BUILD AND OPERATE A CLATHRATE DESALINATION PILOT PLANT

FINAL TECHNICAL REPORT

Thermal Energy Storage, Inc.
6362 Ferris Square, Suite C
San Diego, California 92121

by
Richard A. McCormack
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Assistance Agreement No. 1425-5-FC-81-20690

Water Treatment Technology Program Report No. 31

May 1998

U.S. DEPARTMENT OF THE INTERIOR
Bureau of Reclamation
Technical Service Center
Water Treatment Engineering and Research Group

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY ***(Leave Blank)***

2. REPORT DATE
May 1998

3. REPORT TYPE AND DATES COVERED
Final

4. TITLE AND SUBTITLE
BUILD AND OPERATE A CLATHRATE DESALINATION PILOT PLANT

5. FUNDING NUMBERS
Assistance Agreement No.
1425-5-FC-81-20690

6. AUTHOR(S)
Richard A. McCormack
Glenn A. Niblock

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Thermal Energy Storage, Inc.
6335 Ferris Square, Suite E
San Diego, CA 92121

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Bureau of Reclamation
Denver Federal Center
P.O. Box 25007
Denver, CO 80225-0007

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
Water Treatment Technology
Program Report No. 31

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Available from the National Technical Information Service, Operations Division,
5285 Port Royal Road, Springfield, Virginia 22161

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

This report presents the research, testing, and cost information developed in the design, construction, and operation of a clathrate desalination pilot plant. The plant was constructed on-shore at the Natural Energy Laboratory of Hawaii (NELH).

Construction of the pilot plant was completed in May, 1997, and the plant operated intermittently for 7 months, as budget allowed. The plant produced clathrate ice from seawater that yielded water having a salt content of 500 ppm, some 200 ppm lower than Colorado River water. Information obtained from the pilot plant was used to revise the capital and operating cost estimate for a commercial-sized plant and indicate that the costs reported previously were conservative.

Several dozen alternative clathrate formers were reviewed and seven candidates were found which have the potential for improving the performance of the system, and making operation feasible in warmer oceans.

14. SUBJECT TERMS- -
desalination/clathrate/freeze/pilot plant/ocean engineering/clathrate former/HCF R141B

15. NUMBER OF PAGES
109

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT
UL

18. SECURITY CLASSIFICATION OF THIS PAGE
UL

19. SECURITY CLASSIFICATION OF ABSTRACT
UL

20. LIMITATION OF ABSTRACT
UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. 239-18
298-102

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ACKNOWLEDGMENTS

Thermal Energy Storage, Inc. prepared the work reported herein under financial assistance agreement number 1425-5-FC-81-20690. Under this agreement the Bureau of Reclamation, United States Department of the Interior, provided financial support for the design construction, and operation of the plant.

Thermal Energy Storage, Inc. also acknowledges financial and technical contributions to this work from the following organizations:

Allied Signal Chemical Company, Fluorine Products Division, Morristown New Jersey and the Buffalo Research Facility, Buffalo, New York;
Jaeger Engineering, Inc., San Diego, California;
Makai Ocean Engineering, Inc., Makapuu Point, Oahu, Hawaii;
RAMCO Incorporated, San Diego, California;
The Natural Energy Laboratory of Hawaii, Keahole Point, Kailua-Kona, Hawaii,

and, the following individuals:

Richard K. Andersen, Consultant;
Larry W. Ellefson, PE; Consultant;
The late Wilfred Hahn PE; Consultant;
Richard Hargreaves, Site Coordinator;
James A Heist, PE; Consultant;
Frederic W. Jaeger, PE; Consultant;
Austin F McCormack, PE; Consultant;
Dr. Konstantin Oudatchin, Researcher, Canadian National Research Council;
Dr. John Ripmeester, Physical Chemist, Canadian National Research Council;

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1. EXECUTIVE SUMMARY

The development, design construction, and operation of a clathrate freeze desalination pilot plant were carried out under a firm-price cost-sharing contract with the Bureau of Reclamation. The results of that work indicate that the clathrate desalination process is technically feasible, and economically competitive with alternative technologies. Water obtained from clathrate crystals produced in plant operations was washed in a bench test, and had total dissolved solids of 500 parts per million, meeting EPA potability standards for salinity.

The pilot plant was constructed at the Natural Energy Laboratory of Hawaii (NELH). This facility, on the Kona coast of the big island of Hawaii, has close-to-shore access to deep-ocean water that is cold enough to spontaneously initiate clathrate crystals formation. The plant was sited there to make use of the excess capacity of the NELH deep-seawater pumping system for cooling the clathrate desalination process.

The plant was operated multiple times and produced significant quantities of clathrate crystals even though the plant was operated at temperatures higher than the design levels. The cooling water flow was less than the nominal design level, and the temperature of the cooling water was almost 0.6°C (1 “F) higher than expected.

Estimates for the capital and operating cost of a commercial-scale plant were revised based on the results of this work. The estimated cost of water from a commercial-scale plant with public financing would be from $0.46 to $0.52 per cubic meter ($1.75 to $2.00 per 1000 gallons). Because the cost of electrical energy assumed for the estimate was quite high, it is likely that the cost of fresh water would be slightly lower than these amounts. The costs with private financing would be slightly higher, $0.59 to $0.68 per cubic meter ($2.25 to $2.60 per 1000 gallons), but are still competitive when compared with conventional desalination processes such as distillation and reverse osmosis, and are also competitive with surface water sources.

Further, research into the application of a new clathrate former was completed and the results of tbat work indicate that clathrate formers other than the chemical (R141b) used in the pilot plant should result in performance improvements.

The operation of the plant made use of each of the key components except for the wash column. The design and construction of the wash column had been the responsibility of the late Mr. Wilfred Hahn. Mr. Hahn died before the wash column detail design was completed. His notes, calculations, and sketches were lost in shipment to Thermal Energy Storage Inc. (TESI). Eventually his computer calculations and computer-aided drawings were recovered from his computer but handwritten notes and sketches were unrecoverable. Although Mr. Hahn's experience would have permitted fabrication of the wash column by his company with the level of detail eventually recovered, with his expertise no longer available his company declined to fabricate the wash column. TESI proceeded with the analysis and design of the wash column but could not know if some detail Mr. Hahn intended to implement would have improved the performance of the wash column.

Initially there were several operating difficulties related to the compatibility of materials with the clathrate former R141b. The R141b flowmeter initially installed in the system contained an internal plastic membrane that malfunctioned almost immediately, and was replaced with a stainless steel turbine-type meter. The impellers in two pumps were affected by swelling and partial delamination and resulted in a shutdown for repairs. In addition, several small plastic valves were rendered inoperable by swelling of o-rings. These valves were replaced.

Piping system failures occurred in several locations that also led to cessation of operations. A defective crystallizer end cap failed catastrophically and was repaired temporarily with fiberglass reinforced plastic (FRP) in order to resume operations as quickly as possible. The repaired end cap failed after a short period and had to be replaced.

Several lessons were learned in the construction and operation of the plant. Even though a substantial effort was made in the design phase to assure the compatibility of all materials wetted with R141b several failures still occurred. A greater effort must be made to confirm compatibility of materials with R141b (or other clathrate formers) before components are selected. Since R141b is a solvent, if valves, pumps, and sensors cannot be tested as assemblies, then the elements wetted by R141b should be subjected to compatibility tests to assure long-term performance.

Although a conceptual design for a commercial-scale plant permits a reasonably good estimate of the capital and operating costs, the estimate has significant uncertainty. The results of the operation of the pilot plant indicate that the process is technically feasible. The preliminary cost estimate given here indicates that a commercial-scale clathrate desalination plant would be cost-competitive, and especially so if a higher temperature clathrate former can be employed. A more detailed design is warranted which would allow an improved cost estimate that would answer the question of the cost-competitiveness of the clathrate desalination process.

2. CONCLUSIONS AND RECOMMENDATIONS

2. I Conclusions

2.1.1 The construction and operation of a clathrate desalination plant on a commercial scale is technically feasible.

2.1.2 The clathrate former (R141b) selected for the pilot plant will form clathrate crystals with seawater at a temperature below 7.5”~ (45.5°F).

2.1.3 The clathrate former (R141b) selected for the pilot plant is suitable for coastal regions with access to seawater having a temperature of 6.1 ° C (43 °F) or less.

2.1.4 Where the available cold seawater is not significantly colder than 7.2°C (45°F), the initiation of crystallization can be improved by employing seed crystals to provide a predictable nucleation source to start the system.

2.1.5 The clathrate former (R141b) is incompatible with certain plastic materials, which make them inappropriate for long-term use.

2.1.6 Alternative clathrate formers are available which appear to provide performance superior to R141b and extend the geographic applicability of the process.

2.1.7 The clathrate crystals can be ‘grown” in the crystallizer heat exchanger to a size necessary to improve their separation from the brine.

2.1.8 The crystallizer heat exchanger, and other components wetted by the R14 1 b, can be fabricated of high-density polyethylene (HDPE).

2.1.9 A crystallizer heat exchanger fabricated of HDPE for a commercial size plant of 13.6 million liters (3.6 million gallons) per day will require a heat transfer surface area of about 140,000 square meters (1.5 million square feet) when using cooling seawater at 6.1 °C (43 “F).

2.1.10 The design of a cost-effective commercial plant crystallizer will incorporate different materials and improved design parameters.

2.1.11 The clathrate desalination process yields crystals that have minimal seawater inclusions.

2.1.12 Crystals produced by the pilot plant washed in a bench scale process yielded water with total dissolved solids (salinity plus trace materials) of 504[1] ppm.

2.1.13 The function of the wash column, to separate and wash the clathrate crystal, was not satisfactorily demonstrated in plant testing.

2.1.14 Attempts to measure the crystal size with a microscope were made in San Diego after

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[1] Pacific Treatment Analytical Services, Certificate of Analysis, January 16, 1998

gathering clathrate slurry samples in Kona. Most of the samples were consumed in the bench washing process. Attempts to measure the crystal size in samples remaining were unsuccessful.

21.15 The crystals were **difficult** to keep in suspension in the wash column indicating that they were larger than the design crystal size and had lower relative drag than the crystals anticipated in the design.

2.1.16 The optimal method for separating the clathrate crystals from seawater and for washing seawater from its surface was not determined.

2.1.17 The results of the pilot plant design and construction indicate the need for several design changes in a commercial-scale plant, some of which would increase capital cost above that anticipated in the prehminary research study.

2.1.18 The electrical energy costs previously assumed for the commercial-scale plant were unreasonably high, and inconsistent with the prices anticipated as energy markets respond to deregulation.

2.1.19 The net unit cost of water produced by a clathrate desalination plant would be commercially competitive with that produced by other desalination alternatives.

2.2 Recommendations

From these conclusions, the following recommendations are made:

2.2.1 Operate the pilot plant for a further period of 6 months to evaluate the performance of the wash column and other components, and to determine the conditions under which the system can operate optimally.

2.2.2 Evaluate alternatives to the wash column to find whether a more effective means of separating **clathrate** crystals from seawater, and removing brine **from** its surface, can be found.

2.2.3 Determine the **compatibility** of common and inexpensive construction materials suitable for a commercial plant with other candidate clathrate formers.

2.2.4 Continue research and development to investigate higher temperature clathrate formers.

2.2.5 Prepare a comprehensive prehminary design for a commercial-scale clathrate desalination plant implementing the lessons learned from the pilot plant.

2.2.6 Prepare a detailed cost estimate and pro-forma financial statements for a privately financed commercial-scale plant.

3. BACKGROUND AND INTRODUCTION

The purpose of this work was to test the clathrate freeze desalination process in a pilot facility, and to apply the knowledge gained to estimate the cost of a commercial-scale plant. The pilot facility was constructed at the Natural Energy Laboratory of Hawaii Authority's facility on the Kona coast of the big island of Hawaii. Testing sufficient for the intent of the pilot program was completed and the results are incorporated in the work reported here.

3.1 Technology of Clathrate Freeze Desalination

The technology for obtaining fresh water from the ocean to date has been limited to high-cost processes-distillation and reverse osmosis -with limited applications. Only populations willing and able to pay a high price, principally the Middle East and the Pacific islands, have made use of these technologies. Desalination processes using reverse osmosis technology produce water in quantity in Southern California at prices ranging from $1.06 to $1.59 per cubic meter ($m^{3}$) ($4 to $6 per 1,000 gallons). At these prices water is prohibitively expensive fix municipal and county water authorities. To be competitive with municipal water systems in Southern California, desalination technology must produce fresh water at approximately $0.53/$m^3$ ($2 per 1,000 gallons or about $650 per acre-foot).

Water conservation, wastewater reclamation, and seawater desalination offer a means to avert water crises and foster good government planning. Water conservation and wastewater reclamation are being promoted and applied, but low cost desalination remains an elusive goal. The principal means of reducing the cost of seawater desalination is through the reduction in the level of energy used in the process. A technology for clathrate desalination, developed by Thermal Energy Storage, Inc. (TESI) over the last decade, offers the potential of substantially reducing power consumption while maintaining a comparable capital cost for a commercial desalination plant.

