of solid would be formed at the interface between the layers. When vacuum was applied, the liquid would start boiling and entering the WF layer. The evolution of steam would cause the WF layer to expand. This was the beginning of significant heat transfer. For a very brief time, the WF is cooled to its liquidus temperature, and the liquid from the aqueous layer, now entrained in the expanded WF/aqueous layer, rises to its boiling point. At this point, the active layer is in violent agitation and assumes a definite temperature. This temperature starts out at the liquidus temperature of the WF and slowly decreases. If the run is continued until all the WF is frozen, the temperature

becomes equal to the solidus temperature of the WF. If the run is continued thereafter, the temperature begins to drop quite rapidly. The temperature versus time profile exactly resembles a cooling curve, because that is what it is. Based on these observations, an arbitrary, but guided, definition was established. The time is taken from the comparatively flat part of the temperature versus time curve.

VOLUME

It seems that it should be easy to define volume, and to measure it at least closely. It isn't. The experiments are begun with the flasher, of known diameter, charged with liquid WF at a temperature above its freezing point (actually liquidus temperature). The height of this section is easily measured, since the flasher is glass. Below the liquid, is a concentrate of known composition, the volume of which is known at least to a good approximation. Lines lead out to the condenser. During the run the WF section expands to about twice its previous volume. The total volume of the apparatus seems the wrong volume to use. There may have been too much space occupied by the concentrate, and either too much or not enough vapor disengaging space. Calculations are based on the volume of WF at the start of the run. This choice is not entirely arbitrary. Empty space in the vessel is cheap, compared to space occupied by working fluid.

Since some material could be lost during a run (carried over into the melter/condenser, or deposited on the wall of the vapor space, the height was regularly checked between runs. Very little was carried over once the 4" diameter flasher was adopted and more careful control of initiating the vacuum was practiced. There was always some deposition of solid on the wall of the vapor space, but the error was small. Since the material on the wall of the vapor space was always returned to the main body of working fluid between runs, either by scraping or by melting, the error was not cumulative.

DELTA-T

Accurate determination of ΔT is still unsolved as will be discussed below. It is known to be small. The arbitrary assumption that is 1°F made below is conservative. The situation is not unlike that discussed in a classic paper by Weigandt (ref. 2), "Because both the evaporation and condensation are by direct contact of the refrigerant with water or ice, the opportunity exists for realistic achievement of a low over-all driving force. Experimentally, the evaporation has been found to occur so rapidly, that a rate measurement has not been practical. The melting of ice is more of a problem because the condensate film is a heat transfer barrier. However, the barrier is a fluid and not a stationary wall as in a distillation unit, wherein steam is

condensed against water or boiling water." The reasons for believing that ΔT is very low are detailed below.

In this adiabatic operation, essentially all of the heat required to vaporize water is produced by the freezing of the WF. Therefore, the operation must take place at the WF's freezing temperature. The aqueous phase must be at a temperature below the WF temperature. When the WF and vaporizing liquid are intimately mixed in the active part of the flasher, it is impossible to distinguish between WF temperature and aqueous temperature. However, the continuous aqueous layer below the mixed zone can have a temperature different from that of the mixed portion. Is this temperature significant for determination of ΔT ? The answer to this question is no. If the continuous aqueous phase is lower in temperature than the mixed phase, as liquid is drawn into the mixed phase it will have to be heated to its boiling point. This requires a little heat in addition to the heat of vaporization, but the effect is very small. If the continuous aqueous phase is warmer than the mixed phase when the aqueous phase is drawn in a very small part of it will flash, thus cooling it to boiling temperature. Thus, the temperature of the aqueous phase is of not great importance and has nothing to do with effective ΔT .

In theory, the boiling point of the aqueous phase could be determined by measuring the pressure, reading the equilibrium temperature in the steam tables, and adding the boiling point rise. In practice since the manometer had to be connected to the system downstream of the condenser, and there was appreciable but only approximately known pressure drop in between, this was impractical.

In fact, during a run, the initial temperature of both the WF and the continuous aqueous phase changed slightly. The temperature of the WF would rather quickly drop until solid appeared, and then slowly drop thereafter as more solid formed. The shape of the temperature profile with time was the same shape as a cooling curve of the WF.

It was impossible to distinguish, by these measurements, the difference in temperature (real ΔT) between the actively boiling aqueous phase and the WF. The temperature of the WF must be somewhat higher than that of the boiling aqueous phase, or heat could not be transferred. An estimate that the difference in temperature (ΔT) between the WF and the aqueous phase of below one degree F seems very conservative in view of the fact that the only other temperature available to the process is the temperature of the undisturbed aqueous phase and that this was sometimes above and sometimes below the measured temperature of the boiling section without changing heat flux in a measurable amount. The pressure in the flasher also varied considerably without affecting heat flux.

PROCEDURE - SAMPLE CALCULATIONS - SOURCES OF ERROR - DATA SUMMARY

The problems in obtaining meaningful heat transfer data has been discussed above in broad general terms. The principles involved can be clarified by a specific example. In the pages that follow, the procedure for making a Flasher run, with specific sample calculations, and a discussion of sources of error, are presented. This is followed by a data summary of the Flasher runs.

NOTICE

The procedure which follows is an exact duplicate, including the sketch, of the procedure used to make these runs in the Bench Scale Plant - a stand-alone document. Thus, it contains information that is normally required in the ASTM type procedures, after which it is patterned. It also contains definitions of things already defined elsewhere in this report.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-86ID12619
BENCH SCALE PLANT PROCEDURES

FLASHER SIMULATION

PROCEDURE

Scope

This describes a procedure for determining the Heat Transfer Coefficient for the Flasher:

$$U\Delta T = \text{Btu/hr-ft}^3$$

Summary

These items must be measured for each determination:

1. Q (Btu) - Heat transferred during run
2. V (ft³) - Volume of WF before run (determination in a quiescent condition)
3. t (hours) - Time in which heat is transferred

These are combined in the equation for heat flux:

$$U\Delta T = \frac{Q}{Vt} = \frac{\text{Btu}}{\text{hr-ft}^3}$$

Determination of Q, V, and t

3. Q is determined in three different ways:
 - a. The amount of WF solidified is determined by completely freezing the WF. The amount of WF is determined by weighing it before charging it to the unit. The volume is also measured. The temperature versus time curve is observed during the course of the run. The run is concluded at the time when the temperature versus time curve begins to drop rapidly.
 - b. The temperature rise of the water in the melter/condenser.
 - c. The volume of condensate is measured by observing the increase in height of the water layer of the melter/condenser (The diameter is known). A correction (usually small) is applied for the rise in temperature of the water during the run.

2. V is determined by measuring the height of liquid working fluid at the start of a run (The experimental apparatus is of known diameter).
3. t is determined by the nearly flat portion of the temperature versus time of the cooling curve taken during a run.

Application

This method was developed specifically for its intended purpose-simulation of the flasher in the TEP. No effort has been made to search for other applications.

Definitions

TEP: Tubeless Evaporation Process

WF: Working Fluid

LWF: Liquid Working Fluid

Liquidus temperature: Temperature at which the last drop melts when the mixture is heated.

Apparatus

1. See sketch (following) for the experimental apparatus at the start of a run.
2. At the end of a run, the LWF is partially or completely solid, the concentrate layer is diminished, and there is additional water in the melter/condenser from the condensed steam. Also, some solids are on the walls of the vapor space.
3. Cartesian Diver Absolute Pressure Control with a range 1 mm Hg - 50 psi (not shown).

Procedure

1. During this entire procedure, record any unusual occurrences which are observed.
2. Remove the rubber stopper from the top of the flasher.
3. Fill the lower section of the flasher to a level of 8" above the top flange of the cross with previously prepared concentrate (of known composition) which has been heated to 5 - 10°F above the liquidus temperature of the WF selected for this run.

MELTER/CONDENSER

FLASHER

TUBELESS EVAPORATION PROCESS
FLASHER SIMULATION

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4. Weigh about 1200 gms of the selected WF in a 2 L stainless steel beaker, and heat it to 5 - 10°F above its liquidus temperature, and record the gross weight.
5. Charge the WF into the flasher, and record the gross weight of the beaker and any remaining WF.
6. Replace the rubber stopper in the top of the flasher.
7. Adjust the powerstats to maintain the concentrate and WF temperatures @ 3 - 7°F above the WF liquidus temperature.
8. Remove the rubber stopper from the top of the melter/condenser.
9. Fill the melter/condenser with water to a level near the bottom flange of the top straight section (but in a position where the level can be observed).
10. Replace the rubber stopper in the top of the melter/condenser.
11. At the start of the run, mark the initial levels of the concentrate, WF, and water.
12. Read and record the initial temperatures of the WF, concentrate, and the upper and lower water temperatures.
13. Set the absolute pressure control at a level corresponding to 1°F below the liquidus temperature.
14. Slowly apply vacuum to the system.
15. The following should be observed shortly after vacuum is applied:
 - a. Boiling will commence, at which time the concentrate begins to mix with the WF. This causes the WF section to expand to about twice its quiescent volume.
 - b. Bubbles begin to appear in the melter/condenser. This is air being expelled from the vapor section. When the air has been purged and only steam is entering, the bubbles are confined to the entrance section of the melter/condenser. The bubbles disappear within 1 or 2 inches of the point of entry.
16. As soon as boiling commences, begin reading the WF and concentrate temperatures at half-minute intervals and recording the results to the nearest estimated 0.2°F.

17. Read and record the vacuum to the nearest estimated 0.1" Hg as frequently as time permits.
18. Continue until the WF is completely solidified, as evidenced by a rapid drop in WF temperature.
19. Read and record the final temperatures of the WF, concentrate, and the upper and lower water temperatures.
20. Read and record the final vacuum.
21. Mark the final levels of the concentrate, WF, and water.
22. Release the vacuum slowly.

Calculation and Report

1. Heat transferred:

- a. From WF solidified

$$\begin{aligned}
 Q &= \text{solids} \times \text{heat of fusion} \\
 &= \text{solids} \times 35 \text{ (cal)} \\
 &= \text{solids} \times 35/252 \text{ (Btu)}
 \end{aligned}$$

- b. From temperature rise of the water in the melter/condenser

$$\begin{aligned}
 Q &= \text{water} \times \Delta T_{\text{water}} \text{ (cal)} \\
 &= \text{water} \times \Delta T_{\text{water}}/252 \text{ (Btu)}
 \end{aligned}$$

- c. From condensate collected

$$\begin{aligned}
 Q &= \text{condensate} \times 540 \text{ (cal)} \\
 &= \text{condensate} \times 540/252 \text{ (Btu)}
 \end{aligned}$$

2. The volume (V) of WF:

$$\begin{aligned}
 V &= \text{Area} \times \text{Height} \text{ (A} \times \text{h)} \\
 &= 0.7845 \times 4^2 h = 12.57h \text{ (in}^3\text{)} \\
 &= 12.57h/1728 \text{ (ft}^3\text{)}
 \end{aligned}$$

3. Time (t):

The temperature versus time profile is recorded for determination of actual time (hrs) of the experiment.

4. Calculate heat flux:

$$U_V \Delta T = \frac{Q}{Vt} \frac{\text{Btu}}{\text{hr-ft}^3}$$

SAMPLE CALCULATIONS

Date: August 26, 1986

Start of Run Conditions

Feed: 20% NaCl solution

WF: 1200 gms CW 155/159 paraffin

1580 ml = 96.4 in³

Height of WF = 7.67 in

Volume of WF = 96.4 in³ = 0.0558 ft³

Weight of water in the melter/Condenser: 1480 gms

Vacuum: 24" Hg (Condensing Temperature = 140°F)

End of Run Conditions

Level and Temperature Changes

1480 gms of water in the melter/condenser increased to 1580 gms, and the temperature rose from 23°C to 50°C.

Heat transferred:

a. From WF solidified

$$Q = \text{solids} \times \text{heat of fusion}$$

$$= 1200 \times 35 = 42,000 \text{ cal}$$

b. From temperature rise of the water in the melter/condenser

$$Q = \text{water} \times \Delta T_{\text{water}}$$

$$= 1580 \times 27 = 42,600 \text{ cal}$$

c. From condensate collected

$$Q = \text{condensate} \times 558 \text{ (cal)}$$

$$= 100 \times 558 = 55,800 \text{ cal}$$

Cooling Curve

Time	Temperature °F	
	WF	Bottom
0.00	163	156
0.25	157	156
1.00	156	155
2.00	156	154
2.50	154	154

Elapsed Time (t) = 2 min or 0.033 hrs

Calculate heat flux:

$$\text{Taking } Q = 42,000 \text{ cal} = 167 \text{ Btu}$$

$$U_V \Delta T = \frac{Q}{Vt} = \frac{167}{.0558(.033)} = 90,100 \frac{\text{Btu}}{\text{hr-ft}^3}$$

SOURCES OF ERROR

A discussion of the sources of error in the measurement of heat flux, time, and volume is presented below:

Heat Flux

As discussed above, there are three methods of estimating heat flux from the data taken during a run:

From WF Solidified:

The heat of fusion (35 cal/gm) was not measured. The value was taken from the literature. It could very well be that the value is as low as 33, or as high as 40.

Some of the working fluid may not have solidified.

There were some sensible heat effects (e.g. cooling the liquid working fluid to the freezing point, and cooling the solid working fluid) that could have added 5 cal/gm to the heat of fusion.

Some liquid or solid working fluid may have been entrained in the vapor leaving the working fluid zone.