A clathrate is an aggregation of water molecules surrounding a central, small, non-water molecule, to form an ice-like crystal. The Department of the Interior first investigated clathrate desalination over 20 years ago. That work yielded significant accomplishments and indicated the value of lower energy use offered by the technology. With the demise of the Office of Saline Water (OSW) and the Office of Water Research and Technology (OWRT) within the Department of the Interior, work ceased on clathrate desalination. The background on this work of these agencies is discussed in some detail in a prior report[2] by TESI.

In the years since the OSW's work, reverse osmosis technology became accepted as the prime method of producing fresh water from seawater, joining distillation as an effective if somewhat expensive technology for this purpose. The reverse osmosis process is energy-intensive, as is distillation, so interest in other more energy economical technologies such as clathrate desalination has continued.

TESI is expert in clathrate (or gas hydrate) technology and has found clathrate formers that induce clathrate formation at temperatures well above the normal freezing point of ice. This leads to two economies. First, if cold seawater provides the refrigeration used to accomplish freezing, less energy is required because little energy is used to pump the seawater and mix it with the clathrate former. Second, in the desalination application, clathrate crystal production creates the potential for using deep-ocean water as the heat sink reducing the amount of heating surface required.

TESI also uses clathrates to store thermal energy during the night for use in air conditioning during the day, thus reducing electrical peak demand requirements for electric utilities and reducing electric costs for their customers. TESI has marketed this product as the 'SNOPEAK[3] "warm ice" thermal energy storage system The clathrate formation process was developed and refined over a ten year period of research, testing, and pilot work completed in early 1993, during which time TESI expended some $4 million of internally-generated funds.

With the assistance of the Canadian National Research Council and the Buffalo Research Laboratory of Allied Signal Company, and the several organizations mentioned in the acknowledgements, TESI has advanced clathrate desalination technology substantially in the period since 1993.

The company holds patents on a process to produce the clathrate crystals from the cold water

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[2] Richard A. McCormack *and Richard K. Andersen, Clathrate Desalination Plant Preliminary Research Study,* June 1995, U.S. Department of the Interior, Bureau of Reclamation, Water Treatment Engineering and Research Group, Water Treatment Program Report No. 5, June 1995

[3] A trademark of Thermal Energy Systems, Inc. for an air conditioning peak load shaving system. 'Snopeak is a partial contraction of "there's no peak."

at ocean depths of 610 meters (2,000 feet) or more. The elimination of active refrigeration from the process yields a substantial reduction in energy costs.

3.2 Choice of Clathrate Former for the Pilot Plant

A clathrate is a lattice-like structure in which molecules of one substance are enclosed within the crystal structure of another substance. There are many types of clathrates. The type of clathrate of interest for desalination is a compound of water and another suitable molecule that will form crystals at an elevated temperature while meeting all the environmental criteria that govern chemical use.

Water combines with a multitude of clathrate formers to form a clathrate crystal at various pressures and temperatures, in both liquid and gaseous form. Both inorganic and organic compounds form clathrates with various degrees of toxicity, flammability, and other characteristics.

TESI developed the criteria given in Table 3-1 to define the desirable attributes of a clathrate former suitable for the desalination application.

Table 3-1 Selection Criteria for Clathrate Former

Attribute	**Criteria**
Environmentally acceptable	Approved by the EPA for continued production and use
Non-toxic	Low acute toxicity, non-carcinogenic and non-mutagenic
Non-flammable	High flash point temperature, self-extinguishing if ignitable
Stable	Slow decomposition or deterioration
Low cost	A Class II clathrate former, readily manufactured in commercial quantities, Liquid over the operating temperature range at atmospheric pressure
Suitable transition temperature	56°C to 29.4°C (42°F to 85°F)
Suitable operating pressure	100 to 710 kPa (1 to 7 atmospheres)
Compatible with standard materials	Chemically inactive

In work of Thermal Energy Storage, Inc. (TESI) cited previously, Dr. John Ripmeester of the National Research Council of Canada (NRCC), with the support of Allied Signal's Buffalo Research Laboratory, investigated more than 20 compounds in search for useful clathrate formers for thermal energy storage facilities. TESI reviewed this work to determine which of these clathrate former-s would be appropriate for desalination The following three clathrate formers were selected as primary candidates for the pilot plant.

- R141b (Dichloromonofluoroethane - CCl_2FCH_3)
- Carbon Dioxide - (CO_2)
- R22 (Chlorodifluoromethane - $CHClF_2$)

R141 b is a compound manufactured for the rigid foam insulation industry by two large domestic suppliers as well as several non-domestic suppliers. R141b was found to be the best candidate for deep ocean desalination and air conditioning applications where water temperatures below 6°C (43 °F) are available. One of the important characteristics of this chemical is its relatively low vapor pressure at the temperatures used for the desalination pilot plant. The atmospheric pressure boii temperature is 32°C (90°F) making it possible to use this clathrate former as a liquid in a low pressure surface unit made of high density polyethylene and PVC piping. These materials were the least expensive corrosion resistant materials available for use in the construction of the pilot plant.

Data from NRCC laboratory tests showed that R141b produced a clathrate with distilled water at approximately 8.6°C (475°F). The data indicated that the freezing temperature would be depressed to about to 7.5°C (455°F) when the clathrate forms from seawater. R141b is a clear, colorless liquid with a faint ethereal odor[4]. It is thermally stable at normal conditions and is not considered a hazardous waste under the Resource Conservation and Recovery Act (RCRA). It has an intrinsically low toxicity with a permissible exposure limit of 500 parts per million. The compound is considered nonflammable in the liquid state as defined by the Department of Transportation and the National Fire Protection Association. It is not regulated as a hazardous material. In the vapor state, ignition is difficult. Thus, R14 1 b met all of the desired criteria with the exception that the vapor, although having no flash point, is slightly flammable. The principal disadvantage of R141b is that it is relatively expensive. This resulted in a need for additional plant equipment for recovery of the R141b for reuse as well as to assure an essentially zero level of R141b dissolved in the product water

3.3 Pilot Plant Site Selection

Three of many sites available for the Clathrate Desalination Pilot Plant were considered in

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[4] Allied Signal, R141b Material Safety Data Sheet

detail in the cited study that preceded this work. These sites were selected for detailed evaluation of **suitability,** ocean pipeline costs, and potential for funding. The criteria for selecting sites were included the following which were considered of paramount importance:

- The site has (1) **immediate** deep-water ocean access, (2) space suitable for all equipment, (3) **an** environmentally acceptable method of discharging the water, and (4) capability to obtain all permits at low cost. Therefore the pilot plant is best located within a facility that has similar operations and existing permits.
- An ocean depth of 610-m **(2,000-ft.),** or more, that is reasonably close to shore. Pipeline costs are **minimized** where this is the case. The pipeline must deliver sea water at less than 7°C (45°F) to the on-shore clathrate desalination pilot plant thus, it is necessary to find a location where the pipeline length is not excessive.
- The pilot project will likely require funding in excess of that available from the Bureau of Reclamation, and various governmental groups offer this potential funding if the pilot plant is located at a site suitable to them.

The three sites selected for **evaluation** were:

- The Natural Energy **Laboratory** of Hawaii **(NELH)** located at Keahole Point near **Kailua-Kona** on the **Kona** Coast of the Big Island of Hawaii;
- The Naval Auxiliary Base on San Clemente Island located approximately 70 miles [113 km] off the coast of San Diego, California; and
- Scripps Institute of Oceanography located at La Jolla, a suburb of San Diego.

The site at the NELH was selected as the **preferred** site, based primarily on an existing assured source of deep-sea water at the Hawaiian location. This avoided the expense of **installing** a deep-sea pipeline, which would have been required at the other two sites.

The Natural Energy Laboratory of Hawaii *Authority* ***(NELHA)*** *. - The NELHA manages the Natural* Energy **Laboratory** of Hawaii **(NELH)** and **associated** operations. The NELH **facility** *covers 352* hectares (870 acres or 1.36 square miles), including the Hawaii Ocean Science and Technology (HOST) Park. The **NELH** provides facilities to support many types of research, pilot, and commercial projects in ocean and solar technologies. The Keahole Point location provides close access to pristine deep and **surface** seawater and exceptional weather conditions. Private, corporate, governmental and academic sectors use these resources in innovative projects both in research and applied technologies. **NELH** maintains an infrastructure deemed suitable for the pilot plant, and provides a wide variety of support services that can be rented as required for the project.

The slope to the ocean floor is steep at Keahole Point, allowing access to deep-sea water relatively close to shore. NELH operates and maintains pipelines and pumping stations that access deep-sea water from 675 meters (2,215 feet) with temperatures at or below 6.1 °C (43 “F). There are three deep-sea pipelines in operation with a total capacity of 65,000 liters (17,100 gallons) per minute. The largest pipeline is a 102 cm (40-inch) diameter polyethylene line running to a depth of 640 meters (2,100 feet) that provides 5 1,000 liters (13,400gallons) per minute of cold seawater. The lower portion of the ocean pipeline is an inverted catenary, 1020 meters (3,337 feet) long, that floats some 30.5 meters (100 feet) above the rough bottom. The design also permits the pipeline to move as much as 152 meters (500 feet) horizontally and 76 meters (250 feet) vertically to accommodate ocean currents.

Other significant advantages of the Hawaii location included: 1) the certainty that sea water would be delivered at less than 7°C (45°F); 2) low seawater cost; 3) a permitted site; 4) availability of support services; and 5) a strong expression of interest in the technology by NELHA. The primary disadvantages, the more difficult logistics and project management, were outweighed by the advantages.

Environmental Considerations - The environmental impacts with respect to placing the pilot plant at the NELH site include:

- Land Use: NELH is an approved research and development facility that has responsibility for control of land use. The Laboratory is located in a remote area on dedicated land. Space for the pilot plant would be rented.
- Geology, Soils, and Seismic Hazards: The pilot plant will be located at existing facilities so that no additional development of land will be required. Much of the equipment will be skid-mounted to permit easy dismantling of the facility with no significant cleanup and restoration required. Seismic hazards are not a serious problem since rupture of the equipment piping would not release any significant amount of R141b and any material released can be easily recovered and any residual material will quickly evaporate.
- Groundwater Resources: No groundwater resources are involved in the operation of the pilot plant. No wastes will be discharged to the soil except for minute amounts of R141b that will be mixed with huge volumes of seawater from all NELH operations that are discharged onto lava rock beds to evaporate and percolate back to the sea in an approved manner. The R141b is non-toxic and will quickly evaporate on the lava rock.
- Marine Water Quality: The pilot plant will utilize 500 liters (132 gallons) per minute from an NELH deep-sea system pumping 65,000 liters (17,100 gallons) per minute; this is less than one percent of the total. All water pumped is pristine seawater. The pilot plant

produces no concentrated brine since the fresh water is not retained and all water employed is returned to the sea.

- Marine Biological Resources: The deep-sea pipes used by NELH draw water from the photic zone where the water has been out of contact with the surface for centuries. Therefore it contains few living plants and animals. Thus, the pumping of deep-sea water has essentially no effect on marine biological resources.
- Water Quality: Water quality is not a problem since the fresh water product from the pilot plant is not used and is discharged with the seawater on the lava rock beds.
- Noise: The plant is small and generates little noise and the site is remote.
- Liquid and Solid waste: The cost of the clathrate former makes it economical to recover 99.99 percent of the R141b. Approximately 0.12 liters/day (0.032 gallons/day) of R141b is lost to the seawater or the atmosphere while the pilot plant is operating. The R141b quickly evaporates, is non-toxic and is not a hazardous waste. Thus, there are essentially no liquid or solid wastes generated by the plant.
- Energy: Energy requirements for the pilot plant are insignificant compared to the pumping power already employed at the Laboratory.
- Electromagnetic Fields: A minor increase in electromagnetic fields will arise from the pumps in the pilot facility but this is insignificant compared to other NELH sources.
- Vis and Aesthetics: The NELH facilities are designed for hosting research and pilot operations and its remote location does not require special treatment.

4. PILOT PLANT DESIGN

The de&nation plant is **described** here, generally in its form at the conclusion of the project, but with some background material included to provide context for the description. The plant departs in some **significant** ways from the design anticipated in the prior work funded by Reclamation. Many of the changes were made in response to funding limitations that precluded some of the concepts **discussed** in Reference 1. The primary conceptual **difference** from the prior work was the use of crystallizer heat exchangers installed at the surface. **Installation** of the crystallizer heat exchanger in the NELH intake piping, or at depth, was deemed impracticable for the pilot plant.

4.1 General Arrangement at the NELHA Site

The pilot plant was constructed at the Natural Energy Laboratory of Hawaii adjacent to the **Keahole** Point lighthouse. The general arrangement of the facility, including the location of the major components, is given in Fii **4-1**. The largest components, the crystallizers were placed outside of the fenced part of the NELHA facility. The crystallizers and the ice melter heat exchanger were located along the fence line in a north-south orientation. A location inside the fence that had been considered in initial design analysis became unavailable so this altemative location was selected. Although this was not the first choice, it permitted placing the **crystallizer** heat exchangers in a relatively convenient location that required only **fill** and grading avoiding other expensive site modifications.