Taking all of these sources of error into account, the value of Q (the heat flux) could range from 40,000 to 47,000 calories.

From condensate collected:

The amount of condensate collected was measured by marking the 2 in diameter melter/condenser at the beginning of the run, and noting the rise at the end of the run. The amount was taken as 100 cc, but it could have been as low as 90 cc, or as high as 110 cc.

Some of the condensate could have been entrainment

Some of the steam could have escaped without being condensed (This is unlikely).

Based on these uncertainties, the value of Q could range from 50,250 to 61,300 calories.

From Temperature Rise (of the water in the melter/condenser), Q would be 42,600 calories, which is in the middle of the range based on working fluid solidified.

Time

Time cannot be less than 1.5 minutes, nor more than 3 minutes. The time required for boiling to begin was so brief that it could not be measured accurately, but it was estimated to be between 1.5 and 2 minutes. The total time for the run was three minutes. Therefore, the most probable range is 1.5 to 2 minutes.

Volume

the volume is known more accurately than any other variable in the equation.

Summary

$$Q = 40,000 - 61,300 \text{ cal or } 159 - 243 \text{ Btu}$$

$$V = 0.0558 \text{ ft}^3$$

$$t = 0.025 - 0.033 \text{ hrs}$$

Worst Case:

$$U_V \Delta T = \frac{Q}{Vt} = \frac{159}{.0558(.033)} = 86,350 \frac{\text{Btu}}{\text{hr-ft}^3}$$

Best Case:

$$U_V \Delta T = \frac{Q}{Vt} = \frac{243}{.0558(.025)} = 174,200 \frac{\text{Btu}}{\text{hr-ft}^3}$$

DATA SUMMARY

After tune-up operation was complete, a total of six runs (including the "sample" run described above) were made on the Flasher. The data from these runs are summarized below:

<u>Feed</u> <u>(Viscosity)</u> <u>(cp)</u>	<u>Temperature</u> <u>°F</u>	<u>Working Fluid</u>	<u>U_V min</u>	<u>U_V max</u>
20% NaCl (1)	156	Chevron CW 155	89,900	174,200
20% NaOH (1)	155	Chevron CW 155	62,000	187,000
Pickling Liquor (1)	156	Chevron CW 155	38,700	270,000
18 Brix Sugar (1)	185	Epolene N-10	75,600	111,000

<u>Feed (Viscosity) (cp)</u>	<u>Temperature °F</u>	<u>Working Fluid</u>	<u>U_v min</u>	<u>U_v max</u>
57 Brix Sugar (4)	176	Epolene N-10	38,700	270,000
70 Brix Sugar (30)	156	Chevron CW 155	17,011	62,300

It can be seen that when the viscosity of the feed is about 1 cp, or lower, that the heat transfer rate is very high. Of course, there is a viscosity penalty in evaporators that employ tubes, too. The heat transfer coefficient in a typical fourth effect (46 brix) is less than half of what it is in the third effect (34 brix). The 70 brix case must be regarded as extreme. In any analysis, the U_v of 80,000, which was used in the **TEP ECONOMIC EVALUATION** of this report, must be regarded as extremely conservative for feeds with a viscosity below 4 centipoise.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
BSP PROGRAM

MELTER CONDENSER RUNS

Just as with the Flasher, the primary information desired from the operation of the Melter/Condenser was the heat transfer coefficient. And, once again, the heat transfer coefficient sought would be the heat flux divided by the ΔT , as expressed by the equation:

$$U_v \Delta T = \frac{Q}{tv}$$

Q is obtained from the known amount of WF melted.

v is taken as the volume of the melter/condenser which contains solid below the air/water interface.

t is the time from the admission of steam until the WF is liquid.

PROCEDURE - SAMPLE CALCULATIONS - SOURCES OF ERROR - DATA SUMMARY

In the pages that follow, the procedure for making a Melter/Condenser run, with specific sample calculations, and a discussion of sources of error, are presented. This is followed by a data summary of the Melter/Condenser runs.

NOTICE

The procedure which follows is an exact duplicate, including the sketch, of the procedure used to make these runs in the Bench Scale Plant - a stand-alone document. Thus, it contains information that is normally required in the ASTM type procedures, after which it is patterned. It also contains definitions of things already defined elsewhere in this report.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-86ID12619
BENCH SCALE PLANT PROCEDURES

MELTER/CONDENSER SIMULATION

PROCEDURE

Scope

This describes a procedure for determining the Heat Transfer Coefficient for the Melter/Condenser:

$$U\Delta T = \text{Btu/hr-ft}^3$$

Summary

These items must be measured for each determination:

1. Q (Btu) - Heat transferred during run
2. V (ft³) - Volume of WF before run
3. t (hours) - Time in which heat is transferred

These are combined in the equation for heat flux:

$$U\Delta T = \frac{Q}{Vt} = \frac{\text{Btu}}{\text{hr-ft}^3}$$

Determination of Q, V, and t

1. Q is obtained from the known amount of WF melted.
2. V is taken as the volume of the melter/condenser which contains solid below the air/water interface.
3. t is the time from the admission of steam until the WF is liquid.

Application

This method was developed specifically for its intended purpose-simulation of the melter/condenser in the TEP. No effort has been made to search for other applications.

Definitions

TEP: Tubeless Evaporation Process

WF: Working Fluid

LWF: Liquid Working Fluid

Liquidus temperature: Temperature at which the last drop melts when the mixture is heated.

Solidus temperature: Temperature at which the last drop freezes when the mixture is cooled.

Apparatus

1. See sketch (following) of the melter/condenser and boiler.
2. Cartesian Diver Absolute Pressure Control with a range 1 mm Hg - 50 psi (not shown).

Procedure

1. During this entire procedure, record any unusual occurrences which are observed.
2. Remove the rubber stopper from the top of the melter/condenser.
3. Fill the melter/condenser with water which has been heated to 5 - 10°F below the solidus temperature of the WF selected for this run to a level near the bottom flange of the top straight section (but in a position where the level can be observed).
4. Weigh about 230 gms of the selected WF in a 1 L stainless steel beaker, and record the gross weight.
5. Charge the WF into the melter/condenser. This should produce a WF regimen of about 6" in height - half above and half below the water.
6. Record the gross weight of the beaker and any remaining WF.
7. Replace the rubber stopper in the top of the melter/condenser.
8. Adjust the powerstats to maintain the water temperatures @ 3 - 7°F below the WF solidus temperature.
9. Preheat about 3,500 ml of water in an 8 L stainless steel beaker to 3 - 7°F below the WF solidus temperature.

**TUBELESS EVAPORATION PROCESS
MELTER/CONDENSER SIMULATION**

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10. Charge the water into the bottom of the boiler through a vacuum hose, and applying a vacuum to the top outlet.
11. Adjust the powerstat to maintain the water temperatures @ 3 - 7°F below the WF solidus temperature.
12. At the start of the run, record the depth of WF below the air/water interface.
13. Read and record the initial upper and lower water temperatures.
14. Set the absolute pressure control at a level corresponding to 1°F above the liquidus temperature.
15. Slowly apply vacuum to the system.
16. Admit steam to the melter/condenser at a rate sufficiently low that no condensate is produced in the trap downstream of the melter/condenser.
17. As soon as steam is admitted, begin reading the melter/condenser upper and lower temperatures and, the boiler temperature at one minute intervals and recording the results to the nearest estimated 0.1°C.
18. Read and record the absolute pressure to the nearest estimated 0.1" Hg at one minute intervals.
19. Continue until the WF is completely liquid.
20. Read and record the final melter/condenser upper and lower water temperatures, and the boiler temperature.
21. Read and record the final vacuum.
22. Release the vacuum slowly.

Calculation and Report

1. Heat transferred - From WF solidified:

$$\begin{aligned} Q &= \text{solids} \times \text{heat of fusion} \\ &= \text{solids} \times 35 \text{ (cal)} \\ &= \text{solids} \times 35/252 \text{ (Btu)} \end{aligned}$$

2. The volume (V) of WF:

$$V = \text{Area} \times \text{Height} \text{ (A} \times \text{h)}$$

$$= 0.7845 \times 2^2h = 3.14h \text{ (in}^3\text{)}$$

$$= 3.14h/1728 \text{ (ft}^3\text{)}$$

3. Time (t):

The temperature versus time profile is recorded for determination of actual time (hrs) of the experiment.

4. Calculate heat flux:

$$U_V \Delta T = \frac{Q}{Vt} \frac{\text{Btu}}{\text{hr-ft}^3}$$

SAMPLE CALCULATIONS

Date: July 31, 1986

Start of Run Conditions

WF: 227 gms - 67% hexatriacontane-33% hexadecane

Height of WF = 3 in

Volume of WF = $9.42 \text{ in}^3 = .00545 \text{ ft}^3$

Vacuum: 18.4" Hg

End of Run Conditions

Vacuum: 17.9" Hg

Heat transferred:

$$Q = \text{solids} \times \text{heat of fusion}$$

$$= 227 \times 35 = 7,945 \text{ cal or } 31.53 \text{ Btu}$$

Time	Temperature			Melter/Condenser		
	Melter/Condenser		Boiler °C	Pressure " Hg	Temperature*	
	Top °C	Bottom °C			°F	°C
2:07	75	75	80	11.5	167.4	75.2
2:08	76	76	79	12.0	169.3	76.2

*Calculated from pressure

Elapsed Time (t) = 1 min or 0.0166 hrs

Calculate heat flux:

$$U_V \Delta T = \frac{Q}{Vt} = \frac{31.53}{.00545(.0166)} = 348,513 \frac{\text{Btu}}{\text{hr-ft}^3}$$

SOURCES OF ERROR

DOE CONTRACT No. DE-AC7-86D12549

This problem proved very measurable. Note the very close agreement between measured temperature and theoretical temperature from pressure. The temperature of the boiler indicates a boiler pressure of 13.4 - 14" Hg (There was a water layer of 23 inches between the vapor entrance from the boiler to the liquid interface. This amounts to 1.77" Hg). The only difficulty was that the heat transfer rate was high that the measurement of time was inaccurate. The time was taken as one minute. It could hardly have been more. It could have been less. There was a very high heat transfer rate. It should have been reported. All other known errors would be very small.

DATA SUMMARY

After tune-up operation was complete, a total of four runs (including the "sample" run described above) were made on the Melter/Condenser. Runs were made on hexatriacontane/heptadecane (HTC/HD) mixtures, and Chevron CW 155. The results were the same for both materials within the range of accuracy of the experiments. The data from these runs are summarized below:

Working Fluid	Temperature	Pressure	Time
33%HTC-67%HD	1.69	12.00	348,500
33%HTC-67%HD	1.48	7.20	178,000
33%HTC-67%HD	1.51	7.00	352,000
Chevron CW 155	1.70	2.20	178,000

to interfere with obtaining a representative sample. et cetera - are occurring right at shutdown. All of these tend system to another, reflux running down the walls of the Flasher, pressure, which can cause material to surge from one part of the Unfortunately, however, several transients - rapid change in separation occurs completely, if sufficient time is allowed). quickly the separation occurs (It is already known that the after shutdown. After all, it is important to establish how It is apparently desirable to take the sample as soon as possible

PROCEDURE - DATA SUMMARY AND DISCUSSION OF RESULTS

In the pages that follow, the procedure for making phase separation runs is presented. This is followed by a data summary and a discussion of results.

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-861D12619

BSP PROGRAM

PHASE SEPARATION

In addition to achieving the primary goal of obtaining heat transfer data from the Flasher runs, it was also hoped to observe the speed and completeness of phase separation. To a limited extent, this proved possible, but not without some problems. First of all (for reasons cited under **THE MEASUREMENT OF HEAT TRANSFERRED in FLASHER RUNS**), Flasher runs are continued until all of the working fluid is solidified, thereby making it impossible to take a sample of the working fluid at the end of the Flasher run. Thus, it became necessary to make special (not for obtaining heat transfer data) Flasher runs in order to obtain samples for entrainment tests. This was done, but again, not without difficulty.

Conditions in the Flasher are significantly and unfavorably different than those that would prevail in the Phase Separator, particularly in the working fluid zone. While the concentrate zone is relatively quiet (and, hence may be free of working fluid, even though this section is very short), the working fluid zone is extremely turbulent due to the evolution of vapor. Of course this is a good thing - essential, actually - for the operation of the Flasher. But, it is death to phase separation. Compare this to the Phase Separator, where the velocity of the working fluid is limited to 1 ft/min. Obviously, any sample taken from the Flasher during operation (no provision was made to do this) would be non-representative of Phase Separator performance. There are problems with a sample taken immediately after shutdown, too.

It is apparently desirable to take the sample as soon as possible after shutdown. After all, it is important to establish how quickly the separation occurs (It is already known that the separation occurs completely, if sufficient time is allowed). Unfortunately, however, several transients - rapid change in pressure, which can cause material to surge from one part of the system to another, reflux running down the walls of the Flasher, et cetera - are occurring right at shutdown. All of these tend to interfere with obtaining a representative sample.

PROCEDURE - DATA SUMMARY AND DISCUSSION OF RESULTS

In the pages that follow, the procedure for making Phase Separation runs is presented. This is followed by a data summary and a discussion of results.

NOTICE

The procedure which follows is an exact duplicate of the procedure used to make these runs in the Bench Scale Plant - a stand-alone document. Thus, it contains information that is normally required in the ASTM type procedures, after which it is patterned. It also contains definitions of things already defined elsewhere in this report.

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-86ID12619

PROCEDURE

The procedure which would be used to make these tests at the Bench Scale Plant - a stand-alone standard - is normally required in the ASTM type procedures, after which it is patterned. It also contains definitions of things already defined elsewhere in this report.

Scope

This describes a procedure for determining entrainment in working fluids (WF's).

Summary

The solubility of working fluids used in the TEP in water is so low (parts per trillion range) that it cannot be measured by ordinary laboratory means. However, **entrainment** is possible under certain conditions (misoperation). In this procedure, the amount of entrainment (if any) is determined by allowing a sample of the working fluid to settle in a centrifuge tube.

Application

This method was developed specifically for its intended purpose - determination of entrainment in working fluids. No effort has been made to search for other applications.

Definitions

Percent Entrainment: cc of water per 100 cc of sample.

Liquidus temperature: Temperature at which the last drop melts when the mixture is heated.

Apparatus

The apparatus consists of the following:

100 cc centrifuge

Note: The centrifuge tube is used because its tapered shape permits detection of less than 0.01 cc of water in a 100 cc sample (It is not centrifuged).

Procedure

1. During this entire procedure, record any unusual occurrences which are observed.

2. Remove the rubber stopper from the top of the flasher and insert the centrifuge tube into the flasher as soon as possible after the completion of a run on the flasher. The flasher run which immediately precedes this procedure must be terminated prior to freezing all of the water in order to be able to take a sample which cannot be "permanently" suspended.

3. Withdraw the sample immediately. The apparent conclusion is that the level of dissolved solids of water is not significantly different from that of the water in the flasher. However, this is not really conclusive proof. This can only be provided by a minimum of 5 minutes of settling for a sample. In the opinion of the Authors, there is no question that the quantity of water and the quantity of sample in the centrifuge tube needs to be determined. Details of the Phase Separators.

7. Repeat step 6 after 24 hours.

Report

1. Percent entrainment after 5 minutes.
2. Percent entrainment after 24 hours.

DATA SUMMARY AND DISCUSSION OF RESULTS

After tune-up operation was complete, two runs were made in which satisfactory samples of the working fluid layer were obtained. No water was detected in either sample. As mentioned above, as little as 0.01 cc of water in 100 cc of working fluid would have been detected, provided that sufficient settling time was allowed, as provided for by the Procedure. The droplets cannot be "permanently" suspended.

The apparent conclusion is that the level of dissolved solids of the product water would be satisfactory for potable water (assuming that the solids themselves were edible). However, this is not really conclusive proof. This can only be provided by continuous operation of a Phase Separator, as will be done in the Pilot Plant. In the opinion of the Authors, there is no question that a satisfactory separation can be made. The only thing that needs to be determined in the Pilot Plant is the size and design details of the Phase Separators.

THE UNIVERSITY OF TEXAS AT AUSTIN
THE INSTITUTE FOR ENERGY STUDIES
1980-1981
AUSTIN, TEXAS

11/11/80

Dear Mr. [Name]:

Thank you for your letter of [Date].

I am sorry that I cannot [Action].

I am sure that you will [Action].

I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].
I am sure that you will [Action].

TEP ECONOMIC EVALUATION

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
TEP ECONOMIC EVALUATION

OVERALL

When compared to present commercial practice, estimated savings for the TEP are as follows:

Energy Savings	50 - 75%
Capital Savings	45%
Maintenance Cost Savings	45%

The most significant of the savings is in energy. Because the TEP does not require any ΔT to drive heat through tubes, it can easily fit 20 effects (four effects of one-quarter the capacity for each conventional effect - as illustrated above) into the available temperature range now used up by 5 effects. This would mean a 75 percent savings in energy requirements!!! This is explained in more detail in the following section entitled "ENERGY SAVINGS".

TEP ECONOMIC EVALUATION

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC67-86D12619
TEP ECONOMIC EVALUATION
ENERGY SAVINGS

Since the TEP is a function of concentration and temperature only, and not of the process used, it can be expected to average less than 16.5° per effect, because more water will be removed at the lower BPR's. But, for the sake of simplicity, one degree assumed to have a profile like the five effect plant: one degree, The Tubeless Evaporation Process (TEP) can save 50-75 percent (depending on the nature of the feed) of the energy requirements of conventional evaporators. It has been tested successfully on sugar, salt, and caustic soda solutions; sulfuric and nitric acids; "spent" pickling liquor, and cooling tower blowdown. The TEP has potential application to many other industries - nearly any industry that performs evaporation or concentration of aqueous streams.

	°F	°F	°F
Steam Temperature	274.0	251.0	154.0
First Effect Liquor Temp.	251.0	154.0	147.0
Last Effect Liquor Temp.	154.0	147.0	
Last Effect Vapor Temp.	147.0		

The primary reason for energy savings in the TEP is its use of direct contact heat exchange. Therefore, there is no need for ΔT to drive heat through tubes. This means that more effects can be installed within the temperature range available for heat transfer. Furthermore, because there are no tubes to scale or catalyze undesired reactions, it would be possible in some applications to expand this range. This would allow even more effects to be installed, thereby further reducing energy requirements.

Black Liquor Concentration

Sugar Concentration

The beginning and desired ending composition and concentration vary from plant to plant, but an initial concentration is normally performed in a triple or quadruple effect evaporator. If vapor is bled for preheating or vacuum pans, a fifth "pre-evaporator" effect is added as the first effect. A net performance ratio (PR) of about 5 lbs of water evaporated/1000 btu of heat supplied is realized. Typically, steam is supplied to the first effect at 274°F. The first effect liquor temperature is 251°F and the last effect liquor temperature is 154°F, with a vapor temperature of 147°F. The total temperature difference (274-147) of 127°F is comprised of 16° of boiling point rise (BPR) and 111° of ΔT to drive the heat through the tubes, an average of about 22°F per effect. The top liquor temperature could be limited by the heat sensitivity of the sugar. Brief laboratory experience indicates that a liquor exit temperature of 140°F (vapor at 97°F) could be possible with the TEP, but for the purpose of this discussion 154°F will be assumed. Because of the need for high ΔT to drive the heat through the tubes, conventional evaporators are severely limited in the number of effects that are possible by the available ΔT . But, since the TEP requires only about 2° per effect for heat transfer, the number of effects can be greatly

expanded. An optimization of capital cost versus energy savings would have to be performed to determine the number of effects, but when it is considered that the TEP is comprised primarily of empty vessels, surely at least 20 effects could be justified. Since the BPR is a function of concentration and temperature only, and not of the process used, it can be expected to average less than $16/5^\circ$ per effect, because more water will be removed at the lower BPR's. But, for the sake of simplicity, it will be assumed to have a profile like the five effect plant: one degree BPR in the first four effects, two in the next eight effects, four in the next four effects, and seven in the last four effects, for a total of 64 degrees. With these assumptions, a comparison of the two plants would look like:

	<u>Conventional</u>	<u>TEP</u>
Steam Temperature - $^\circ\text{F}$	274.0	253.0
First Effect Liquor Temp. - $^\circ\text{F}$	251.0	251.0
Last Effect Liquor Temp. - $^\circ\text{F}$	154.0	154.0
Last Effect Vapor Temp. - $^\circ\text{F}$	147.0	147.0
Total BPR - $^\circ\text{F}$	16.0	64.0
Delta-T Available for Heat Transfer	111.0	42.0
Number of Effects	5	20
Average Delta-T per Effect	22.2	2.1

The TEP would have a PR of about 19 compared to 5 for the conventional plant, a savings of at least 75 percent.

Black Liquor Concentration

The beginning and desired ending compositions in black liquor concentration vary from plant to plant, but an initial concentration of 15% solids and final concentration of 50% solids can be regarded as typical. At about 30% solids, the organics begin to become insoluble and the "skimming" operation begins. What is proposed here is that the TEP be used in place of the conventional evaporators up to a concentration of 30% solids. After the skimming operation is completed, the final desired concentration of 50% solids would be achieved in conventional evaporators.

Energy Savings. Analysis of the potential for improvement by using the TEP is quite similar to that for sugar. Steam is supplied at 249°F , and the vapor leaves the last effect at 125°F . For soda process black liquor, the BPR is moderate. If a 15 effect TEP is assumed the two plants would look like this:

	<u>Conventional</u>	<u>TEP</u>
Steam Temperature - $^\circ\text{F}$	249	249
First Effect Liquor Temp. - $^\circ\text{F}$	232	247
Last Effect Liquor Temp. - $^\circ\text{F}$	130	130

	<u>Conventional</u>	<u>TEP</u>
Last Effect Vapor Temp. - °F	125	125
Total BPR - °F	31	93
Delta-T Available for Heat Transfer	93	31
Number of Effects	5	15
Average Delta-T per Effect	18.6	2.1

The TEP would have a PR of about 14, compared to 4.3 for the conventional plant, a savings of nearly 70 percent. Since concentration from 30% solids to 50% solids would be unaffected the overall savings would be about 50 percent.

Caustic Soda Concentration

The previous examples have shown that the ability of the TEP (or any other direct contact heat exchange process) to save energy in evaporators is much greater when the BPR is low. This is because the energy required is proportional to the total ΔT , comprised of the BPR and the ΔT for the process selected. But, even when the BPR is very high, some energy can be saved, and in some instances the other advantages of eliminating tubes become important. The concentration of caustic soda is a good example, as will be discussed in the section on "OTHER BENEFITS".

Energy Savings. In this typical case (ref. 3) the BPR for the solution is so high that it totals 103°F for only two effects. This left only 56°F for heat transfer which had to be divided between the two effects. A third effect could have been added, but that would have made the ΔT available for heat transfer even smaller in all three effects, which goes against the expressed importance." Because of the low ΔT required for heat transfer by the TEP, at least six effect could be put in with the following results:

	<u>Conventional</u>	<u>TEP</u>
Steam Temperature - °F	274	261
First Effect Liquor Temp. - °F	246	260
Last Effect Liquor Temp. - °F	141	121
Last Effect Vapor Temp. - °F	115	115
Total BPR - °F	103	134
Delta-T Available for Heat Transfer	56	12
Number of Effects	2	6
Average Delta-T per Effect	28	2

The PR of the conventional plant was estimated at 1.73. The TEP should be at least 5, for a savings of about 65 percent.

Brine Concentration (Salt Industry)

Some salt is mined as rock salt. The remainder is obtained by evaporating natural brines or artificial brines (created by pumping water into a mine and returning saturated brine), or seawater. In warm climates, solar evaporation is used. The rest is evaporated in conventional single or triple effect evaporators. According to the Bureau of Mines (ref. 4), during 1979, of the 46.3×10^6 tons of salt produced, 32 percent came from rock salt, 5 percent from solar evaporation, and the remainder, 29.2×10^6 tons, from brine. The overall savings would be about 50 percent.

About 2.7 pounds of water must be evaporated for each pound of salt produced. If a PR of 2 is taken as the average for the industry 2.7×10^6 btu/ton is required. The TEP should be capable of achieving a PR of at least 8, for savings of 75 percent.

WHY DIRECT CONTACT SAVES SO MUCH ENERGY
The energy required in process is much greater when the BPR is low. This is because the BPR and the AT for the process selected. But, even when the

The foregoing examples all involved multi-effect evaporators. The advantage of the lower AT of the TEP could be seen in the fact that so many more effects can be packed into the same temperature range, thus providing the opportunity to reuse the same energy several times. This section looks at the theoretical reason for the TEP being so energy efficient from another aspect, that of the single effect plant. The solution is so high that it totals 103. This left only 56°F for heat transfer which had to be divided between the two effects. A third effect would have made the AT available for heat transfer even

THE TEP AS A CONCENTRATOR
The Tubeless Evaporation Process (TEP) was originally envisioned as a more energy efficient and economic process than any of today's commercial or emerging processes to produce fresh water from seawater, and other saline waters. However, it soon became apparent that the widest application of the process by far would be as a concentrator, because of the rapidly increasing interest in concentrators. At first, it was thought of as a brine concentrator (a concentrator of saline waters) which could be applied effectively to such streams as cooling tower blowdown. Upon further thought, it became obvious that the TEP was applicable to practically any situation where evaporators are used. In the examples that follow, the TEP is compared to the two most important types of advanced concentrators (other than the TEP): the RCC (type) process (the most prominent of this advanced type concentrator), and the eutectic freezing process. Seawater is used in these examples, because so much data was already available, but the results are directly transferable to the other evaporation applications. Of course, the results would have to be adjusted for a different boiling point rise, which would affect the percentage of energy saved, as has been done for the sugar, pulp and paper, and caustic soda industries, in the examples given above.

The RCC process would be developed for the BPR would be the same amount for both processes (for other conditions of the Resource Conservation Company (RCC) in Renton, Washington, has developed a process specifically intended to meet the need for a low cost concentrator. The process uses vapor recompression to save energy, and seeding techniques to control scaling. While this method permits satisfactory operation, scaling is not entirely eliminated. This process has been highly successful, but, the presence of tubes would make it difficult to concentrate to moist solids. This is a significant drawback for the RCC process. While the ability of the TEP to handle this problem was not demonstrated during this project, the fact that all of the selected working fluids are lighter than water and all of the solids are considerably heavier, one would not expect any phase separation problems. These assumptions need to be still lower. The principal In addition, the TEP will have substantially lower capital and operating costs, as well as energy consumption.