One disadvantage of the location was that the crystallizer length was constrained by NELH system piping and valves at the South end, and a need for access through a gate at the North end. The crystallizer nominal length of 76 meters (250 feet) was reduced to 69 meters (225 feet) to lit into the space available. At the time of this decision, the heat **transfer** surface area was thought to be substantially larger than was needed.

The site configuration before starting construction of the pilot plant is shown in photographs included as Figures 4-2 through 4-5. The photograph in Figure 4-2 is a view along the **North-**South fence line that shows the area for the crystallizers to the **left** of the fence, and for the balance of the plant on an existing concrete pad (to the right inside the fence). The gate at the fence comer provided access to plant equipment outside the fence. The area for the **crystallizers,** between the fence and the large pipe at the left, is shown more clearly in Figure 4-3. This photograph also shows the horizontal NELH pipe at the Southern limit of

Figure 4-1: General Arrangement of Plant at NELH

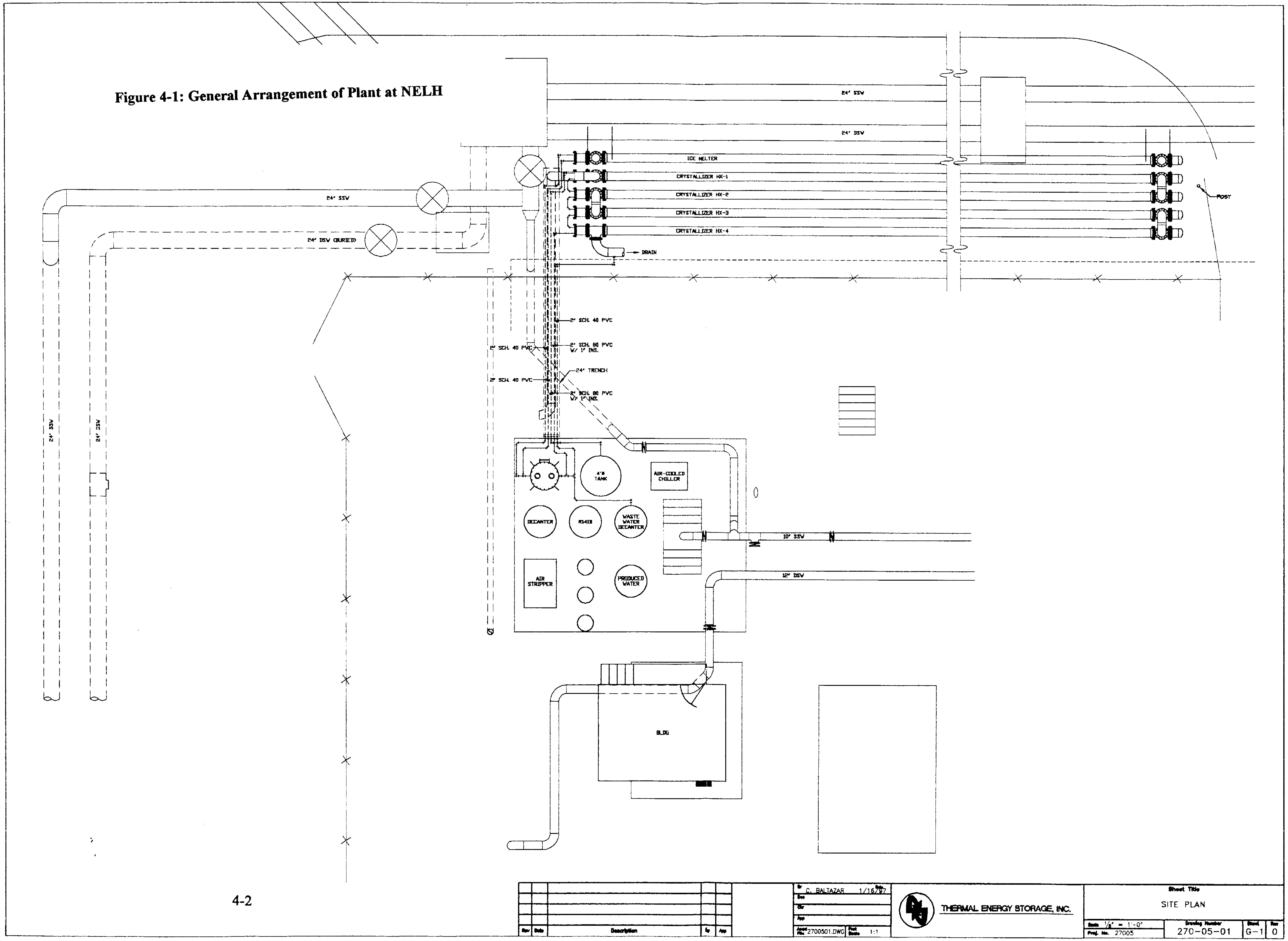


Figure 4-2: View North along NELH West fenceline

Figure 4-3: View of Crystallizer Laydown Area and Pedestal

useful area. The presence of this pipe created a difficulty in using this area for the crystallizers in that the area North of the existing NELH horizontal pipe was not level, and required substantial fill just to establish a uniform slope. The NELH pipe could not be removed so the alternatives were: 1) to bridge the pipe and fill on the South side; 2) to provide enough fill to cover the pipe, and build full-length crystallizers; or, 3) to fill to within a meter (3 feet) of the pipe and reduce the length of the crystallizers. Sufficient fill to level the site was prohibitively expensive so a decision was made to provide a uniform slope for the crystallizers and to limit their length to avoid having to go over the NELH pipe.

A view from inside the gate given in Figure 4-4 shows the existing concrete pad area that was eventually used for the balance of plant equipment. Most of the pumps and tanks were installed on an existing concrete pad A small office was moved adjacent to the concrete pad to serve as the project office. The project office building was placed at the East side of the concrete pad over the existing cold water piping (large blue pipe) shown in Figure 4-4. Another photograph (Figure 4-5), taken from inside the gate after the office building was in place, shows the proximity to the Keahole Point lighthouse.

4.2 General Process Flow Description

The pilot plant forms clathrate crystals by mixing a clathrate former (Refrigerant R141 b) into cold seawater 5.6°C (42 °F) while removing the heat of formation in a heat exchanger. The dimensions of the crystal lattice of the clathrate are such that the presence of salt and other minerals in ice is essentially zero. The clathrate formation process thus excludes salt, leaving a clathrate crystal product that is essentially pure water and the clathrate former. The heat of fusion of the clathrate is only slightly less than that of pure water ice, and is removed in a shell and tube heat exchanger with the cold seawater. This occurs in the crystallizers that are shown in the process and instrumentation diagram (P&ID) given in Figure 4-6. In a commercial design the crystallizers likely would be placed in the deep ocean and would be configured differently than the design for this surface plant.

The clathrate forms a slurry in the seawater from which it is grown, slightly increasing the salt content of the remaining liquid. The slurry is transported through the tube side of the heat exchanger to the wash column. The wash column is designed so the clathrate crystals agglomerate into a permeable “ice pack.” This clathrate crystals pack would be contained in a center tank in the wash column. The “ice pack” is raised by differential pressure as it grows by accumulation from the bottom. As the pack is raised, the top of the pack is scraped off into an annular tank that surrounds the center tank.

Figure 4-4: Concrete Pad used for Balance of Plant Equipment

Figure 4-5: Proximity of Site to Keahole Point and Lighthouse

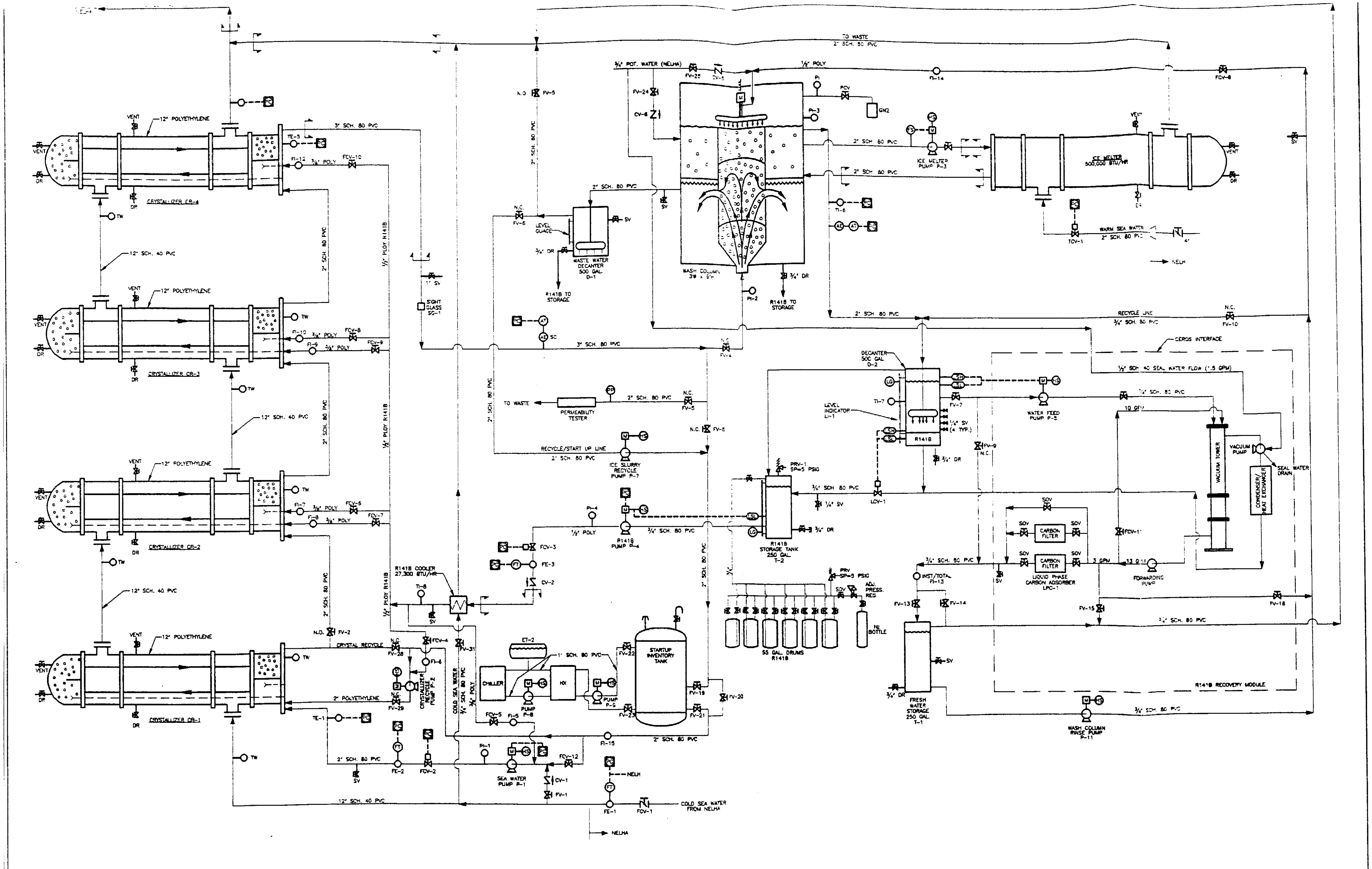


Figure 4-6: P&ID Diagram

Rev	Date	Description	By	App
1	1/16/97	TANK SIZE REVISION		

Dr C. BALTAZAR 7/15/96
Des L. ELLEFSON 7/15/96
Chk
App
Acad File 2700201.DWG
Plot Scale 1:1

THERMAL ENERGY STORAGE, INC.

Sheet Title
PIPING DESALINATION PILOT PLANT

Scale NONE
Proj. No. 27002
Drawing Number 270-02-01
Sheet PI-1
Rev 1

In the inner tank, the crystals are washed by a downflow of product water to remove brine water from the crystals. The clathrate crystals scraped into the annular tank create a slurry of product water and clathmte. The slurry in the annulus is melted by recirculating the slurry through the annulus and through an ice melting heat exchanger. The ice melting heat exchanger is essentially identical to the crystallizer but uses warm surface seawater to melt the slurry of clathrate crystals.

The product water flows from the annulus to a decanter where most of the R141 b is separated out by gravity for recovery and is returned to the R141b storage tank for reuse.

The product water flows from the decanter to an R141b recovery module. In this module R141 b is recovered in a vacuum distillation process which reduces the dissolved R14 1 b level to about 80 parts per million (ppm). This additional small quantity of recovered R141b is returned to the R141b storage tank for reuse. The product water then is polished with a carbon bed adsorber and delivered to a fresh water storage tank. The carbon adsorber reduces the R141b from 80 ppm to levels at or below those consistent with potable water, around 0.020 ppm.

4.3 Subsystems Description

The system consists of several major subsystems and components, including the crystallizer and ice melter heat exchangers, the wash column the R141b storage and delivery module, the R14 1 b recovery module, and the potable water cleanup and storage subsystem.