Energy Consumption: RCC vs. TEP. For processes of the type under consideration, the RCC process and the TEP, the theoretical energy requirement for heat pumping work is approximately proportional to the total ΔT . Part of this ΔT is due to boiling point rise (BPR). Since the BPR is independent of the process, the requirement would be the same for both the RCC process and the TEP. However, in addition to this, the RCC process requires ΔT to drive heat through the tubes. The normal design value for this purpose is 5°C . Although the TEP has no tubes, an allowance must be made for the melting range of the working substance and for mixing. This allowance is estimated to be 0.5°C . At 100°C , it can be shown (ref. 5) that the following equation is substantially correct for the theoretical heat pumping work (kilowatt hours per kilogram).

$$\text{Energy Consumption - Freezing} = \frac{6.34 \times \Delta T}{\text{kgal}} \quad \text{For two times concentrated seawater, the BPR is about } 1^\circ\text{C}.$$

For two-times concentrated seawater, the BPR is about 1°C . Theoretical work requirements for the two processes would be:

where the total ΔT is comprised of the ΔT due to freezing depression (FPD), instead of BPR, and the ΔT for the process. In this case, the coefficient of a freezing process do have the advantage of a much lower latent heat, which is reflected in the coefficient of 1.40 in the work equation for freezing processes, compared to 6.34 for evaporation processes. However, at equivalent drive conditions, the BPR is about 4.2°C . The theoretical work

$$\text{RCC} - \text{TEP} = 38.02 - 9.51 = 28.51 \frac{\text{kw-hr}}{\text{kgal}}$$

While the energy requirement for the BPR would be different (by the same amount for both processes) for other conditions of temperature concentration, the substantial difference in the process energy requirements, about 29 kw-hr/kgal, would remain.

The Eutectic Freezing Process

The RCC process represents a significant advance. However, it appears that the freezing process could have still more favorable economics, and lower energy requirement. A comparison of the flow sheets and operating conditions would lead one to expect lower capital cost for the eutectic freezing process. And, while vapor recompression is a great energy saver when compared to other distillation processes, the freezing process requirements should be still lower. These assumptions needed to be confirmed and quantified. Such a study (ref. 5) was made. The principal conclusion drawn from this work was that once they have been developed and demonstrated, freezing processes will be superior concentrators. Also, they will become more attractive from both the energy and concentration cost viewpoints as the desired concentration of final effluent increases. This is particularly evident when the desired final effluent is a solid.

These findings were reaffirmed in a later study (ref. 6). Good as these study results are, the TEP has the potential to do better. As will be shown below, the TEP will only be slightly better than the freezing process in the single effect version, because it is being compared to another direct contact process - the secondary refrigerant version of the freezing process (As will be explained below, it could be significantly better in the multieffect version). But, the TEP also offers the possibility of substantially lower capital and operating costs, simplicity, and ease of operation.

Energy Consumption - Freezing Process vs. TEP. For the freezing process the appropriate equation (ref. 5) is:

$$W = 1.40 \times \Delta T \text{ total kw-hr/kgal}$$

where the total ΔT is comprised of the ΔT due to freezing point depression (FPD), instead of BPR, and the ΔT for the process. In the studies discussed above (refs. 5 & 6) the ΔT for the process was taken as 5°C. Freezing processes do have the advantage of a much lower latent heat, which is reflected in the coefficient of 1.40 in the work equation for freezing processes, compared to 6.34 for evaporation processes. However, at equivalent brine concentrations, the FPD is approximately 3.5 times the BPR. For two-times concentrated seawater, the FPD is about 4.2°C. The theoretical work

requirement for freezing process would be:

$$W = \frac{1.40 \text{ kw-hr/kgal}}{^{\circ}\text{C}} \times (4.2^{\circ}\text{C FPD} + 5^{\circ}\text{C PROC.}) = 12.88 \frac{\text{kw-hr}}{\text{kgal}}$$

compared to 9.51 kw-hr/kgal for the TEP, a difference of 3.37 kw-hr/kgal.

It should be noted that the 5°C driving force for the freezing process was based on using a secondary refrigerant process, which was being piloted at the time that the studies were made (Thus, the TEP and the secondary refrigerant process were both unproven in their ability to concentrate to moist solids). This work was not entirely successful. A potential replacement is the indirect freezing process. Unlike the secondary refrigerant process, this process employs tubes in the freezer. Presumably, additional ΔT will be required to drive heat through these tubes, thus increasing the energy requirement.

Furthermore, the multieffect version of the TEP can be run on heat instead of electricity (which throws away about two-thirds of the energy in the fuel while it is being generated).

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619

TEP ECONOMIC EVALUATION

CAPITAL AND MAINTENANCE COST SAVINGS
 compared to 2.25% of 3.37 kW-hr/kgal. = 0.76 kW-hr/kgal. x (4.2% PROC.) = 12.88 kW-hr/kgal.

DESIGN BASIS

PRESENT COMMERCIAL PRACTICE

The basis for comparison is information provided by the Amalgamated Sugar Company on one of their existing evaporators. The costs for auxiliaries, such as piping, instruments, and controls, should be somewhat less for the TEP, because of its simplicity, but the effect would be small. The third effect was used for these calculations because it is the middle effect, and is about average for the train in overall U, area, absolute pressure, and concentration (see Exhibit 1, ASC).

The existing third effect consists of two vessels operating in parallel:

Effect 3A - 14 ft Dia x 32.5 ft - 9,352 ft²

4,120 - 1-1/4" x 7'-3" tubes

Effect 3B - 14 ft Dia x 32.5 ft - 9,928 ft²

4,903 - 1-1/4" x 8'-4" tubes

Current cost for replacement with 304 S.S. tubes and tubesheets (in a carbon steel shell) is about \$15.00/ft² or about \$290,000.

MINI-CASSIA EVAPORATORS

1A & 1B EFFECTS - 6,850 TUBES $1\frac{1}{4}"$ O.D. 0.084" WALL 8'-4 $\frac{1}{4}"$ LONG
TUBESHEET $\frac{3}{4}"$

AREA INSIDE OF TUBES $A_i = 15,938 \text{ ft}^2$ each
(SUBTRACTING TUBESHEET THICKNESS)

2A - 2,640 TUBES $1\frac{1}{4}"$ O.D. 14 ga. 9'-0 $\frac{3}{8}"$ TUBESHEETS 1"
2,028 " 2" O.D. 14 ga. 9'-0 $\frac{3}{8}"$
12 " 2" O.D. 14 ga. 9'-0 $\frac{3}{8}"$
66 " 2" O.D. 4 ga. 9'-0 $\frac{3}{8}"$

$$\underline{\underline{A_i = 15,272 \text{ ft}^2}}$$

2B - 8,172 TUBES $1\frac{1}{4}"$ O.D. 9'-8 $\frac{1}{4}"$ LONG 0.084" WALL TUBESHEET $\frac{3}{4}"$

$$\underline{\underline{A_i = 22,144 \text{ ft}^2}}$$

3A - 4,120 TUBES $1\frac{1}{4}"$ O.D. 15 ga. 8'-0 $\frac{1}{4}"$ TUBESHEET 1"

$$\underline{\underline{A_i = 9,352 \text{ ft}^2}}$$

3B - 4,903 TUBES $1\frac{1}{4}"$ O.D. 7'-3 $\frac{1}{4}"$ 0.084" WALL TUBESHEET $\frac{3}{4}"$

$$\underline{\underline{A_i = 9,928 \text{ ft}^2}}$$

4A - 4,120 TUBES $1\frac{1}{4}"$ O.D. 6'-0 $\frac{1}{4}"$ 14 ga. TUBESHEET 1"

$$\underline{\underline{A_i = 6,842 \text{ ft}^2}}$$

4B - 4,000 TUBES $1\frac{1}{4}"$ O.D. 3'-6 $\frac{1}{4}"$ 0.084" TUBESHEET $\frac{3}{4}"$

$$\underline{\underline{A_i = 3,848 \text{ ft}^2}}$$

5A - 4,120 TUBES $1\frac{1}{4}"$ O.D. 6'-0" 0.083" TUBESHEET 1"

$$\underline{\underline{A_i = 6,820 \text{ ft}^2}}$$

5B - 4,160 TUBES $1\frac{1}{4}"$ O.D. 6'-0" 0.083" TUBESHEET $\frac{3}{4}"$

$$\underline{\underline{A_i = 6,933 \text{ ft}^2}}$$

EXHAUST - 27.8 PSIG

1st VAPOR - 19.4 #

2nd VAPOR - 12.1 #

3rd VAPOR - 3.2 #

4th VAPOR - 8.1" Hg GAUGE

5th VAPOR - 20.9" Hg GAUGE

EVAPORATOR SHELL & WELL DATA

EVAP	A	B	C	D
A1	14' 0"	32' 6"	3' 0"	8' 4"
A2	14' 0"	33' 4"	3' 0"	8' 4"
A3	11' 0"	27' 10"	3' 0"	9' 0"
A4	11' 0"	24' 6"	2' 10"	6' 0"
A5	11' 0"	24' 6"	2' 10"	4' 3"
B1	14' 0"	32' 6"	3' 0"	7' 5"
B2	15' 0"	34' 4"	3' 0"	9' 0"
B3	12' 0"	29' 3"	3' 0"	7' 3"
B4	11' 0"	24' 0"	2' 11"	3' 6"
B5	11' 0"	24' 6"	2' 10"	7' 0"

EVAPORATOR SHELL & WELL DATA	SCALE	NTS	DATE	FACTORY
	CHECKED BY	17	FLA B7	MINI-CASSIA
		UWL NO	ECM	5425/5207
				25 45

THE TEP REPLACEMENT

The TEP replacement for this would be four effects in series.
Each effect would consist of:

Flasher - three 4 ft Dia x 24 ft vessels, containing about
3,500 # of WF (@ \$0.30/#)

Melter/Condenser - three 4 ft Dia x 24 ft vessels.
containing about 1,550 # of WF

WF Pump - 3,000 gpm @ 10 ft

Estimated Cost per Effect:

6 - 4 ft Dia x 24 ft vessels @ \$5,000 =	\$30,000
5,000 # of WF @ \$0.30/#	5,000
1 - 3,000 gpm x 10 ft pump	<u>5,000</u>
Subtotal	\$40,000

Total Estimated Replacement Cost:

Four Effects @ \$40,000	\$160,000
-------------------------	-----------

DESIGN BASIS

Flasher

Heat flux - 80,000 Btu/(hr-ft³[of WF])

Delta-T - 1°F

Overall Heat Transfer Coefficient (U_v):

80,000 Btu/(hr-ft³[of WF]-°F)

WF depth - 1 ft

Melter/Condenser

Heat flux - 320,000 Btu/(hr-ft³[of WF])

Delta-T - 1°F

Overall Heat Transfer Coefficient (U_v):

320,000 Btu/(hr-ft³[of WF]-°F)

WF depth - 0.25 ft

DESIGN

FLASHER

Vapor leaving:

$$\begin{array}{rclcl} 80,200 & + & 26,063 & - & 11,319 & = & 94,944 \text{ \#/hr} \\ \text{(to process)} & & \text{(to Effect 4)} & & \text{(from Flash Tank)} & & \end{array}$$

Heat Transferred

$$Q = 94,944 \text{ \#/hr} \times 965 \text{ Btu/\#} = 91.6 \times 10^6 \text{ Btu/hr}$$

WF Required

$$\begin{aligned} \text{Volume} &= 91.6 \times 10^6 \text{ Btu/hr} / 80,000 \text{ Btu/(hr-ft}^3\text{[of WF])} \\ &= 1145 \text{ ft}^3 \text{ of WF} \end{aligned}$$

Number of Effects

$$\begin{array}{rclcl} \text{TTD} & = & 240.9 & - & 220.5 & = & 20.4^\circ\text{F} \\ \text{Total Temp. Diff.} & & \text{2nd Effect Temp.} & & \text{3rd Effect Temp.} & & \end{array}$$

Assume 3rd Effect is divided into 4 Effects:

$$\begin{array}{rclclclcl} \text{Total BPR} & = & 2.4 & + & 3.0 & + & 3.4 & + & 3.6 & = & 12.4^\circ\text{F} \\ & & \text{Effect 3A} & & \text{Effect 3B} & & \text{Effect 3C} & & \text{Effect 3D} & & \end{array}$$

$$\text{Total } \Delta T = 20.4 - 12.4 = 8^\circ\text{F}$$

$$\text{Delta-T per Effect} = 8/4 = 2^\circ\text{F}$$

WF Volume per Effect

$$\text{Volume per Effect} = 1145/4 = 286 \text{ ft}^3$$

Effect Cross Sectional Area

$$\begin{array}{rcl} 286 & / & 1 \\ \text{Volume per Effect} & & \text{Depth} \end{array} = 286 \text{ ft}^2$$

Vessel Dimensions

A horizontal cylindrical vessel 4 ft in diameter would provide ample space for the concentrate, WF, and vapor disengagement. Three 24 ft vessels would provide the required length.

Nozzles

Feed: 700 gpm - 8 in

Concentrate: 650 gpm - 8 in

DESIGN (CONTINUED)

FLASHER (CONTINUED)

Nozzles (Continued)

Vapor: $95,000/4 = 23,800 \text{ \#/hr}$ [per Effect] - 24 in

$$\begin{aligned}\text{Volume} &= 23,800 \text{ \#/hr} \times \text{hr}/60 \text{ min} \times 21.6 \text{ ft}^3/\# \\ &= 8,570 \text{ ft}^3/\text{min}\end{aligned}$$

$$\text{Velocity} = 8,570/2.948 = 2,910 \text{ fps}$$

Manway: PSV - 20 in

WF Inlet - 20 in

$$Q = 91.6 \times 10^6/4 = 22.9 \times 10^6 \text{ Btu/hr}$$

$$\begin{aligned}\text{WF frozen} &= 22.9 \times 10^6 \text{ Btu/hr} / 63 \text{ Btu/\#} = 363,500 \text{ \#/hr} \\ &= 363,500 \text{ \#/hr} / (8.33 \times 0.78) \text{ \#/gal} / 60 \text{ min/hr} \\ &= 933 \text{ gpm}\end{aligned}$$

If 1/3 of total is frozen,

$$\text{WF flow} = 933 \times 3 = 2,800 \text{ gpm}$$

WF Outlet - 24 in

WF PUMP - 3,000 gpm @ 10 ft TDH

MELTER/CONDENSER

Volume per Effect

$$\begin{aligned}\text{Volume} &= 22.9 \times 10^6 \text{ Btu/hr} / 320,000 \text{ Btu}/(\text{hr}\cdot\text{ft}^3[\text{of WF}]) \\ &= 72 \text{ ft}^3\end{aligned}$$

Effect Cross Sectional Area

$$\begin{array}{rcl} 72 & / & 0.25 \\ \text{Volume per Effect} & \text{Depth} & = 286 \text{ ft}^2 \end{array}$$

Vessel Dimensions

A horizontal cylindrical vessel 4 ft in diameter would provide ample space for the product water, WF, and vapor disengagement. Three 24 ft vessels would provide the required length.

Nozzles

Vapor (from previous Flasher) Inlet: - 24 in

DESIGN (CONTINUED)

MELTER/CONDENSER (CONTINUED)

Nozzles (Continued)

Product Water: 50 gpm - 2 in

Vapor (non-condensables only) Outlet: 2 in

Manway: PSV - 20 in

WF Inlet: 24 in

Wf Outlet 24 in

ESTIMATED SAVINGS

$\$(290,000 - 160,000) = \$130,000$

or 45 percent on purchased equipment cost.

To this must be added the other substantial savings. The 45 percent figure would also apply to installed cost, since the equipment to be installed is so much simpler. These savings would be reflected in a similar (45 percent) savings in maintenance cost.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
TEP ECONOMIC EVALUATION

OTHER BENEFITS

The section entitled "CAPITAL AND MAINTENANCE COST SAVINGS" focused primarily on the savings in capital (and, hence maintenance) costs ascribable to the comparatively simple TEP design. This section amplifies that work, and discusses some other specific advantages of the TEP.

PROBLEMS RELATED TO TUBES

In addition to being the reason for the energy savings of the TEP, the elimination of tubes is also the primary reason for the potential reduction in capital and maintenance costs. It has been estimated that the total installed cost of tubes is greater than the cost for the working substance which replaces them, even if a rather ordinary alloy like copper-nickel is used. If a special material, such as nickel (as used in the caustic soda industry) or titanium (which is used in some evaporators) is required, the cost for tubes would be much higher. It has also been confirmed that the evaporator vessel for the TEP would be much less than for conventional evaporators.

Tubes frequently need cleaning. They also develop leaks. In some instances, this causes unscheduled shutdowns of the plant to plug the offending tube(s). When sufficient tubes are lost this way, the tubes (generally all of the tubes) must be replaced.

Sugar Industry

There are also problems peculiar to each application. For example, in the sugar industry, not only do the tubes contribute heavily to the energy consumption and to capital and maintenance costs, but they act as catalysts for decomposition of the sugar. This problem would be eliminated by use of the TEP. This may also make it possible to operate at a higher top temperature, thus effecting further economies!

Caustic Soda Industry

As previously mentioned, the concentration of caustic soda is a good example of when the other advantages of eliminating tubes

become important. The following is a verbatim quotation from Reference 3:

CAUSTIC SODA CONCENTRATION FORCED-CIRCULATION EVAPORATORS

Description of the Process. The concentration of caustic soda solution obtained either from the reaction of soda ash and lime or from the electrolytic decomposition of brine present certain distinct problems: (2) Caustic solutions have a high elevation in boiling point which causes a large loss of available temperature difference in multiple-effect evaporators.

(2) The concentrated solutions are highly viscous, reducing sharply the heat-transfer rate in natural-circulation evaporators. (3) Caustic solutions may have detrimental effects on steel, causing caustic embrittlement. (4) Caustic solutions may require the removal of large quantities of salt as the solution becomes concentrated.

In order to avoid caustic embrittlement of steel, solid nickel tubes are used along with nickel-clad plate-steel bodies. All auxiliary equipment which comes in contact with the caustic solution is also nickel clad. Due to the costliness of nickel as a material of construction, however, it is important to design this class of equipment for as small a heating surface as possible. Since the liquor-film heat-transfer coefficient is a function of the velocity of the caustic solution through the tubes, a high velocity must be established in order to obtain a large transfer coefficient in the tubes.

The RCC Process

The RCC process was developed specifically to meet the need for low cost concentrators, yet it has several significant disadvantages when compared to the TEP. These disadvantages stem from the fact that the evaporator in the RCC process contains tubes, whereas the TEP evaporator is tubeless. The principal disadvantages caused by these tubes in the RCC evaporator are as follows:

1. Cost of tubes, tubesheets, water boxes, etc. Currently specified material is titanium which is very expensive
2. Much higher energy cost to drive the heat through the tubes
3. Inability to go to moist solids (tubes would plug)

4. At least some tendency to form scale
5. Cost of seeding equipment (required to control scale)
6. Higher capital cost
7. Higher operating cost

THE EUTECTIC FREEZING PROCESS

In addition to lower capital and energy costs, the TEP has other significant advantages in comparison to the eutectic freezing process:

Eutectic Salts and Moist Solids.

Frequently, eutectic salts are hydrates; hence, they contain water of hydration, as well as entrainment. Sodium chloride, for example, comes out as the dihydrate - 38 percent water (plus entrainment) which will be released when the solids are heated to above 0°C . Sodium sulfate, the first eutectic salt to come out of many brackish waters, is even worse. It comes out as the decahydrate - 56 percent water (plus entrainment) which will be released when the solids are heated to 32°C . If these eutectic salts are removed from the brine by a filter they will have to be filtered again after they are warmed up. Since the TEP operates above these temperatures, this problem does not exist. Solids that are filtered out of the brine will contain entrainment only.

Viscosity Problems.

The TEP can do some jobs that freezing processes either cannot do at all, or cannot do as completely. For some feeds (sugar, for example), the degree to which the feed can be concentrated by the freezing process is limited by viscosity problems, because of the low temperature at which the freezing processes operate.

Product Quality

Finally, the TEP produces distilled water.

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-861D12619

TEP ECONOMIC EVALUATION

OPERATING COSTS

Quantitative determination of annual costs is a site and company specific task, because these costs are a function of accounting methods, local conditions, et cetera. But there can be no doubt that the large reductions in such important contributors to cost as energy consumption (now replacing beets as the largest cost factor for beet sugar), and capital and maintenance costs will result in proportionate reductions in annual costs.

CONFIDENTIAL - SECURITY INFORMATION

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APPARENT PROBLEMS CONSIDERED

TUBELESS EVAPORATION PROCESS DEVELOPMENT

DOE CONTRACT No. DE-AC7-861D12619

APPARENT PROBLEMS CONSIDERED

Concern has been expressed about certain apparent problems with the process, particularly with regard to losses and degradation of the WF, and entrainment of the WF in the vapor from the Flasher. Also, the economic impact of heat-tracing, and the controllability of the process in the multieffect mode, have been questioned. An attempt has been made to consider and deal with these questions. This examination has revealed that these problems are more apparent than real. The latest information on these most frequently asked questions is presented here.

WORKING FLUID LOSSES

"Is there a significant loss of working fluid in the concentrated stream?"

No.

Solubility

Great care was taken in the selection of WF's to choose materials that are as insoluble as glass. Fortunately, the materials finally selected - the paraffins and polyethylene are as insoluble as glass. It is unlikely that anyone is seriously concerned about the milk that they drink being contaminated with paraffin (which coats the milk's container); or that they have similar fears about polyethylene contamination when they drink bottled water from a polyethylene container. However, although these materials are universally known for their insolubility and inertness, definitive data are hard to come by, since most references simply list the materials that were under consideration as "insoluble" without defining the sensitivity of the data. This is probably due to the fact that below a certain level, solubility is difficult to determine. Something can be inferred by the trend of the solubilities of homologs. For example, in the normal alcohol series, the C_{14} compound's solubility is listed as 9 grams per 100 grams of water, the C_{13} as 2.7, the C_{12} as 0.6, the C_{11} as 0.18, the C_{10} as 0.054 and the C_9 and C_8 as insoluble. This is the normal trend as molecular weight increases. Of more importance, it is also the trend for paraffins. The paraffin series in going from C_{14} to C_8 drops in solubility from about 7 to about 2 parts per billion. Losses due to solubility in either the product water or concentrate stream are expected to be negligible. They are certainly too small to measure by ordinary (or even somewhat extraordinary) laboratory

means. However, it is possible to put the "problem" into perspective by using the published data:

The flow diagram of Exhibit ASC (a commercial sugar plant) shows the quantity of solution leaving the last evaporator to be 165,270 lb/hr or 1.31 billion lb/(330 day)year. At 2 parts per billion solubility, WF losses would be 2.62 lb/yr.

Suspension

In numerous laboratory and bench scale tests performed over several years, the Authors have not observed any tendency for the WF's to remain suspended in the either of the aqueous layers. But, this is as expected. The WF's are of sufficiently different density, and of low enough viscosity, that a rapid and complete separation would be expected. The real question is not whether a separation will occur, but how long does it take to happen. As discussed **PHASE SEPARATION**, the Bench Scale Plant did not lend itself to obtaining this type of information. The time required for complete separation, and the tendency of various feeds to suspend the WF's (or any other unforeseen separation problem) will have to be learned in the Pilot Plant.

Of course, it is almost a certainty that a troublesome feed that wont separate, or separates poorly or slowly, can be found. Perhaps the presence of a surfactant in the right amount in some feeds might be such a case. This will simply have to be determined on a case by case basis. We repeat. We see no evidence of a problem of this type with the feeds that were studied during this project.

But, suppose that for some feeds there was some WF carried over to the next effect. This would simply mean a loss in efficiency, until the last effect were reached. The concentrate from the last effect would simply have to be stored longer (see Misoperation, below).

Misoperation

One could postulate ways in which losses could occur due to serious misoperation of the process. In the unlikely event that this becomes a problem, it could be solved by installing an extra "guard" tank on the offending stream. WF's are lighter than water, and they could be returned to the system from the top of the tank periodically. A maximum of two such tanks (product water and concentrate) would be required. In most instances, these tanks already exist.

WORKING FLUID DEGRADATION

Working Fluid Contamination

"Does working fluid performance degrade due to accumulation of contaminants?"

No, with the possible exception of unknown organic substances, as yet unobserved.

"What is their effect on melting points?"

Negligible.

In the normal operation of the process, if the feed is scale forming, scale will come out of the feed as it is concentrated. If the feed is concentrated sufficiently, other solids will come out. The WF's are completely melted once on each flow through the process, and are lighter than water. All of the scale and other solids that come out of the feeds that were used in the laboratory program are considerably heavier than water. The normal expectation is that they would separate by gravity and leave the system with the brine. This phenomenon has been observed in numerous experiments in the laboratory and bench scale plant. In fact, laboratory experience shows that impurities are removed from the "as received" WF by contact with water. Compare the "WATER WASHED" and "UNTREATED" curve for durene on the cooling curves following.

Inorganic compounds are positively rejected from paraffins (Phosphates could, perhaps, cause foaming). Paraffins are really not good solvents for organics. Sugar, for example, which is very soluble in water is listed as 1% soluble in alcohol, and insoluble in ether, and chloroform. This does not preclude the possibility that some unexpected impurity may cause a problem. If so, we will have to deal with that particular problem then. None are apparent now. This will be a subject of further observation in the Pilot Plant.

Small quantities of dissimilar material have little effect on the temperature at which most of the material solidifies. This phenomenon would have a negligible effect on the process.

Stability

A WF which was not stable (inert) in the presence of all materials with which it would come in contact (feed, concentrate, product water, impurities, materials of construction, et cetera) in the process would be virtually useless. Fortunately, materials exist which are stable under the most severe conditions. Paraffins, for example, are inert, except to burning and chlorination with dry chlorine. Nevertheless, in the

interest of completeness, tests were made which demonstrated stability in the presence of boiling:

- 10% nitric acid
- 10% sulfuric acid
- 10% caustic soda (the principal ingredient in one of the "black liquors" from the pulp and paper industry)
- 10% ferric chloride hexahydrate and 10% concentrated hydrochloric acid solution
- Spent pickling liquor from a steel mill

Polyethylene - the other principal WF besides the paraffins - is similarly stable.

ENTRAINMENT

"Is there a significant carry-over of working fluid in the Flasher vapor?"

No.

The Flasher is designed as a short-fat rather than a tall-thin vessel, in order to minimize submergence losses in the working fluid section. Hence, in the Pilot Plant, and the commercial plants that follow (as contrasted with the Bench Scale Plant), the vapor velocity leaving the Flasher will be so low, that significant entrainment would probably not occur even without an entrainment separator. However, entrainment separators of various types (mesh, hook and vane) will be considered. Heated entrainment separators, while not in widespread use, have been designed before for use in freezing processes. This type of design would be utilized.