Crystallizer heat exchangers. This subsystem consists of four crystallizer heat exchangers. The heat exchangers are shell and tube type constructed of a polyethylene outer shell and polyethylene tubes. The tube sheets and end boxes are constructed with PVC plates and piping. Details of the crystallizer construction are shown in Figure 4-2.

The function of the crystallizer heat exchangers is to remove the heat of fusion of the clathrate crystals, while providing sufficient residence time for crystal formation and growth. Heat is removed by cold seawater 56°C (42°F) flowing through the shell side of the heat exchangers. The shell side flow is in series through the four heat exchangers and there is a total temperature rise of less than 0.6°C (1 “F). The cooling sea water flow is manually controlled to be 3,800 to 4,200 liters (1,000 to 1,100 gallons) per minute. The cooling water flow is mixed with brine from the wash column and flows into existing drams.

The product clathrate crystals are formed inside the tubes of the crystallizer heat exchanger

(CHX). The heat exchanger is configured with two tube passes. Refrigerant R141b is injected into the primary pump upstream of the CHX-1. The temperature of the seawater is lower than the clathrate crystal formation temperature and crystal formation begins spontaneously. Small crystals form, creating a slurry of clathrate crystals, seawater, and unreacted R141b. The mixture flows from the end box through the tubing and through a tube sheet at the far end of the heat exchanger. At the far end, the mixture flows into a smaller return end box and back into the second pass tubes. It then flows into the crystallizer inlet/outlet end box. The outlet side of the inlet/outlet end box is connected to the inlet side of the CHX-2 inlet/outlet end box. Each of the crystallizers have ten tubes in parallel and each crystallizer is connected in series.

The crystallizers have provisions for injection of R14 lb at multiple locations, and provisions for recycling crystals to permit additional crystal growth. The recycling can occur at the first crystallizer, or the entire product stream can be recycled in a startup mode. The tube sheet at the first crystallizer was constructed to permit creation of multiple passes in that heat exchanger should test results indicate that would be beneficial.

Wash column and ice melter heat exchanger. From the crystallizers the slurry of seawater and clathrate crystals flow either to the wash column, or is recycled for additional crystal growth. The latter condition is both a startup mode of operation, and a means of providing seed crystals to continue the process. In normal operation the majority of the slurry flows to the wash column. The wash column consists of a fiberglass reinforced polyethylene inner tank and a steel annular tank that is divided into upper and lower chambers.

The slurry flows into the inner tank of the wash column and agglomerates into a permeable "ice pack." The "ice pack" is raised by differential pressure against a scraper, which also contains a rinse water spray bar. The rinse water flows down through the ice pack and washes the saline water from the surface of the crystals. The rinse water and the saline water exit through holes in the inner tank into the lower annular tank. The saline water exits the inner tank, is mixed with the cooling water flow, and flows into existing drains. Product water flows from the wash column upper annular tank to the fresh water decanter.

The crystals are scraped into the upper annular tank and form a slurry with product water. The slurry is pumped through an ice melter heat exchanger and back to the upper annular tank The ice melter heat exchanger uses warm surface seawater 22°C (72°F) to provide the heat to melt the clathrate crystals. The ice melter heat exchanger is essentially identical to the crystallizer heat exchanger. Because the temperature difference available for heat transfer is much greater, a single heat exchanger suffices.

The fresh water decanter permits gravity separation of R141b, which is more dense than water, from the product water. The R141b then flows back to the R141b storage tanks. The product water flows into the R14 1 b recovery module.

R141b storage and delivery. Commercially pure R141b is stored in the 500-lb. drums used for delivery. Air overpressure transfers the R141b from the storage tank via a dip tube to the subsystem R141b storage tank. This tank contains makeup R141b, recovered R141b, and also water. There is a small amount of water dissolved in the R141b and a layer of water several centimeters deep floating on the R141b to minimize evaporation. The storage tank is also cooled with deep seawater flowing through a polyethylene coil. In normal operation., the R141b is pumped from the storage tank through a flowmeter and then through a small heat exchanger which cools it to near 5.6°C (42°F) and then into the inlet to the primary seawater pump.

R141b recovery module. The R141b recovery module consists of a 25 foot tall vacuum distillation tower, the associated vacuum pump, a low head forwarding pump, condenser, and the various valves, gauges, and flowmeters needed to control its operation. Product water from the decanter is pumped into the top of the vacuum tower through a spray nozzle. The water flows over a packing that assures a large surface area is exposed to the low pressure inthetower. The vacuum pump maintains the absolute pressure in the tower at about 74 mm Hg. (1.4 psia) promoting the removal of the higher vapor pressure R14 1 b from solution in the product water.

Potable water cleanup and storage. The final polishing of the product water is accomplished with activated carbon adsorberbeds. Two adsorbtion modules are arranged in parallel so that the system can operate continuously. The carbon beds reduce the R141b content to about 0.020 ppm. At present there are no potability standards for R141b but TESI anticipates standards that are comparable to those for other hydrochloroflourocarbons.

4.4 Plant Design Analysis

The crystallizer heat exchangers, the wash column, and the R141b recovery module required the major portion of the design analysis work. The funding under the Bureau of Reclamation contract provided for the design and construction of the crystallizer heat exchanger, the wash column and associated pumps, instruments and controls. These components/subsystems are unique to the pilot plant and must function for the plant to operate on a continuous basis.

4.4.1 Crystallizer Heat Exchanger (CHX) Thermodynamics and Heat Transfer.

The selection of R141b as the clathrate former gave a design clathrate formation temperature of about 7.5°C (45.5°F). Clathrate formation temperatures of 4.4°C (40°F) or higher are desirable for de&nation processes to allow for both (1) higher temperature seawater to form the clathrate crystals and (2) the relative ease of melting the clathrate to form fresh water.

The initial concept for the system anticipated that the clathrate former would be diffused from an inner coaxial pipe into seawater flowing upward in the annulus between the inner and outer pipe. The seawater would be drawn from a depth of 610 meters (2,000 ft.), and would form clathrate crystals as it was pumped to the surface. At a 2,000-foot ocean depth the temperature of the water is approximately 5.6°C (42°F) and the pressure is approximately 6,205 kPa (900 psig) providing conditions for clathrate crystal initiation and growth.

A major advantage of the injection of the clathrate former at the 610 meter (2,000-ft.) depth is that the latent heat of fusion to form the clathrate can be rejected during the upward flow of the seawater slurry to the surface. The second major advantage is that larger crystals can be formed due to (1) the increased retention time by virtue of the length of the pipe and (2) heat removal over the length of pipe in which clathrate formation occurs. The amount of seawater required and the pumping power consumed to produce a unit of fresh water is thus greatly reduced.

The R141b clathrate former produces a Type II clathrate structure. This structure results in 17.2 moles of water per mole of clathrate former. In ocean water recovered from the 2,000-foot level, the forming agent produced clathrate at 8.6°C (47.5°F). Thus, the 5.6°C (42°F) seawater temperature at a 610 meter (2,000-foot) depth would provide about a 3.1 °C (5 °F) temperature differential.

The configuration of the pilot plant was brought by practical considerations to a design with a surface heat exchanger simulating the undersea thermodynamics and heat transfer. Some small advantage in formation temperature would be lost by the lower pressures at the surface, but this was not thought to be highly significant.

The surface area required for heat transfer was driven by the amount of heat released by the formation of clathrate crystals. The plant size was set to produce about 21.2 liters (5.6

gallon) per minute of product water. The heat load on the crystallizers at that production rate is about 527 Megajoules (500,000 BTU) per hour. This heat load and the heat transfer coefficients established the heat transfer area required as about 315 square meters (3,400 square feet). These calculations are shown in detail in Appendix A.

Given that a 2.54 cm (l-inch) diameter tube provides about 0.08 square meters per meter (0.26 square feet per foot) of length, the heat load required a total tube length of about 3960 meters (13,000 feet). The space available at NELH would not provide room for this much heat exchange surface unless a multiple tube heat exchanger design was employed, NELH indicated a **willingness** to contribute about 366 meters (1200 feet) of 30 cm (12 inch) HDPE pipe that could be used as the shell for a shell and tube heat exchanger. A shell of this size would readily accept multiple tubes, and it was clear that a shell and tube configuration could meet the area requirement.

Initially, several continuous shell configurations were considered, including a **"hairpin"** and a circular layout. Because of a variety of uncertainties regarding the assembly of the **crystallizer** heat exchangers, TESI opted for a multi-pass shell and tube design which would use the 30 cm (12 inch) HDPE piping provided by **NELH**.

The design called for four **250-foot** long heat exchangers, each containing 20 tubes in a **two-pass internal configuration**. This gave a heat transfer area of about 485 square meters (5,200 square feet), providing substantial margin to accommodate uncertainties in the heat transfer characteristics.

Subsequently, after a site visit, it became apparent that the site tentatively selected for the **CHXs** would permit 250 **foot** long **crystallizers** only **if the** crystallizers were placed above the **existing NELH** supply line shown in the Figure 4-4 photograph. About 70 meters (225 feet) were available for the **CHXs** between the gate at the North end of this area and the pipe at the **Southern** end. Although that would reduce the heat transfer surface to about 437 square **meters (4,700** square feet), it still provided an excess of heat transfer surface of more than 33 percent. This excess heat **transfer** area was considered helpful in promoting continuing growth of the clathrate crystals **after** the initial **formation**.

Increasing the size, or “growing” the crystal, is desirable since the size of the wash column is determined by crystal size. This relationship was determined from tests conducted at the Wrightsville Beach Test Facility. Several factors are involved in this process, including increased retention time at formation temperature, minim&d supercooling adequate rate of heat removal, and appropriate nucleation.

The crystallizer design included end boxes that contained a tube sheet and appropriate manifolds. At one end the crystallizer design provided for return flow so each CHX was a two-pass heat exchanger. one pass was co-current flow and the second was counter-current. The details of the crystallizer ends are given in drawings provided in the design Appendix.

4.4.2 **Wash Column Design.**

Work by the Office of Saline Water (OSW) and the Office of Water Research and Technology (OWRT) had previously demonstrated that crystal size was a key element in washing a clathrate crystal. The size of the clathrate former molecule is 5 angstroms. Ideal crystal size is in excess of 400,000 angstroms or, since there are 1000 angstroms in a micron, 400 microns. Work performed at Wrightsville Beach suffered from small crystal size on the order of40 microns, or one-tenth the desired size. Small crystal size fosters high interstitial water quantities and low yields while large crystals foster high yields approaching 100 percent for the desalination process. Larger crystal size is important in the thermal energy storage process as well. TESI has successfully grown crystals in its Clathrate Test Facility well in excess of 400 microns.

The design of this key component was subcontracted to Exstar International Corp., primarily to obtain the services of Mr. Wilfred Hahn. Mr. Hahn had been intimately involved in the Wrightsville Beach work and had both empirical data and analytical models for sizing the wash column for specific criteria.

Mr. Hahn completed an initial analysis based on a crystal size of 300 microns in 1996, and prepared preliminary design drawings. His company, Exstar International, also prepared a bid that was considered competitive given the criteria, but in the context of the project budget was too expensive. After some discussion about the criteria, it was revised somewhat and Mr. Hahn made corresponding revisions in the design analyses and the design drawings.

Unfortunately, before the drawings were completed, Mr. Hahn died of a heart attack. Exstar was reluctant to continue the project because Mr. Hahn was the only employee with the requisite expertise. TESI agreed to take the design analyses and drawings and to seek to complete the design and obtain other bidders for fabrication of the wash column.

Mr. Hahn's work product was collected, packaged and accepted by UPS for delivery to TESI. No copies were made prior to the shipment and it was uninsured. The shipment was lost by UPS, according to their tracking system, somewhere between Wilmington, North Carolina and Atlanta, Georgia.

Exstar and Mr. Hahn's son assisted TESI with recovery of certain data files and drawing files from Mr. Hahn's work and home computers. The handwritten notes and empirical data he relied upon were not recovered. The design analysis is included in Appendix D.

To provide some assurance that the wash column would work, TESI located and retained Mr. James A. Heist to review the design calculations. Mr. Heist confirmed that there were no apparent serious flaws in the design and that the wash column should work as intended. In addition, Mr. L. Ellefson and Mr. F. Jaeger reviewed the design of the wash column. The reviews resulted in some minor changes in the wash column configuration but the primary configuration and dimensions remained as determined by Mr. Hahn.

4.4.3 R141b Recovery Module Design.

Initially, the solubiity of R141b in water was quickly estimated by Allied Chemical at about 300 ppm at atmospheric pressure and nominal ambient temperature. Subsequent testing determined that the solubility was several times greater, about 1350 ppm under standard conditions. This higher solubiity caused TESI to redesign its recovery system to insure acceptable levels of R141b in the product effluent.

Several approaches for removal of R141b from the product water were considered. Extensive analysis of an air stripper design was completed and it was clear that R141b could be successfully removed from the product water. This design required a large condenser to remove R141b from the air stream while avoiding the release of R141b into the atmosphere.