If, in spite of these precautions, some material is carried over, it is not a serious problem. Just as was the case for carry-over in the concentrate, it represents a small loss in efficiency. In all but the last effect, the material is recovered in the next effect. Over a long (probably very long) time, the melting range of the next effect could be affected sufficiently to require the addition of a very small amount of the higher melting component of the working fluid. It would be presumptuous to say that we know enough now to estimate how much and when. None and never is one definite possibility. This is another matter which will be under close scrutiny during the Pilot Plant operation. Losses from the last effect would be prevented in the same manner as those potential losses in the product stream leaving (see Misoperation).

ECONOMIC IMPACT OF HEAT-TRACING

"Is extensive heat-tracing needed to prevent buildup of solidified working fluid?"

No.

No meaningful comparison can be made between the Bench Scale Plant and the Pilot Plant, regarding heat tracing requirements. The Bench Scale Plant was uninsulated. The pilot plant requirements are useful only in establishing a conservative (almost meaningless) upper limit for commercial plants, because the surface to volume ratio for commercial plants for equipment and piping is so much lower. Heat tracing and insulation accounted for less than 10 percent of the estimated cost of the Pilot Plant. It is premature to try to estimate requirements for commercial plants, but an educated guess would be in the range of

1 - 4 percent of total costs, with the expectation being nearer to the low end.

PROCESS CONTROLLABILITY

"Is a multieffect TEP stable and controllable?"

Yes.

Control problems will not arise from the fact that the process is operated in the multieffect mode. Hundreds of multieffect evaporation processes have been successfully designed, built, and operated. This technology can be regarded as standard Chemical Engineering-In-Practice. The Principal Investigator of this project has personally performed many such designs and has successfully started-up and operated processes which are very similar to the TEP.

The control problems will arise from within an effect. Even here, most of the technology is rather old and familiar state-of-the-art. Control of the interface level in the various pieces of equipment is probably the most difficult job. This will require employment of relatively new, but nonetheless proven technology.

CONVINCING EVIDENCE

The Authors find the weight of evidence arising from numerous experiments, and supported by theoretical considerations and data from standard reference works, convincing. No serious problem in any of the areas discussed above is anticipated.

ACTUAL COOLING CURVES FOR DURENE FROM SUN CHEMICAL CORP.

PILOT PLANT DESIGN

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
TUBELESS EVAPORATION PROCESS PILOT PLANT

PILOT PLANT DESIGN

The following is a summary the current design basis for the Pilot Plant:

OVERALL

The overall objective is a Pilot Plant with the capability of operating over the range of temperatures (and their corresponding pressures) for the various feeds as presented in ENERGY SAVINGS in PART II. These included:

Liquor Temperature: 250°F

Pressure: 15 psig

Vacuum: 23 in Hg

Feeds: sugar, salt, caustic soda, and other solutions; cooling tower blowdown, "spent" pickling liquor, and other waste streams

Capacity: The general intent is to provide for testing both high turndown and above design capacity. Particulars are provided under the discussions of equipment which follow.

BASE CASE

The case shown on the Process Flow Sheet (the "Base Case") is at the last effect conditions in a sugar solution evaporation train.

V-1 FLASHER - L/S PHASE SEPARATOR

Basis - As Phase Separator:

One minute holdup and 1 ft/min

One ft/min requires about 30 in dia, with 1 min holdup/ft

Basis - As Flasher

Heat flux - 20,000 - 80,000 btu/(hr-ft³[of WF]-°F)
WF depth - 1 ft

Vapor space height - 1-3 ft
Concentrate depth - 1-2 ft
Delta-T - 1°F

Bench Scale Plant (BSP) experience showed that 1 ft of depth of WF is ample; more would increase submergence losses unnecessarily. At this depth a 6 ft dia would provide the required volume for the minimum heat flux.

A 6 ft dia x 6 ft vessel would meet this requirement. However, a 10 ft vessel would cost little more, and would greatly increase flexibility in testing various heights of vapor space, concentrate space, et cetera.

The minimum case could be regarded as the turn down test of the expected 80,000 btu rate.

Operating fluxes could also be varied by adjustment of the feed rate and/or the ΔT .

V-1A - AUXILIARY PHASE SEPARATOR L/S

Basis - Same as V-1 as Phase Separator

30 in dia x 2 ft required - use 30 in dia x 4 ft

This vessel is used to remove concentrate from the L/S WF that may have carried over from V-1. It is hoped that Pilot Plant tests may prove that this vessel is unnecessary.

V-2 MELTER/CONDENSER - PHASE SEPARATOR L

Basis - As Phase Separator - Same as V-1

Basis - As Melter/Condenser

Heat flux - 60,000 btu/(hr-ft³[of WF]°F)

Disc & Doughnut (D&D) empty tower velocity - 1 ft/sec

The entire amount of steam could be condensed in a 3 in deep WF layer. At the high temperature end of the plant (240-250°F), this loss would be acceptable (<0.2°). But, at low temperatures (say 100-110°F) the loss would be nearly 3°F - clearly unacceptable. So a D&D tower would be required at least for the low temperature effects. The Pilot Plant should have the capability of testing both of these concepts.

A 6 ft dia x 4 ft section would meet these requirements for the lower section.

A four in dia x 6 ft D&D would just meet the requirement at design flow rates. An 8 in nozzle should be provided to permit testing at higher rates.

V-2A AUXILIARY PHASE SEPARATOR L

Same as V-1A

TANKS

Basis - 8 hr holdup

PUMPS

Pump will be selected so that less than the maximum impeller for that pump can provide at least 125 % of the base case flow rate. Still larger flows can be accommodated by the installation of larger impellers.

The WF pump (Process Flow Sheet) capacity is based on freezing one-third of the WF in the Flasher.

CONDENSERS

The final condenser is sized for the base case with a 10° approach.

The guard condenser is the same as the final condenser.

PROJECT DRAWINGS

MATERIAL BALANCE

STREAM No.	1	2	3	4	5	6	7	8	9	10
STREAM	FEED	W	PRODUCT VAPOR	CONDENSATE	CONCENTRATE	L/S W	STEAM	PRODUCT WATER	COND WATER IN	COND WATER OUT
FLOW - LB/HR	2,222	24,048	200	200	1,722	24,048	200	200	7,222	7,222
W										
FLOW - GPM	3.67	61.7		1.00	2.71	61.7		1.01	15.1	15.1
AS CONC - Wt%	48.8				60.0	55.5				
W - POLYMER - Wt%										
FEED - G	164.0	164.0	164.0	167.0	164.0	164.0	164.0	164.0	70.0	157.0
FEEDS - PSIG										
VAC - 12 Hg			25.0				21.0			
PS G	1.81	0.78		1.00	1.29	0.78		1.00	1.00	1.00

DEPARTMENT OF ENERGY IDAHO OPERATIONS OFFICE IDAHO FALLS, IDAHO	
TORRELESS EVAPORATION PROCESS DEVELOPMENT DOE CONTRACT No. DE-AC07-86ID18619	
TORRELESS EVAPORATION PROCESS PILOT PLANT	
SCHROEDER PROCESS CORPORATION SANTA ANA, CALIFORNIA	DEPARTMENT OF ENERGY
DESIGNED <u>P.J.S.</u> DATE <u>10-28-88</u> DRAWN <u>GAD</u> DATE <u>10-28-88</u> CHECKED <u>_____</u> DATE <u>_____</u> APPROVED <u>_____</u> DATE <u>_____</u>	REVIEWED <u>_____</u> APPROVED <u>_____</u>
DRAWING TITLE PROCESS FLOW DIAGRAM	DATE <u>10-28-88</u> DRAWING NUMBER <u>DCS-2-1</u>

NOTES:

1. ALL PIPE IS 1" SCH. 40 FRP, EXCEPT AS NOTED
2. ALL PIPE IS INSULATED, EXCEPT AS NOTED

DEPARTMENT OF ENERGY IDAHO OPERATIONS OFFICE IDAHO FALLS, IDAHO	
TUBELESS EVAPORATION PROCESS DOE CONTRACT No. DE-AC05-80-OR21400	
TUBELESS EVAPORATION PROCESS PILOT PLANT	
SCHROEDER PROCESS CORPORATION SANTA ANA, CALIFORNIA	DEPARTMENT OF ENERGY
DESIGNED: P.J.S. DATE: 10-18-86	REVIEWED: _____
DRAWN: CAD DATE: 10-18-86	APPROVED: _____
CHECKED: _____ DATE: _____	DATE: 10-18-86
DRAWING TITLE: PIPING & INSTRUMENT DIAGRAM	DRAWING NUMBER: DOE-2-2

PRELIMINARY

PLAN

FRONT ELEVATION

SIDE ELEVATION

<p>PRELIMINARY</p>			
<p>DEPARTMENT OF ENERGY OPERATIONS OFFICE FALLS, IDAHO</p>			
<p>EVAPORATION PROCESS DEVELOPMENT CONTRACT NO. DE-AC07-86D12619 TUBELESS EVAPORATION PROCESS PILOT PLANT</p>			
<p>SCHROEDER PROCESS CORPORATION SANTA ANA, CALIFORNIA</p>		<p>DEPARTMENT OF ENERGY</p>	
DESIGNED	PKS	DATE	10-30-88
DRAWN	CAD	DATE	10-30-88
CHECKED		DATE	
APPROVED		DATE	
<p>DRAWING TITLE EQUIPMENT PLAN & ELEVATION</p>		DATE	10-30-86
		DRAWING NUMBER	DOE-2-3

DEPARTMENT OF ENERGY IDAHO OPERATIONS OFFICE IDAHO FALLS, IDAHO			
TUBELESS EVAPORATION PROCESS DEVELOPMENT DOE CONTRACT No. DE-AC07-661D12619			
TUBELESS EVAPORATION PROCESS PILOT PLANT			
SCHROEDER PROCESS CORPORATION SANTA ANA, CALIFORNIA		DEPARTMENT OF ENERGY	
DESIGNED	P.J.S.	DATE	11-22-66
DRAWN	CAD	DATE	11-22-66
CHECKED		DATE	
APPROVED		DATE	
DRAWING TITLE ONE LINE ELECTRICAL		DATE 11-22-66	DRAWING NUMBER DOE-2-1

EQUIPMENT DRAWINGS

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 0 PSIG
 VACUUM
 TEMPERATURE 300 °F
 WT. EMPTY 700#
 WT. FULL OF WATER 22,000#

OPERATING CONDITIONS:

PRESSURE 0 PSIG
 VACUUM in Hg
 TEMPERATURE 170 °F
 * NORMAL LIQUID LEVEL FT
 SpG of LIQUID -1.2

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL FRP
 INTERNALS S.S.
 PACKING
 LINING EPOXY

CODE STAMP - NONE

HEAT TRACING - NONE

INSULATION - JACKETED FIBERGLASS

NOZZLE SCHEDULE

MARK	SIZE	SERVICE	ETC	TYPE
B	2	BOTTOM OUTLET	150#	RF-FLG
C	2	CONDENSATE OUT	"	NOTE 2
F	2	CONDENSATE IN	"	RF-FLG
G1-S	1	LEVEL GAGE	"	"
S	2	STEAM IN	"	NOTE 2
T	1	TEMP TRANSMIT	"	RF-FLG
T1	1	TEMP INDICATOR	"	"
V	2	VENT	"	"
M1-S	80	MANWAY	"	STD

NOTES:

1. VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL
2. DOUBLE FLANGE
3. VESSEL FABRICATOR TO INSTALL HEATING COIL CONSISTING OF APPROX. 16 FT OF 2-IN SCH 80 S.S. PIPE, FLANGED AS REQUIRED TO FACILITATE REMOVAL

BY P.J.S.

DATE 12-8-86

CHK'D ---

APP'D ---

T-1
 FEED TANK

TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
 IDAHO OPERATIONS OFFICE
 IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
 DOE CONTRACT No. DE-AC07-86ID12618
 REVIEWED --- APPROVED ---

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

ATC MOBILE VESSEL

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 25 PSIG
 VACUUM FULL
 TEMPERATURE 200 °F
 WT. EMPTY 8,800#
 WT. FULL OF WATER 25,000#

OPERATING CONDITIONS:

PRESSURE 15 PSIG
 VACUUM 23 in Hg
 TEMPERATURE 200 °F
 NORMAL LIQUID LEVEL FT
 SpG of LIQUID - 1.2

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL C.S.
 INTERNALS FRP
 PACKING
 LINING EPOXY

CODE STAMP - ASME

HEAT TRACING - NONE

INSULATION - JACKETED FIBERGLASS

NOZZLE SCHEDULE

MARK	SIZE	SERVICE	HTG	TYPE
B	2	BOTTOM OUTLET	150#	RF-FLG
P	2	CONCENTRATE IN	.	.
G1-2	1	LEVEL GAGE	.	.
P	1	PRESSURE TRAN	.	.
TI	1	TEMP INDICATOR	.	.
V	2	VENT	.	.
M1-2	20	MANWAY	.	STD

NOTES:

1. VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL
2. VESSEL FABRICATOR TO INSTALL VESSEL SUPPORT

BY P.J.B.

DATE 11-3-86

CHK'D

APP'D

T-2

CONCENTRATE TANK

TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
 IDAHO OPERATIONS OFFICE
 IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
 DOE CONTRACT No. DE-AC07-86ID18619
 REVIEWED APPROVED

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

VESSEL DESIGN DATA																																																													
DESIGN CONDITIONS: PRESSURE 25 PSIG VACUUM FULL TEMPERATURE 300 °F WT. EMPTY 1,450# WT. FULL OF WATER 8,600#																																																													
OPERATING CONDITIONS: PRESSURE 15 PSIG VACUUM 23 in Hg TEMPERATURE 250 °F NORMAL LIQUID LEVEL FT SpG of LIQUID -1.0																																																													
CORROSION ALLOWANCE NONE																																																													
MATERIAL: SHELL C.S. INTERNALS FRP PACKING LINING EPOXY																																																													
CODE STAMP - ASME																																																													
HEAT TRACING - NONE																																																													
INSULATION - JACKETED FIBERGLASS																																																													
NOZZLE SCHEDULE																																																													
<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: center;">MARK</th> <th style="text-align: center;">SIZE</th> <th style="text-align: center;">SERVICE</th> <th style="text-align: center;">RTG</th> <th style="text-align: center;">TYPE</th> </tr> </thead> <tbody> <tr> <td style="text-align: center;">B</td> <td style="text-align: center;">2</td> <td style="text-align: center;">BOTTOM OUTLET</td> <td style="text-align: center;">150#</td> <td style="text-align: center;">RT-FLG</td> </tr> <tr> <td style="text-align: center;">F</td> <td style="text-align: center;">2</td> <td style="text-align: center;">PRODUCT IN</td> <td style="text-align: center;">.</td> <td style="text-align: center;">.</td> </tr> <tr> <td style="text-align: center;">G1-G</td> <td style="text-align: center;">1</td> <td style="text-align: center;">LEVEL GAGE</td> <td style="text-align: center;">.</td> <td style="text-align: center;">.</td> </tr> <tr> <td style="text-align: center;">P</td> <td style="text-align: center;">1</td> <td style="text-align: center;">PRESSURE TRAN</td> <td style="text-align: center;">.</td> <td style="text-align: center;">.</td> </tr> <tr> <td style="text-align: center;">TI</td> <td style="text-align: center;">1</td> <td style="text-align: center;">TEMP INDICATOR</td> <td style="text-align: center;">.</td> <td style="text-align: center;">.</td> </tr> <tr> <td style="text-align: center;">V</td> <td style="text-align: center;">2</td> <td style="text-align: center;">VENT</td> <td style="text-align: center;">.</td> <td style="text-align: center;">.</td> </tr> <tr> <td style="text-align: center;">M</td> <td style="text-align: center;">20</td> <td style="text-align: center;">MANWAY</td> <td style="text-align: center;">.</td> <td style="text-align: center;">STD</td> </tr> <tr><td> </td><td> </td><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td><td> </td><td> </td></tr> </tbody> </table>	MARK	SIZE	SERVICE	RTG	TYPE	B	2	BOTTOM OUTLET	150#	RT-FLG	F	2	PRODUCT IN	.	.	G1-G	1	LEVEL GAGE	.	.	P	1	PRESSURE TRAN	.	.	TI	1	TEMP INDICATOR	.	.	V	2	VENT	.	.	M	20	MANWAY	.	STD																					NOTES: 1. VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL 2. VESSEL FABRICATOR TO INSTALL VESSEL SUPPORT
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B	2	BOTTOM OUTLET	150#	RT-FLG																																																									
F	2	PRODUCT IN	.	.																																																									
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V	2	VENT	.	.																																																									
M	20	MANWAY	.	STD																																																									
	<div style="text-align: center;"> T-3 PRODUCT TANK </div> <div style="text-align: center; font-size: small;"> TUBELESS EVAPORATION PROCESS P. PLANT </div>																																																												
BY <u>P.J.E.</u> DATE <u>11-3-86</u> CHK'D _____ APP'D _____	<div style="text-align: center;"> DEPARTMENT OF ENERGY IDAHO OPERATIONS OFFICE IDAHO FALLS, IDAHO </div> <div style="text-align: center; font-size: small;"> TUBELESS EVAPORATION PROCESS DEVELOPMENT DOE CONTRACT No. DE-AC07-86ID12619 REVIEWED _____ APPROVED _____ </div>																																																												

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 0 PSIG
 VACUUM
 TEMPERATURE 300 °F
 WT. EMPTY 400#
 WT. FULL OF WATER 5,400#

OPERATING CONDITIONS:

PRESSURE 0 PSIG
 VACUUM 1 in Hg
 TEMPERATURE 170 °F
 NORMAL LIQUID LEVEL FT
 SpG of LIQUID - 1.0

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL FRP
 INTERNALS FRP
 PACKING
 LINING EPOXY

CODE STAMP - NONE

HEAT TRACING - NONE

INSULATION - JACKETED FIBERGLASS

NOZZLES SCHEDULE

MARK	SIZE	SERVICE	RTG	TYPE
B	2	BOTTOM OUTLET	150#	RF-FLG
F	2	CONDENSATE IN	"	"
G1-2	1	LEVEL GAGE	"	"
TI	1	TEMP INDICATOR	"	"
V	2	VENT	"	"
M	20	MANWAY	"	STD

NOTES:

1. VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL

BY P.J.S.
 DATE 11-3-88
 CHK'D _____
 APP'D _____

T-4
CONDENSATE TANK

TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
 IDAHO OPERATIONS OFFICE
 IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
 DOE CONTRACT No. DE-AC07-86ID18619
 REVIEWED _____ APPROVED _____

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 25 PSIG
VACUUM FULL
TEMPERATURE 300 °F
WT. EMPTY 4,200#
WT. FULL OF WATER 25,000#

OPERATING CONDITIONS:

PRESSURE 15 PSIG
VACUUM 23 in Hg
TEMPERATURE 250 °F
NORMAL LIQUID LEVEL FT
SpG of LIQUID - 1.2 BOT & 0.78 TOP

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL C.S.
INTERNALS FRP
PACKING
LINING EPOXY

CODE STAMP - ASME

HEAT TRACING - ELECTRICAL

INSULATION - JACKETED FIBERGLASS

NOZZLE SCHEDULE

MARK	SIZE	SERVICE	RTC	TYPE
B	2	BOTTOM OUTLET	150#	RT-FLG
F1-16	2	FEED OR WF IN	"	"
G1-2	1	LEVEL GAGE	"	"
I	2	INTERFACE TRAN	"	"
L	2	LEVEL CONTROL	"	"
O1-16	3	CONC OR WF OUT	"	"
P	1	PRESSURE TRAN	"	"
V	2	VENT	"	"
M1-2	20	MANWAY	"	STD

NOTES:

1. VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL

BY P.J.S.
DATE 11-3-88
CHK'D _____
APP'D _____

V-1
FLASHER-PHASE SEPARATOR L/S
TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
IDAHO OPERATIONS OFFICE
IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC07-86ID18618
REVIEWED _____ APPROVED _____

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 25 PSIG
VACUUM FULL
TEMPERATURE 300 °F
WT. EMPTY 1,000
WT. FULL OF WATER 2,600#

OPERATING CONDITIONS:

PRESSURE 15 PSIG
VACUUM 23 in Hg
TEMPERATURE 250 °F
NORMAL LIQUID LEVEL FT
SpG of LIQUID - 1.2 BOT & 0.78 TOP

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL C.S.
INTERNALS FRP
PACKING
LINING EPOXY

CODE STAMP - ASME

HEAT TRACING - ELECTRICAL

INSULATION - JACKETED FIBERGLASS

NOZZLE SCHEDULE

MARK	SIZE	SERVICE	ETC	TYPE
B	2	BOTTOM OUTLET	150#	RP-FLG
F1-4	2	WP IN	.	.
G1-2	1	LEVEL GAGE	.	.
I	2	INTERFACE TRAN	.	.
L	2	LEVEL CONTROL	.	.
O1-4	3	CONC OR WP OUT	.	.
P	1	PRESSURE TRAN	.	.
T1-2	1	TEMP TRANSMIT	.	.
V	2	VENT	.	.
M	20	MANWAY	.	STD

NOTES:

- VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL

BY P.J.S.
DATE 11-9-86
CHK'D _____
APP'D _____

V1-A

AUXILIARY

PHASE SEPARATOR L/S

TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
IDAHO OPERATIONS OFFICE
IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC07-86ID12819
REVIEWED _____ APPROVED _____

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 85 PSIG
 VACUUM FULL
 TEMPERATURE 800 °F
 WT. EMPTY 2,000#
 WT. FULL OF WATER 12,600#

OPERATING CONDITIONS:

PRESSURE 16 PSIG
 VACUUM 85 in Hg
 TEMPERATURE 880 °F
 NORMAL LIQUID LEVEL FT
 SpG of LIQUID - 1.0 BOT & 0.76 TOP

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL C.S.
 INTERNALS FRP
 PACKING
 LINING EPOXY

CODE STAMP - ASME

HEAT TRACING - ELECTRICAL

INSULATION - JACKETED FIBERGLASS

NOZZLE SCHEDULE

MARK	SIZE	SERVICE	RTG	TYPE
B	2	BOTTOM OUTLET	150#	RF-FLG
CR	1	COND RETURN	"	"
D	8	DISC & DOUGHT	"	"
F1-6	2	L/S WF IN	"	"
G1-2	1	LEVEL GAGE	"	"
I	2	INTERFACE TRAN	"	"
L	2	LEVEL CONTROL	"	"
O1-6	3	STM IN/WF OUT	"	"
P	1	PRESSURE TRAN	"	"
V	2	VENT	"	"
M	20	MANWAY	"	STD

NOTES:

1. VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL

BY P.J.S.
 DATE 11-3-86
 CHK'D _____
 APP'D _____

V-2
 MELTER/CONDENSER
 PHASE SEPARATOR L
 TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
 IDAHO OPERATIONS OFFICE
 IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
 DOE CONTRACT No. DE-AC07-86ID12619
 REVIEWED _____ APPROVED _____

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

VESSEL DESIGN DATA

DESIGN CONDITIONS:

PRESSURE 86 PSIG
 VACUUM FULL
 TEMPERATURE 800 °F
 WT. EMPTY 1,000
 WT. FULL OF WATER 2,600#

OPERATING CONDITIONS:

PRESSURE 15 PSIG
 VACUUM 23 in Hg
 TEMPERATURE 250 °F
 NORMAL LIQUID LEVEL FT
 SpG of LIQUID - 1.0 BOT & 0.78 TOP

CORROSION ALLOWANCE NONE

MATERIAL:

SHELL C.S.
 INTERNALS FRP
 PACKING
 LINING EPOXY

CODE STAMP - ASME

HEAT TRACING - ELECTRICAL

INSULATION - JACKETED FIBERGLASS

NOZZLE SCHEDULE

MARK	SIZE	SERVICE	ETC	TYPE
B	2	BOTTOM OUTLET	150#	RF-FLG
P1-4	2	WF IN	"	"
G1-2	1	LEVEL GAGE	"	"
I	2	INTERFACE TRAN	"	"
L	2	LEVEL CONTROL	"	"
O1-4	3	COND OR WF OUT	"	"
P	1	PRESSURE TRAN	"	"
T1-2	1	TEMP TRANSMIT	"	"
V	2	VENT	"	"
M	20	MANWAY	"	STD

NOTES:

- VESSEL FABRICATOR TO INSTALL LIFTING LUGS ON VESSEL

BY P.J.S.

DATE 11-9-86

CHK'D ---

APP'D ---

V-2A

AUXILIARY

PHASE SEPARATOR L

TUBELESS EVAPORATION PROCESS P. PLANT

DEPARTMENT OF ENERGY
 IDAHO OPERATIONS OFFICE
 IDAHO FALLS, IDAHO

TUBELESS EVAPORATION PROCESS DEVELOPMENT
 DOE CONTRACT No. DE-AC07-86ID12819

REVIEWED --- APPROVED ---

SCHROEDER PROCESS CORPORATION - SANTA ANA, CALIFORNIA

PILOT PLANT COST ESTIMATE

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619

PILOT PLANT COST ESTIMATE

The Pilot Plant Cost Estimate is presented in the following principal sections:

SUMMARY

This section presents purchase, installation, and total costs in each of the following categories:

Process Equipment
Instruments
Control Valves
Total Purchased Equipment
Piping
Electrical
Heat Tracing and Insulation
Sales Tax, Freight, and Contingency

PURCHASED EQUIPMENT

This section presents information on the supplier, source of quote, basis of inquiry, service, cost and hours to install for each item (process equipment, instruments, and control valves) on the Piping and Instrument Diagram.

STRUCTURAL

This section provides a breakdown of Structural Costs.

PIPING

This section provides a breakdown of Piping Costs.