An alternative design that made use of a vacuum distillation tower was also evaluated, and was shown to have greater long-term promise. Because the wncentration of R141b in the effluent would be much higher than in an air stripper, the condenser heat load and size would be substantially smaller with a vacuum tower. Consequently, TESI decided to implement R141b recovery with a vacuum tower subsystem.

This subsystem was deleted from the scope of work proposed by TESI to the Bureau of

Reclamation. Consequently, Relamation did not fund the R141 b recovery module design for the pilot plant. Subsequent to the contract award for this work, TESI proposed to the Center of Excellence for Research in Ocean Sciences (CEROS) to implement R141b recovery in a vacuum tower module. The design and construction were two of several Research and Development tasks funded by CEROS.

A vacuum tower design was prepared and fabricated by Austin McCormack, and installed and tested by TESI with funding provided by CEROS. That work will be reported separately under the provisions of CEROS contract number 41367. The CEROS work is planned for completion in Mid- 1998.

5. PLANT CONSTRUCTION

5.1 Site Preparations

One of the early tasks was the grading and filling of the area where the crystallizers were to be installed. Figure 5-1 shows the crystallizer area after the grading and filling had been done. About 50 cubic yards of fill material was required to achieve a more uniform slope from end-to-end of the crystallizers. The actual slope is close to the target slope of 1 percent, although some local deviations were accepted. For example, near the mid-point a high-point that deviated a few centimeters (an inch or two) was accepted. The high point consisted of contiguous volcanic rock. Removal would have required extensive demolition and that was not considered to be cost-effective. Alternatively, the entire crystallizer bed could have been filled to raise it enough to avoid this bump. Far more fill would have been necessary to raise the entire crystallizer bed sufficiently and this was also considered excessively costly. The volcanic aggregate fill is the dark material extending North from the horizontal pipe in the foreground between the fence and the large pipe on the left.

The concrete pad shown in Figure 4-4 required some work to make it suitable for installation of plant equipment. It was necessary to remove a multitude of small fastener stubs remaining from the mounting of previous equipment on the slab. The stubs protruded from 6 to 9 millimeters (1/4 to 3/8 inch) above the slab, making them capable of penetrating the polyethylene tank bottoms. In addition, two reinforced concrete pedestal supports were also removed to make the entire surface accessible for components. One of the pedestals can be seen just to the right of the large surface sea water pipe at the West edge of the pad. The removal of the pedestals and rebar left roughly 46 cm (18 inch) square holes in the pad, requiring some relatively minor repair of the concrete surface.

A small frame building was moved adjacent to the East side of the concrete pad, as shown by the photograph in Figure 5-2. This building served as the site project office, and also housed the plant control system and data acquisition system. The photograph was taken after delivery and temporary staging of the HDPE tanks on the concrete pad.

5.2 Crystallizer Construction

After the crystallizer bed had been filled and graded the shell pipe was brought from the NELH storage area, trimmed in appropriate length segments and welded to the length needed.

Figure 5-1: Crystallizer Graded Bed

Figure 5-2: Site Office Building

The 30 cm (12-inch) HDPE pipe segments for the CHX shells were towed to a road adjacent to the crystallizer bed, as shown in Figure 5-3, with a four-wheel drive forklift. The segments were cut, trimmed, and fusion welded as shown in Figure 5-4 to produce five approximately 220 foot long shells; four for the crystallizers and one to be used as the ice melter.

The shells were then lifted over the NELH pipe and placed on the graded crystallizer bed between the pipe and the fence as shown in the photograph in Figure 5-5. Van Stone style end flanges were then fusion welded at each end of the crystallizer and ice melter shells.

These end flanges were provided for mounting the transition tees at the ends of each crystallizer. One of the end flanges is visible in Figure 5-5 between the transition tees in the foreground. At the time of this photograph, three of the transition tees had been installed. A (30 cm) 12-inch PVC pipe dram for the cooling flow that was provided by NELH is visible in the right foreground.

Prior to mounting the transition tees, each of the shells was cleaned by passing a dense plastic foam “pig” through the 30 cm (12-inch) pipe. This was accomplished with the use of a cover flange fitted with a connection that permitted pressurizing behind the “pig” with sea water to force it through the pipe. The “pig,” the flanged pipe end, and the flange cover are visible in Figure 5-6. This photograph shows the “pig” being started into the pipe and the flange cover (lower right) that provided the connection to the surface seawater used to force the “pig” through the pipe.

After the shell piping had been cleaned the transition tees were installed, and the tubes that provide the heat transfer surface for cooling the R141b/seawater mixture were installed. A pulling cord leader was first installed with a radio-controlled model car that was driven through each pipe, pulling the leader through the pipe. The leader then was used to pull a stronger pull line that was looped through the piping. The tubes were bundled in two major groups, and each was pulled as shown in the Figure 5-7 photograph. Multiple 300 foot reels of 2.5 cm) l-inch tubing were mounted and fed through a converging segment of HDPE pipe to facilitate assembly of the bundles. The collected tubes were bundled together around tube segments about one foot in length that served to set the tube spacing. Nylon cable ties were used to fasten the active tubes into bundles around the tube segments. The tube bundles had a nominal cross-section like that shown in Figure 5-8. The two primary bundles provided 14 tubes. The remaining six tubes were each pulled individually, and positioned to prevent them from nesting and adversely affecting heat transfer. The tubes extended through PVC transition tees at each end of the 30 cm (12 inch) HDPE piping.

Figures 5-3: Crystallizer Shell Pipe Delivery

Figure 5-4: Thermal Welding High Density Polyethylene Shell Pipe

Figure 5-5: Crystallizer Shell Pipe Emplacement

Figure 5-6: Shell Pipe Cleaning with "Pig"

Figure 5-7: Installation of Crystallizer Heat Exchanger Tubing

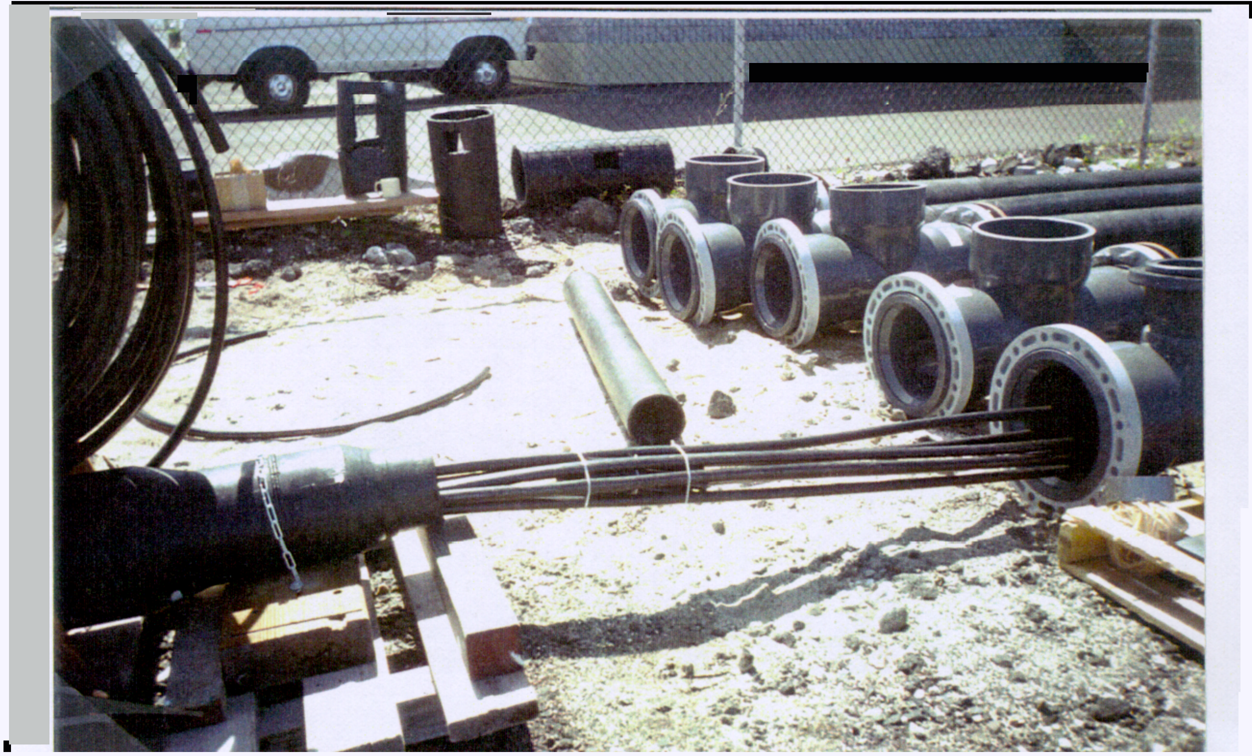
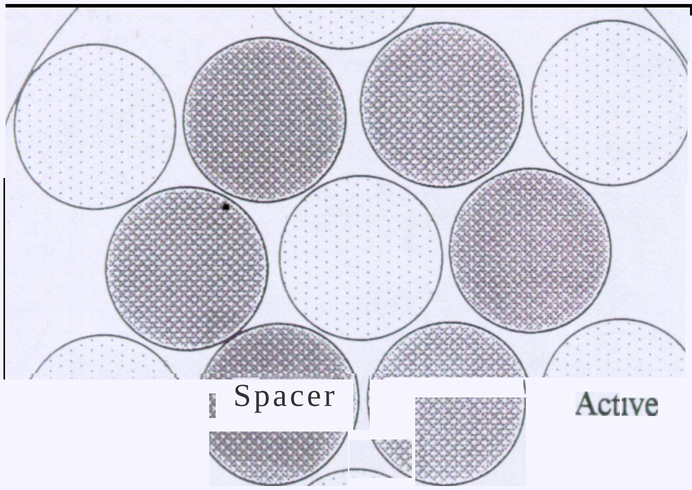


Figure 5-8: Tube Bundle Pattern



Tube sheet end boxes were mounted to the flanged ends of each crystallizer. These end boxes each consisted of a tube sheet for connection of the cooling tubes to a common manifold at each end, the inlet and outlet manifolds, and appropriate end covers. At the North end of the crystallizers the end box simply turned the product side flow to return to the South end. At the South end the product flow inlets and outlets were connected to place the crystallizer heat exchangers (CHX) in series. The crystallizer end components were manufactured by Special Plastic Systems, Inc., a company that produces machined plastic parts for the film and semiconductor processing industries.

The HDPE tubing inside the heat exchangers was extruded as a special order to provide a thinner wall than standard 2.5 centimeter (1 -inch) tubing. In this instance, a manufacturer in Hawaii produced 9150 meters (30,000 feet) of tubing with a 1 millimeter (0.040-inch) wall thickness. This provided somewhat better heat transfer than would be obtained with the standard 1.5 millimeter (0.060-inch) wall thickness. The tubing was connected to PVC hose barbs that had been screwed into the tube sheets. The tubing was clamped on the hose barb with a standard stainless steel worm gear-type clamp.

The transition tees provided the connection for shell-side cooling flow from one crystallizer to another. The crystallizer heat exchangers were designated CHX-1 through CHX-4, numbering from left to right at the near end of the crystallizers. Generally, the cooling flow went North in CHX-1, South in CHX-2, North in CHX-3 and South in CHX-4. The ice melter heat exchanger was identical in design with CHX-2 through CHX-4 and was placed to the left of CHX-1 as viewed from the South.

The tees were inverted and the stems of the tees were connected in a short vertical loop formed with 45-degree ells. At the near end CHX-2 and CHX-3 were connected as shown in the Figure 5-7 photograph This photograph also shows the connection to the NELH deep seawater cooling supply, the pipe extending horizontally to the inlet of the CHX-1 tee. The line from CHX-4 to the drain is also shown (Figure 5-7) at the right but is somewhat obscured in this view. At the far end CHX-1 and CHX-2 were connected with a similar vertical loop, as were CHX-3 and CHX-4. These connections are depicted in Figure 5-8.

The crystallizer piping was installed with restraints to prevent side to side movement but not longitudinal movement. The modulus of elasticity of the HDPE pipe is low enough that axialthermal stresses are low and the weight of water and pipe is usually sufficient to restrain axial movement.

The **crystallizer** shells, and other black **HDPE** components, were painted white to reduce the solar heat load. The reduction was not measured but was **significant**. The water tanks, while **empty, had an surface temperature** estimated (painful to touch) as approaching **50°C (120°F)** by late morning. After painting the tanks the temperature was substantially lower, with the surface of the tank approaching ambient air temperature.

5.3 Installation of Wash Column, Tanks, and other Components

The wash column fabrication was ordered from Q-DOT, Inc. a San Diego area metal forming and fabrication shop. The wash column shell **and** inner tank and support stand, were fabricated **from** low-carbon steel. The inner tank of fiberglass-reinforced plastic **(FRP)** with a polyethylene liner had been ordered and received by Exstar prior to Mr. Hahn's death and was provided by Exstar to Q-DOT.

The wash column was assembled to the stand on-site and placed on the concrete slab at the southwest corner. The completed wash column **installation** is shown in Figure 5-9 which also shows the platform mounted 1900 liter (500 gallon) tank, and several of the slab mounted tanks.