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
PILOT PLANT COST ESTIMATE

SUMMARY

<u>PROCESS EQUIPMENT</u>	<u>PURCHASE COST</u>	<u>INSTALLATION HOURS</u>	<u>COST</u>	<u>TOTAL COST</u>
Tanks	\$14,090	80	\$3,200	\$17,290
Vessels	12,990	130	5,200	18,190
Pumps	13,580	17	680	14,260
Heat Exchangers	2,800	8	320	3,120
Vacuum Pumps	<u>1,360</u>	<u>4</u>	<u>160</u>	<u>1,520</u>
TOTAL PROCESS EQUIPMENT	\$44,820	239	\$9,560	\$54,380
TOTAL INSTRUMENTS	103,760	537	16,110	119,870
TOTAL CONTROL VALVES	<u>13,490</u>	<u>34</u>	<u>1,020</u>	<u>14,510</u>
TOTAL PURCHASED EQUIPMENT	\$162,070	810	\$24,300	\$188,760
STRUCTURAL				12,740
PIPING				42,910
ELECTRICAL				2,000
HEAT TRACING AND INSULATION				<u>30,600</u>
SUBTOTAL				\$277,010
SALES TAX @ 4% OF MATERIALS				7,950
FREIGHT @ 4 % OF MATERIALS				<u>7,950</u>
SUBTOTAL				\$292,910
CONTINGENCY @ 7%				<u>20,500</u>
GRAND TOTAL				\$313,410

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
PILOT PLANT COST ESTIMATE

PURCHASED EQUIPMENT

TANKS - FIBERGLASS REINFORCED PLASTIC (FRP)

Supplier: TEC Tanks

Source of Quote: Court & Thomas

Basis of Inquiry: Drawings

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
T-1	Feed Tank	\$5,300	25
T-4	Condensate Tank	<u>1,700</u>	<u>15</u>
TOTAL FRP TANKS		\$7,000	40

TANKS - EPOXY COATED STEEL

Supplier: Ace Buehler

Source of Quote: Ace Buehler

Basis of Inquiry: Drawings

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
T-2	Concentrate Tank	\$4,420	25
T-3	Product Tank	<u>2,670</u>	<u>15</u>
TOTAL STEEL TANKS		\$7,090	40

VESSELS

Supplier: Ace Buehler

Source of Quote: Ace Buehler

Basis of Inquiry: Drawings

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
V-1	Flasher-Phase Separator L/S	\$ 5,760	50
V-1A	Auxiliary Phase Separator L/S	2,090	20
V-2	Melter/Condenser-Phase Separator L	3,050	40
V-2A	Auxiliary Phase Separator L	<u>2,090</u>	<u>20</u>
TOTAL VESSELS		\$12,990	130

PUMPS

Supplier: Crane/Chempump

Source of Quote: Fallon Engineering Company

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
P-1	Feed Pump	\$ 2,550	3
P-2	Concentrate Pump	2,550	3
P-3	Product Pump	2,550	3
P-WF	WF Pump	3,380	5
P-4	Condensate Pump	<u>2,550</u>	<u>3</u>
TOTAL PUMPS		\$13,580	17

HEAT EXCHANGERS

Supplier: American Standard

Source of Quote: American Standard

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
HX-1A	Final Condenser A	\$ 700	2
HX-1B	Final Condenser B	700	2
HX-2A	Guard Condenser A	700	2
HX-2B	Guard Condenser B	<u>700</u>	<u>2</u>
TOTAL HEAT EXCHANGERS		\$2,800	8

VACUUM PUMPS

Supplier: Dayton/Speedaire

Source of Quote: Grainger

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
U-1	Hi-Vac Pump	\$ 680	2
U-2	Lo-Vac Pump	<u>680</u>	<u>2</u>
TOTAL VACUUM PUMPS		<u>1,360</u>	<u>4</u>
TOTAL PROCESS EQUIPMENT		\$44,820	239

INSTRUMENTS

Level Gages

Supplier: Penberthy

Source of Quote: Control Specialists Incorporated

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
LG-T1	Feed Tank	330	18
LG-T2	Concentrate Tank	180	10
LG-T3	Product Tank	160	9
LG-T4	Condensate Tank	160	9
LG-V1	Flasher-Phase Separator L/S	170	18
LG-V1A	Auxiliary Phase Separator L/S	160	9
LG-V2	Melter/Condenser-Phase Separator L	160	9
LG-V2A	Auxiliary Phase Separator L	160	9

Level Controls (Displacement Type)

Supplier: Fisher

Source of Quote: Caltrol

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
LIC-HX1	HX-1 Condensate	1,290	16

INSTRUMENTS (CONTINUED)

Level Transmitters (Sonic Type)

Supplier: National Sonics

Source of Quote: Arens Industries

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
LT-V1	Flasher-Phase Separator L/S	1,920	16
LT-V1A	Auxiliary Phase Separator L/S	1,920	16
LT-V2	Melter/Condenser-Phase Separator L	1,920	16
LT-V2A	Auxiliary Phase Separator L	1,920	16

Interface Transmitters (Conductivity Type)

Supplier: Drexelbrook Engineering

Source of Quote: RPS Industries

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
IT-V1	Flasher-Phase Separator L/S	2,270	16
IT-V1A	Auxiliary Phase Separator L/S	2,270	16
IT-V2	Melter/Condenser-Phase Separator L	2,270	16
IT-V2A	Auxiliary Phase Separator L	2,270	16

INSTRUMENTS (CONTINUED)

Flowmeters (Variable Area Type - Rotameters)

Supplier: Fischer & Porter

Source of Quote: Fischer & Porter

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
FT-1	Flasher Feed Flow	1,150	4
FT-2	Concentrate Flow	1,150	4
FT-3	Product Flow	1,150	4
FT-HX2	Guard Condenser Flow	1,150	4
FT-4	Final Condenser Flow	1,150	4
FT-CW	Final Condenser Water Flow	1,160	5

Flowmeters (Magnetic Type)

Supplier: Fischer & Porter

Source of Quote: Fischer & Porter

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
FT-WF	LWF Flow to Flasher	2,600	10
FT-V1A	L/SWF Flow to Melter/Condenser	2,600	12

INSTRUMENTS (CONTINUED)

Differential Pressure Transmitters

Supplier: Foxboro

Source of Quote: Foxboro

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
FT-V2	Steam Flow to Melter/Condenser	1,140	10

Dial Thermometers

Supplier: Cole-Parmer

Source of Quote: Cole-Parmer

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
TI-T1	Feed Tank	80	1
TI-T2	Concentrate Tank	80	1
TI-T3	Product Tank	80	1
TI-T4	Condensate Tank	80	1

INSTRUMENTS (CONTINUED)

Temperature Transmitters

Supplier: Foxboro

Source of Quote: Foxboro

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
TT-T1	Feed Tank Temperature	1,190	4
TT-V1-1	Flasher Bottom Temperature	1,190	4
TT-V1-2	Flasher Second Temperature	1,190	4
TT-V1-3	Flasher Mid Temperature	1,190	4
TT-V1-4	Flasher Fourth Temperature	1,190	4
TT-V1-5	Flasher Top Temperature	1,190	4
TT-V1A-1	Aux. Phase Sep. L/S Bottom Temp.	1,190	4
TT-V1A-2	Aux. Phase Sep. L/S Top Temp.	1,190	4
TT-V2	Melter/Condenser Control Temp.	1,190	4
TT-V2-1	Melter/Condenser Bottom Temp.	1,190	4
TT-V2-2	Melter/Condenser Second Temp.	1,190	4
TT-V2-3	Melter/Condenser Mid Temp.	1,190	4
TT-V2-4	Melter/Condenser Fourth Temp.	1,190	4
TT-V2-5	Melter/Condenser Top Temperature	1,190	4
TT-V2A-1	Aux. Phase Sep. L Bottom Temp.	1,190	4
TT-V2A-2	Aux. Phase Sep. L Top Temp.	1,190	4
TT-HX-1	Final Condenser Condensate Temp.	1,190	4
TT-CWO	Cooling Water Outlet Temperature	1,190	4
TT-CWI	Cooling Water Inlet Temperature	1,190	4

INSTRUMENTS (CONTINUED)

Absolute Pressure Transmitters

Supplier: Foxboro

Source of Quote: Foxboro

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
PT-V1	Flasher-Phase Separator L/S	1,360	6
PT-V1A	Auxiliary Phase Separator L/S	1,360	6
PT-V2	Melter/Condenser-Phase Separator L	1,360	6
PT-V2A	Auxiliary Phase Separator L	1,360	6

Pressure Gages

Supplier: Ashcroft

Source of Quote: Cole-Parmer

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
PI-1	Feed Pump	80	1
PI-2	Concentrate Pump	80	1
PI-3	Product Pump	80	1
PI-WF	WF Pump	80	1
PI-4	Condensate Pump	80	1

INSTRUMENTS (CONTINUED)

Recorder-Controllers - Recorders

Supplier: Foxboro

Source of Quote: Foxboro

Basis of Inquiry: Catalog

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
LRC-V1	Flasher-Phase Separator L/S Level	2,330	8
FR-CW	Final Condenser Water Flow	360	0
LRC-V1A	Aux. Phase Separator L/S Level	2,330	8
FR-WF	LWF Flow to Flasher	360	0
LRC-V2	Melter/Cond.-Phase Sep. L Level	2,330	8
FR-V1A	L/SWF Flow to Melter/Condenser	360	0
LRC-V2A	Aux. Phase Separator L Level	2,330	8
IRC-V1	Flasher-Phase Sep. L/S Interface	2,330	8
FR-3	Product Flow	360	0
IRC-V1A	Aux. Phase Sep. L/S Interface	2,330	8
FR-HX2	Guard Condenser Flow	360	0
IRC-V2	Mel./Cond.-Phase Sep. L Interface	2,330	8
FR-4	Final Condenser Flow	360	0
IRC-V2A	Aux. Phase Sep. L Interface	2,330	8
FR-V2	Steam Flow to Melter/Condenser	360	0
FRC-1	Flasher Feed Flow	2,330	8
FR-2	Concentrate Flow	360	0
TRC-1	Feed Tank Temperature	2,330	8
TR-V1-1	Flasher Bottom Temperature	360	0

INSTRUMENTS (CONTINUED)Recorder-Controllers - Recorders (Continued)

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
TR-V1-2	Flasher Second Temperature	360	0
TR-V1-3	Flasher Mid Temperature	910	8
TR-V1-4	Flasher Fourth Temperature	360	0
TR-V1-5	Flasher Top Temperature	360	0
TR-V1A-1	Aux. Phase Sep. L/S Bottom Temp.	910	8
TR-V1A-2	Aux. Phase Sep. L/S Top Temp.	360	0
TR-V2	Melter/Condenser Control Temp.	2,330	8
TR-V2-1	Melter/Condenser Bottom Temp.	360	0
TR-V2-2	Melter/Condenser Second Temp.	360	0
TR-V2-3	Melter/Condenser Mid Temp.	910	8
TR-V2-4	Melter/Condenser Fourth Temp.	360	0
TR-V2-5	Melter/Condenser Top Temperature	360	0
TR-V2A-1	Aux. Phase Sep. L Bottom Temp.	360	0
TR-V2A-2	Aux. Phase Sep. L Top Temp.	360	0
TR-HX-1	Final Condenser Condensate Temp.	360	0
TR-CWO	Cooling Water Outlet Temperature	910	8
TR-CWI	Cooling Water Inlet Temperature	360	0
PRC-V1	Flasher-Phase Sep. L/S Press.	2,330	8
PR-V1A	Aux. Phase Separator L/S Press.	360	0
PRC-V2	Melter/Cond.-Phase Sep. L Press.	2,330	8
PR-V2A	Aux. Phase Separator L Press.	<u>360</u>	<u>0</u>
TOTAL INSTRUMENTS		\$103,760	537

CONTROL VALVES

<u>Tag</u>	<u>Service</u>	<u>Cost</u>	<u>Hours to Install</u>
TCV-CWO	Cooling Water from Final Condenser	670	2
LCV-V-1	L/SWF from Flasher	980	3
LCV-V-1A	L/SWF from Aux. Phase Separator	980	3
LCV-2	LWF from Melt./Cond.-Phase Sep.	980	3
LCV-V-2A	LWF from Aux. Phase Separator	980	3
TCV-T-1	Steam to Feed Tank	890	2
TCV-V-2	Steam to Melter/Condenser	890	2
FCV-1	Feed to Flasher	890	2
ICV-V-1	Concentrate from Flasher	890	2
ICV-V-1A	Concentrate from Aux. Phase Sep.	890	2
ICV-V-2	Product from Melter/Condenser	890	2
ICV-V-2A	Product from Aux. Phase Sep.	890	2
LCV-HX1	Condensate from Final Condenser	890	2
PCV-V-1	Bleed into Hi-Vac System	890	2
PCV-V-2	Bleed into Lo-Vac System	<u>890</u>	<u>2</u>
TOTAL CONTROL VALVES		\$13,490	34
TOTAL INSTRUMENTS AND CONTROL VALVES		<u>117,250</u>	<u>571</u>
TOTAL PURCHASED EQUIPMENT		\$44,820	810

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
PILOT PLANT COST ESTIMATE

STRUCTURAL

<u>Item</u>	<u>Quantity</u>	<u>Installed Cost</u>	
		<u>Unit</u>	<u>Total</u>
Standard Grating (SF)	70	\$30	\$2,100
Expanded Steel Grating (SF)	260	4	1,040
Ladder w/Cage (VLF)	50	70	3,500
Railings, Industrial (LF)	130	30	3,900
W6 x 9 Struct. Stl. Members (LF)	200	11	2,200
TOTAL			\$12,740

TUBELESS EVAPORATION PROCESS DEVELOPMENT
DOE CONTRACT No. DE-AC7-861D12619
PILOT PLANT COST ESTIMATE

PIPING

<u>Item</u>	<u>Quantity</u>	<u>Installed Cost</u>	
		<u>Unit</u>	<u>Total</u>
1 in pipe (ft)	600	\$12	\$7,200
2 in pipe (ft)	100	14	1,400
2 in elbows	70	43	3,010
3 in pipe (ft)	200	16	3,200
3 in elbows	30	59	1,770
Total FRP			\$16,580
1 in pipe (ft)	1,050	\$7	\$7,350
1 in elbows	110	23	2,530
1 in unions	110	27	2,970
1 in ball valves	80	23	1,840
1 in globe valves	1	50	50
1 in check valves	4	30	120
2 in check valves	1	70	70
2 in ball valves	20	52	1,040
3 in gate valves	56	185	10,360
Total Galvanized/Bronze			\$26,330
TOTAL PIPING			\$42,910

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