A supplier of HDPE water storage tanks was located in Honolulu with Makai Ocean Engineering's assistance. Five low pressure HDPE tanks were supplied by Rainwater, Inc. : three 1900 liter @O-gallon) capacity and two 950 liter **(250-gallon)** capacity tanks. **One** of the 1900 liter **(500-gallon)** tanks was placed on a platform constructed on-site and the remainder **of the** tanks were set on the concrete slab. The tanks were molded of HDPE with carbon black filler to provide for ultraviolet radiation absorption to prevent deterioration in the intense sunlight at the NELH location. The tanks were painted white with an exterior latex paint to reduce the solar heat load on the system

The **R141b** recovery module vacuum tower can also be seen in Figure 5-9. The tower is **approximately** 7.6 meters (25 feet) **high,** and is to the right of the **platform** mounted 1900 liter **(500-gallon)** tank.

5.4 Control System and Instrumentation

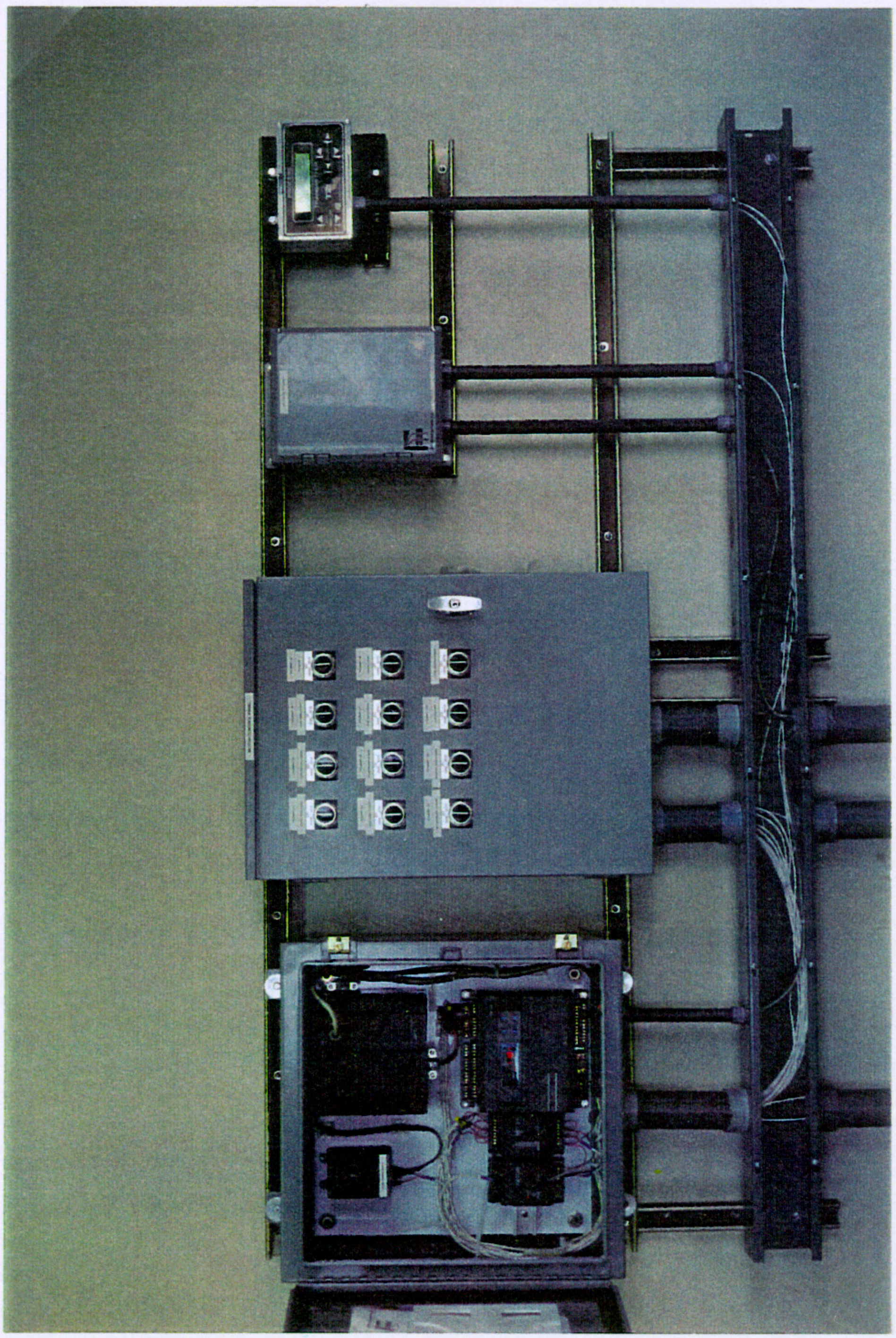
The control system and instrumentation was provided under subcontract by Electronic

Figure 5-9: Wash Column, Product Water Tank and Vacuum Tower

Control Systems, Inc. (ECS), of San Diego, California. The system was installed in the project office. The system, shown during installation in Figure 5- 10, provides for automatic flow control of the product side seawater flow, and the flow of R141b. The system also permits automatic or manual operation of the pumps and provides data logging for product side flow, R141 b flow, and temperatures at the inlet and outlet of the crystallizer, at the outlet of the startup tank, and the product water outlet of the wash column. The system also incorporates a conductivity cell for gauging the salinity of the brine/clathrate crystals slurry entering the wash column.

A Compaq Pentium computer provides a graphical interface for real-time observation of plant performance, and also provides the interface for changing control system and data logging parameters. The system may be accessed remotely by modem, either to change settings, or to acquire data. The control system operates independently of the computer that provides data logging. Plant operation is thus independent of the operation of the interface/control computer.

Figure 5-10: Control System Installation



6. PLANT OPERATION

The plant was tested in April and operated in June, July, August, September, and October of 1997, and January and February of 1998. Significant quantities of clathrate crystals were produced during each of the operations of the plant except in July. The wash column did not function as intended during any of the periods of plant operation.

6.1 June 1997

During June the pilot plant was operated for the first time and some difficulties were encountered. One difficulty was that a compatibility problem in the R141b flowmeter became evident when R141b was introduced into the system. An internal plastic component of the flowmeter was deformed and the meter immediately malfunctioned. Replacement of this meter took several months, and in the interim R141b additions to the product side were estimated by level changes in the R141b storage tank, at about 11.2 liters per centimeter (7.4 gallons per inch) of level change.

The clathrate crystals that formed occurred without adding seed crystals, and was unexpected. Insufficient in quantity to form the "ice pack" in the wash column, the crystals simply plugged the drain holes in the wash column. This in turn led to an overflow of the brine into the product water tank The plant was operating in a recirculation mode so the overflow resulted in a net transfer of seawater from the clathrate crystals production loop into the product water tank. The transfer of water out of the recirculation loop eventually resulting in insufficient head to continue operation of the pumps.

Initially, it was not clear that clathrate crystals had been produced. The plant was shut down to diagnose the event. In that process samples taken from the crystallizer drains and from the wash column contained clathrate crystals. A sample of the clathrate crystals was obtained from the crystallizer drains, and from the wash column sample port. Over a liter of crystals was obtained, mixed with a substantial quantity of excess R141b.

An attempt was made to wash the clathrate crystals to determine whether the clathrate crystals accomplished any desalination. The wash process was crude, simply pouring cold water over the clathrate crystals that had been placed in a sieve. The clathrate crystals were then placed into a jar and allowed to melt. The resulting product contained a black residue believed to be from the failed flowmeter part and about 20% R141b by volume. The large amount of R141b is an indicator that the washing process was not effective. The water amount of **R141b** is an indicator that the washing process was not effective. The water floating on the **R141b** (see Figure 6-l) was drawn off and analyzed for salinity. The result was a substantial reduction in the salt content, from 34,000 ppm to about 4,000 ppm, but a higher level than expected in the product water.

While the plant was shut down, R141b settled out of the seawater/R141b mixture in the pump housings. In most instances, this resulted in no damage to the pumps. Two pumps, however, had FRP impellers that were not compatible with **R141b** which damaged the impellers. The impellers partially delaminated and swelled sufficiently to seize in the impeller housing and bearing to prevent operation. One of the pumps was in the startup loop (P-9) and the other was the primary product side pump. It became necessary to replace the impellers in both pumps before plant operations could continue. The damage seemed to be minimal when the pumps were operated continuously and the **R141b** concentration was at the design level of about 4 percent. For that reason, a decision was made to obtain replacement impellers and take procedural steps to avoid accumulating a high concentration of **R141b** in the pumps. At the same time, the manufacturer was requested to provide impellers made of a material compatible with **R141b**.

6.2 July I997

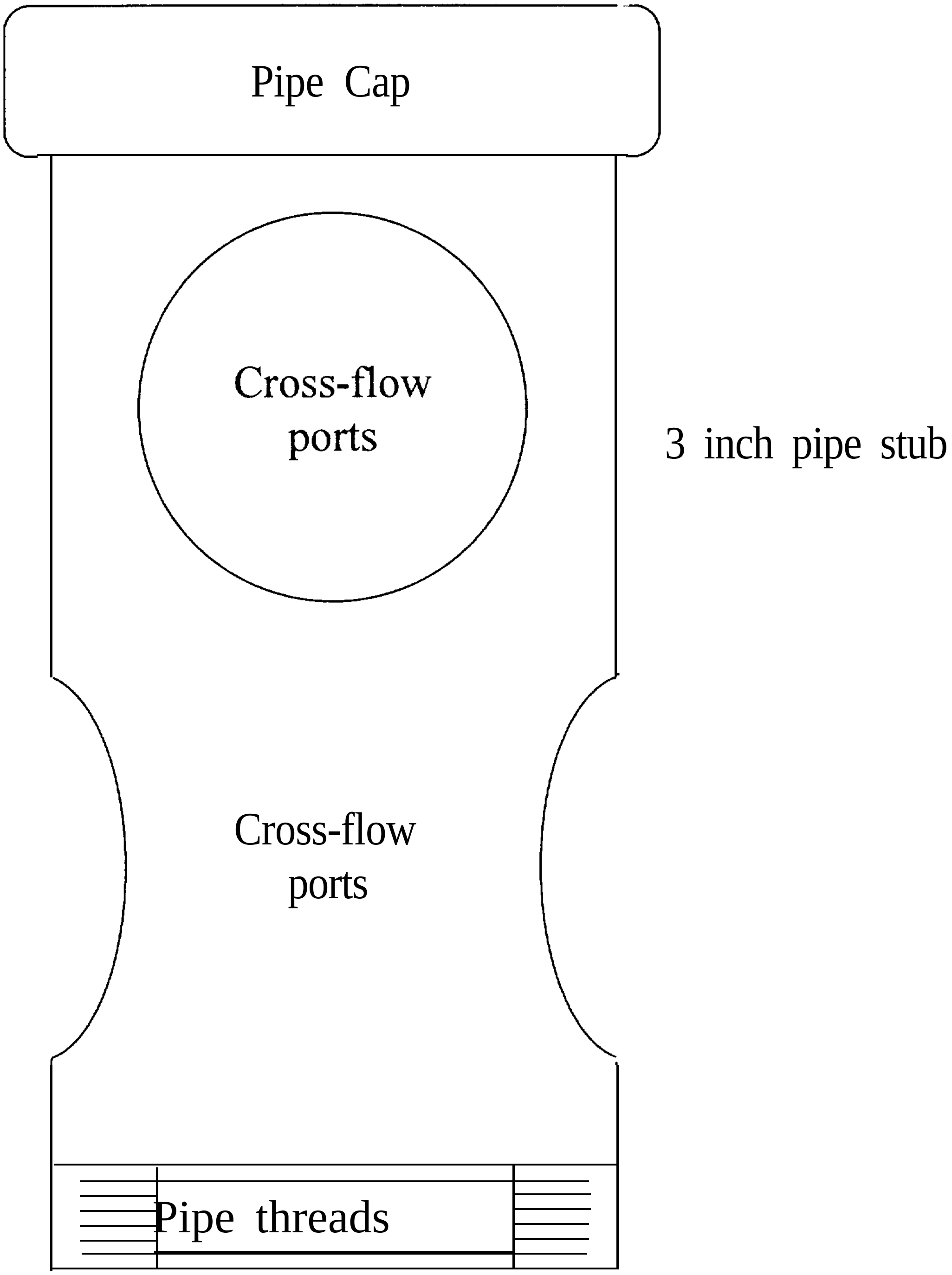
During July operation of the system was attempted during record high temperatures along the Kona coast. The environmental heat loads were such that it was difficult to reduce the temperatures in the crystallizers to levels that would produce clathrate crystals.

New impellers for the pumps were received and installed. The wash column inlet was modified as shown in Figure 6-2 in an attempt to accomplish a more uniform vertical velocity at the bottom of the "ice pack." Evaluation of the original configuration showed that the inlet velocity was about 0.76 meters (2.5 feet) per second, but the average vertical velocity in the inner tank of the wash column was only 0.006 meters (0.02 feet) per second. The inlet velocity was thus over 100 times the average velocity; in effect, a high velocity inlet flow "jet" that could tunnel through the center of the forming "ice pack."

The inlet was modified to divide the flow into several smaller horizontal streams that would dissipate the kinetic energy in horizontal flow while stirring the bottom of the wash column inner tank as it was turned upward. It was thought that this design would produce a more

Figure 6-1: Initial Sample of Product Water and R141b

Figure 6-2: Modified Wash Column Inlet



uniform vertical velocity, and lessen the tendency of a central "jet" to break through the forming ice pack.

Multiple attempts to produce clathrate crystals without seed crystals were unsuccessful despite the spontaneous initiation of crystallization and growth observed in the June operation. This prevented evaluation of the changes in the wash column inlet. The horizontal streams created by the change in the inlet could be observed after R141b had been introduced.

The lack of crystal initiation was discussed with Dr. Ripmeester and his advice was that initiation of crystallization is a highly uncertain random event, but that it was made more likely with significant subcooling. The record high ambient temperatures worked against producing much subcooling during the July operating cycle.

Tests done by Makai Ocean Engineering had indicated a freezing point in seawater of about 7.2°C (45°F). This was lower than the value used in the design of the CHXs, and the combination of extremely hot weather and the lower freezing temperature combined to make clathrate crystals production difficult.

Again, no seed crystals were used during this operating cycle. The lack of crystals production was not expected, and indicated the importance of seeding the process. This was especially true given the small difference between the freezing temperature and the cold water sink temperature. The random process of crystallization could result in very long times between reaching suitable temperature conditions and initiation of crystallization.

During the attempts to operate the plant, the effect of salinity on freeze temperature was evaluated with bench tests. Three salinity levels were examined, a sample of deep-sea water having a salinity of about 34,400 ppm, a 50 percent seawater-SO percent potable water mixture with a salinity of about 17,200 ppm, and potable water with negligible salinity. The results of this work are given in Figure 6-3.

6.3 August/September 1997

During these tests ambient temperatures were again near record highs for the Kona coast. In this instance, the startup system was used to provide seed crystals for the process. The startup system was operated until detectable quantities of clathrate crystals were produced. Crystals were observed by retrieving small samples with a dip net that had a nominal mesh

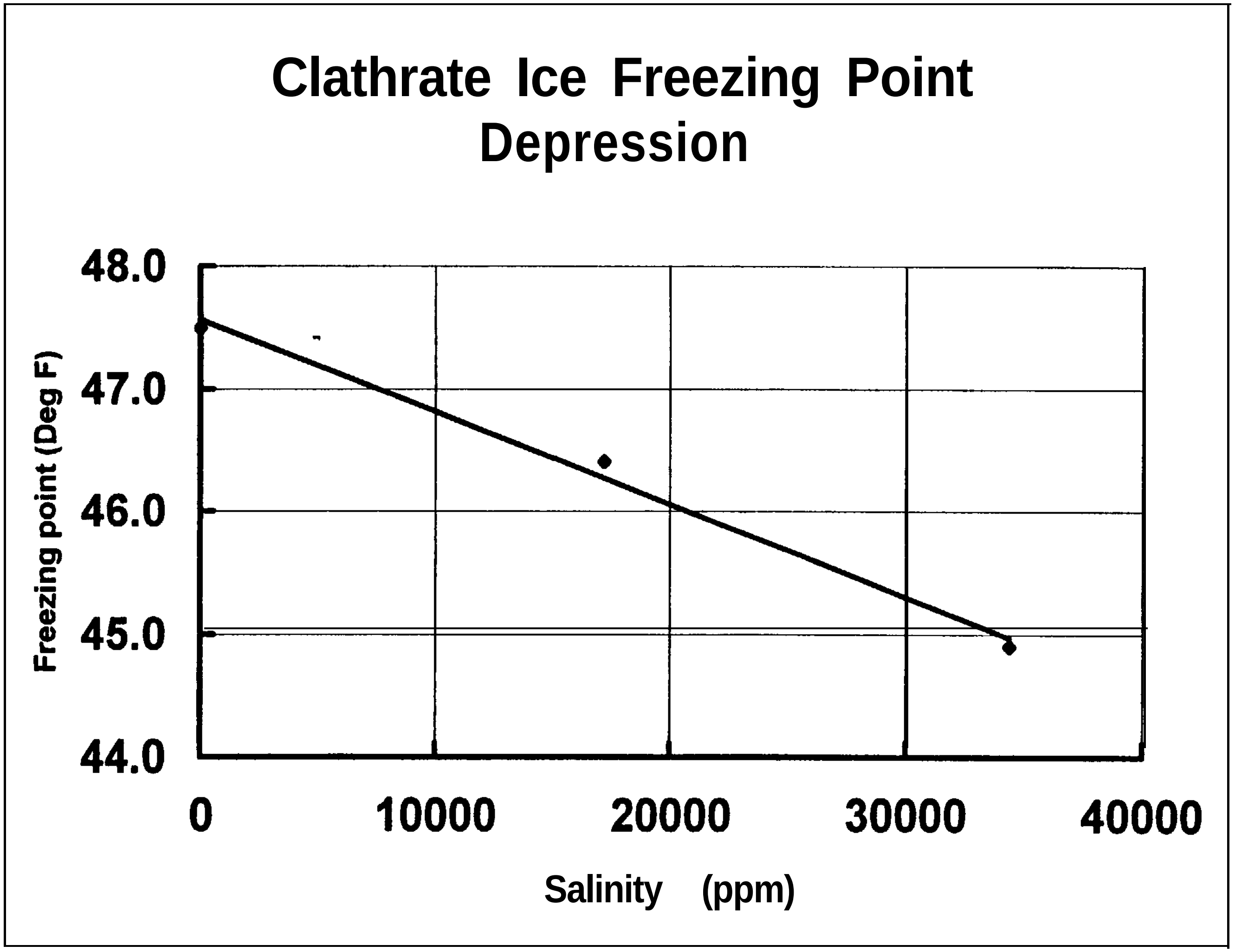


Figure 6-3: Clathrate Freezing Point Depression

size of 200 microns. The seed crystal size was not measured but they were small enough that many passed through the net. The quantity of those captured was small (estimated as about a milliliter) and had the consistency of a fine grit. The sample probably consisted of particles smaller than the mesh size that were bound together by surface tension effects. The quantity captured was quite small relative to the amounts later obtained in samples taken from the system.

The system was cooled down in parallel with the startup system operation. Seed crystals were introduced from the startup system after the return temperature from the crystallizers dropped below 6.1 °C (43 “F).

The production of clathrate crystals began with the system in a recirculation mode, with a small makeup seawater flow. Clathrate crystals were produced but the modifications to the wash column inlet did not produce the desired result. The wash column again failed to perform as intended. The drain holes plugged and a coherent ice pack did not form, again resulting in an overflow of seawater from the wash column into the product water storage tank.

In this instance, the overflow was detected in time to make adjustments so that clathrate crystals production could continue while an attempt was made to flush the seawater from the product water storage tank. During the cleanup, a catastrophic failure of the CHX-1 return end cap occurred, requiring shutdown of the system. The end cap failure, shown in Figures 6-4 and 6-5, resulted in the loss of much of the inventory of the recirculating clathrate crystals and R14 1 b/seawater mixture.

Shortly after the end cap failure, samples of clathrate crystals were taken from each of the crystallizer drains, and from the wash column drain with a mesh bag. Part of the clathrate sample is shown in the photograph given as Figure 6-6. This sample was still in the collection bag when the photograph was taken. A total of about four liters of crystals were obtained and stored in polyethylene containers. More crystals were in the system but no attempt was made to gather a larger sample. The containers holding the crystals were initially placed on ice in picnic coolers, and then in the freezer compartment of a refrigerator.

The end cap was repaired as quickly as possible with fiberglass reinforced plastic (FRP) resin, and an attempt to continue operation was made. During the repair and curing of the FRP, circulation through the balance of the plant piping and storage tanks was not possible.

Figure 6-4: Crystallizer End Cap Rupture

Figure 6-5: Detail of Crystallizer End Cap Rupture

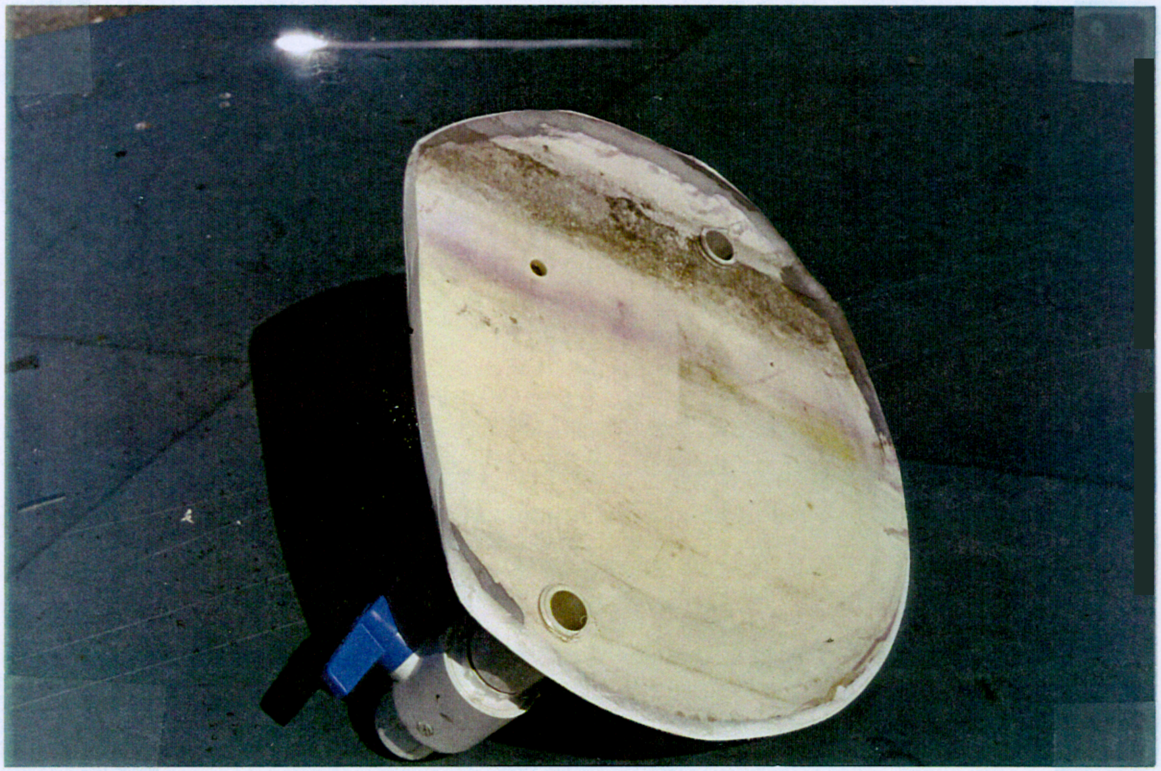


Figure 6-6: Clathrate Ice Sample

Consequently, these elements of the system had nearly equilibrated with ambient temperatures. The clathrate crystals in the system outside of the CHXs melted and when flow was restored, the crystals that had remained in the crystallizers melted.

Because the wash column had not worked as intended, a decision was made to defer further attempts at operation until a method for obtaining satisfactory operation of that unit could be devised.

6.4 October 1997

In mid-October the plant was readied for another attempt to bring it to an equilibrium operating state. After discussion of the wash column ice pack break-through that occurred in September a device to initiate formation of an ice pack in the wash column was built for use in this operating cycle. The device was a removable screen that was intended to temporarily retard the flow of the ice particles, establish an ice pack, and prevent the turbulence in the wash column from tunneling through the forming ice pack. The system was cooled down in a recirculating flow mode, and R141b was pumped into the startup tank. The chiller was operated to produce a small batch of seed crystals to initiate crystal formation and growth in the CHXs. After the seed crystals were introduced into the product side flow a leak in one of the crystallizer end caps was noted. This was the same end cap that had failed earlier and had been repaired with fiberglass reinforced epoxy. The leak was large enough that significant amounts of clathrate crystals were accumulating on the ground near the leak.

A decision was made to attempt a second temporary repair in order to avoid having to shut down the plant. However, during the attempt to make the temporary repair, the end cap completely fractured, rendering the plant inoperable. The product side flow was shut off to avoid losing any additional R141b, and to permit installing a replacement end cap. The original end cap was replaced with a Schedule 80 end cap but in the interim, with no product side flow, the balance of plant warmed up enough to prevent immediately resuming operation.

Again it was not possible to get 3,800 liters (1,000 gallons) per minute through the shell side of the CHXs. Because the heat transfer possible set the amount of clathrate crystals that could be formed, the lower flow was a matter of concern. An investigation of the shell side piping was done to determine if air in the vertical loops might be causing a set of independent vertical legs and an accumulation of head in each leg. Each vertical loop was vented again to assure that no air was trapped in them. No discernible change in **flowrate** occurred. A flow of about 3,100 liters (800 gallons) per minute was obtained when the vertical loops were vented. Little or no air was vented when this was done.

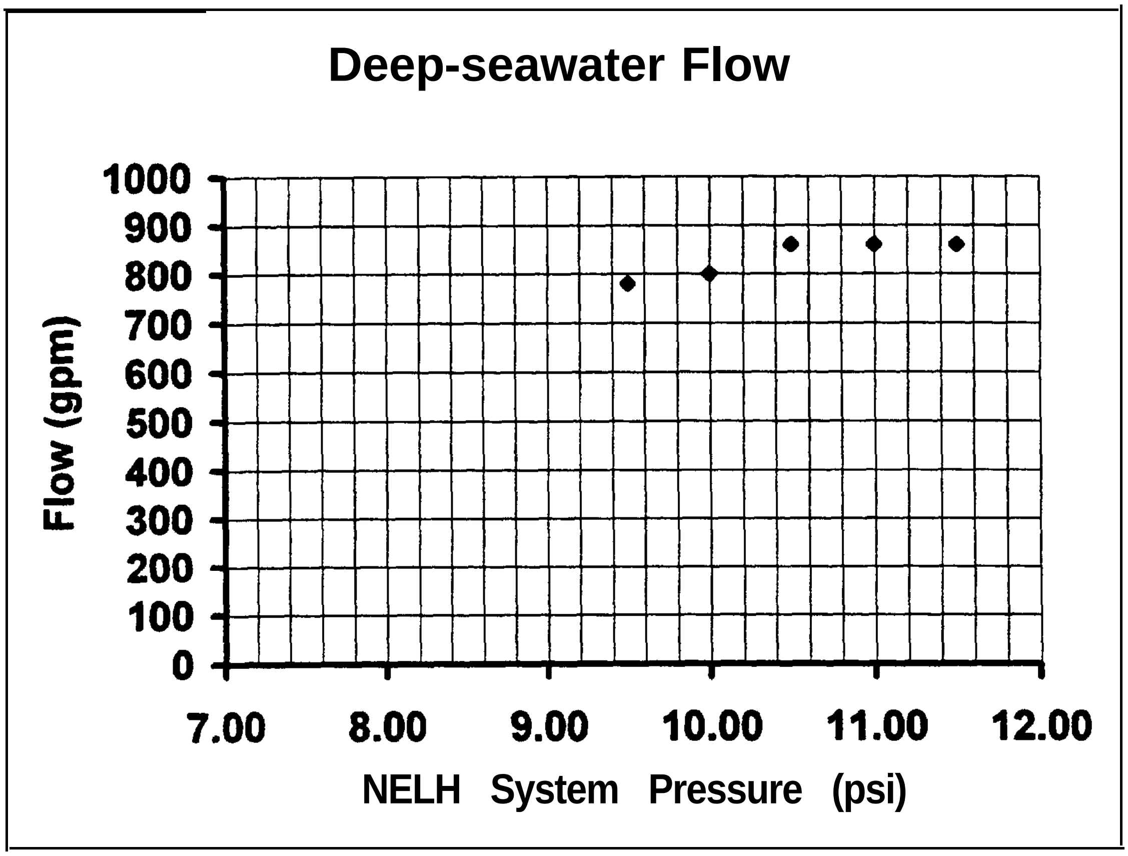
The exit vertical drain exhii a strange characteristic, venting water and then aspirating air in a cycle that lasted several minutes. Clearly this section of pipe was not running **full**. Because of the vertical leg **from** the crystallizer exit to the drain it was thought that as the drain was not solidly **full** of seawater, an adverse pressure drop might have developed. The air in this leg was removed with the vacuum pump in the **R141b** recovery module. A small but measurable improvement **from** about 3332 liters (860 gallons) per minute to 3,388 liters (900 gallons) per minute resulted, but the hoped for increase to 3,785 liters (1,000 gallons) per minute was not accomplished.

Two tests were then completed to try to determine why the cooling flow remained below 3,875 liters **(1,000** gallons) per minute. In the first of these tests, the pressure of the **NELH** deep seawater supply was **increased** in 3.5 **kPa** (0.5 psi) increments, and the effect on **flowrate** was measured. The data obtained showed an increase in **flowrate** until 72.4 **kPa** (10.5 psi) was reached., and then no **further** increase as the pressure was increased to 79.2 **kPa** (11.5 psi). These data are shown in Figure 6-7.

A second test was run with the cooling **flowrate** through the **CHXs** varied as inlet and outlet pressure measurements were made. The data from this test are given in Figure 6-8 and indicates that the **crystallizers** in **series** behave predictably as hydraulic devices. The **flowrate** should increase in proportion to the square root of the pressure drop. The data of Figure 6-8 were **analyzed** using linear regression, with an additional data point added-zero flow at zero pressure drop-to estimate the **pressure** drop required to produce a flow of 3785 liters (1,000 gallons) per minute. Those results indicate a pressure drop of about 110.3 **kPa (16-psi)** would be required.

The pressure at the outlet is **somewhat** less than atmospheric after the air is evacuated, about -23.4 **kPa** (-3.4 psig) at 3 100 liters (800 gallons) per minute. If the NELH system pressure were increased to 82.7 **kPa** (12 psig), the outlet pressure might increase to -17.2 **kPa** (-2.5 psig). If so, the pressure drop would be about 100 **kPa** (14.5 psi) and the expected **flowrate** would be about 3,640 liters (940 gallons) per minute. The apparent **flowrate** limiting of Figure 6-7 seems to be an **artifact** of measurement error: pressure, flow, or a combination of errors. It appears that the **crystallizer** pressure drop limits the flow with the available NELH

Figure 6-7: Crystallizer Flow/NELH System Pressure Data



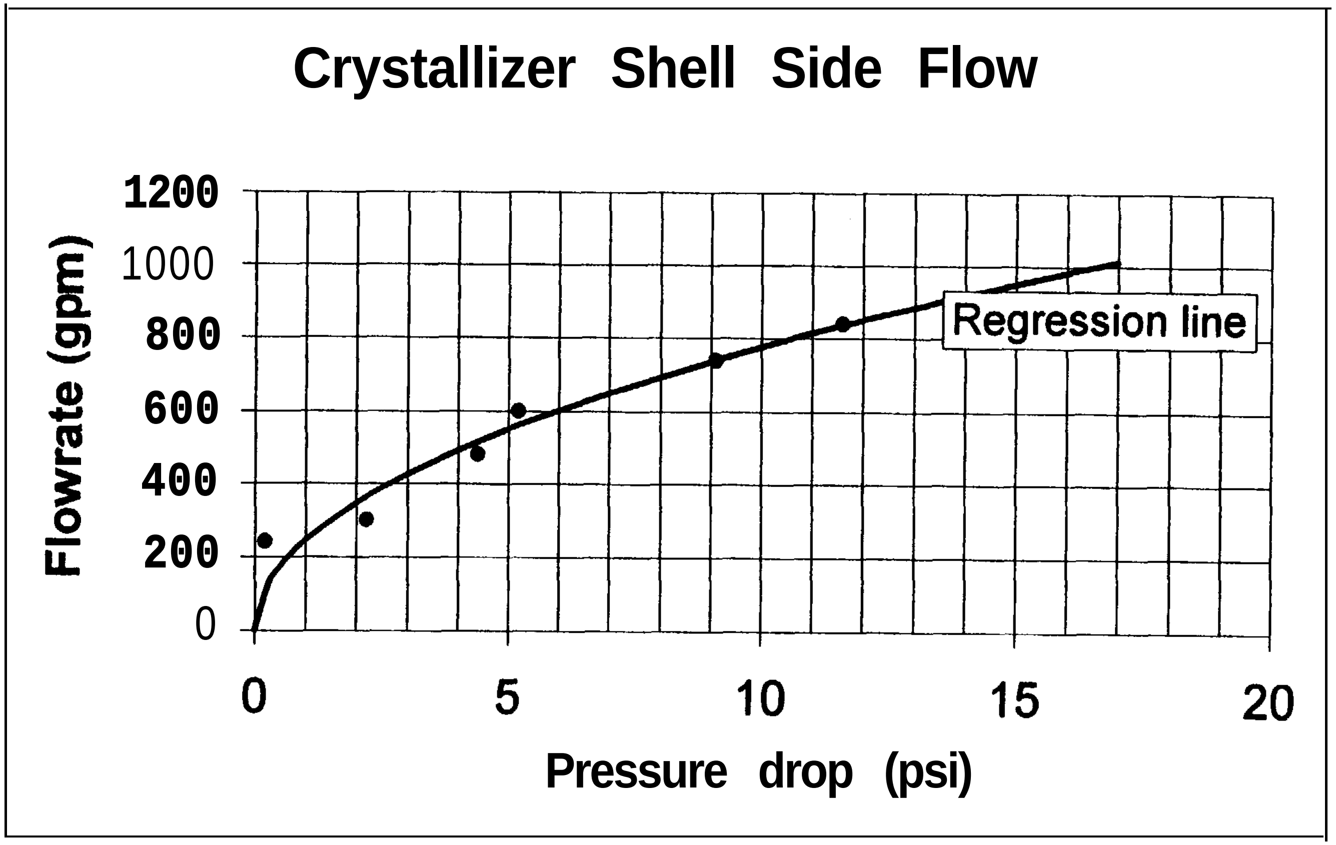


Figure 6-8: Crystallizer Shell Flow and Pressure Drop

pressures to about 3,640 liters (940 gallons) per minute. A potential solution for this is to place a pair of the crystallizers in parallel, with each taking 1940 liters (500 gallons) per minute. This would increase the flow as needed and allow the use of the same NELH supply and drain lines.

Although clathrate crystals were made in this operating cycle, no attempt to recover them was made. The system had a product side leak of about 4.7 liters (1.2 gallons) per minute, so a decision was made to find and stop the leak(s) rather than to continue to try to operate the plant.

Several leaks were found subsequent to the operation, including a leak in a buried product side pipe. These were subsequently repaired and the leakage was made negligible.

6.5 January/February 1998

In late January and early February 1998 the plant was readied for a set of tests intended to evaluate the function of the wash column with higher clathrate slurry loadings. Prior testing had been done with relatively low concentrations of clathrate slurry. It was suspected that the low concentrations of clathrate slurry and consequent relatively small amounts of clathrate obtained in previous tests prevented formation of the semi-solid matrix of crystals required for successful operation of the wash column.

To increase the amount of clathrate produced, the tests were conducted with potable water in the product side loop. This increased the driving temperature difference in the crystallizers to almost double the difference with seawater. The greater temperature difference would result in higher production rates of clathrate crystals. The abiity of the plant to produce clathrate crystals from seawater in quantity, and to reduce the salinity levels to 500 ppm had already been demonstrated but as yet the wash column had not functioned as designed. In one respect, the test of the wash column was more severe than under design conditions because the difference in density of the clathrate crystal and fresh water was greater than for seawater.

The ambient temperature and solar heat loads on the system were significantly lower than in the prior test series. Ambient temperatures were lower than the norm for January, dropping below 18° C (65 °F) overnight, and remaining below 29°C (85 °F) during the day.

Startup System

Several tests were completed prior to tests of the wash column to obtain information about elements of the system. The plate/frame heat exchanger in the startup system had been replaced with a shell and tube version. The plate/frame heat exchanger had plugged with clathrate crystals frequently during startup and the shell and tube version was less susceptible to plugging.

A test of the shell and tube heat exchanger indicated it was indeed less susceptible to plugging as the clathrate crystals formed. Flow was maintained but at somewhat lower levels, with some increased pressure drop in the startup loop. The thermal response of the startup system shown in Figure 6-9 indicates that instead of plugging the tubes, the clathrate crystals lined the inside of the heat exchanger tubes and acted as an insulator. This generally resulted in increasing startup system temperatures and higher pressure drop.

Referring to Figure 6-9, a mixture of R141b and water was circulated through the system and the chiller was activated to cool the system. Data recording was initiated at 9:O0. At about 9:45, after the mixture was subcooled to about 7.1°C (44.8°F), clathrate crystal formation and growth began and the outlet temperature from the heat exchanger increased rapidly to about 7.9°C (46.3°F). The temperature then settled into an oscillation from about 7.9°C (462°F) to about 8.1°C (46.5°F).

The temperature oscillations were observed to correlate with the cycling of the chiller in response to the thermostat limits on the chiller. During chiller operation the temperature decreased and, when the chiller cycled off, the temperature increased. Flow was observed throughout this time period, confirm@ that the heat exchanger was not blocked. The pressure drop across the heat exchanger did increase about 34.5 kPa (5 psi) and the flow was reduced but not stopped.

At about 10:46, R141b was added to the startup system. The temperature immediately increased and began oscillating between about 8.5°C (47.3°F) and 8.7°C (477°F).

The evident cause of the observed increase in average exit temperature was crystal formation on the inside of the tube walls. This would also constrict flow, and cause the higher observed pressure drop. The temperature in the chiller side of the heat exchanger oscillated between limits controlled by the chiller thermostat. Crystals forming on the inside of the tube added

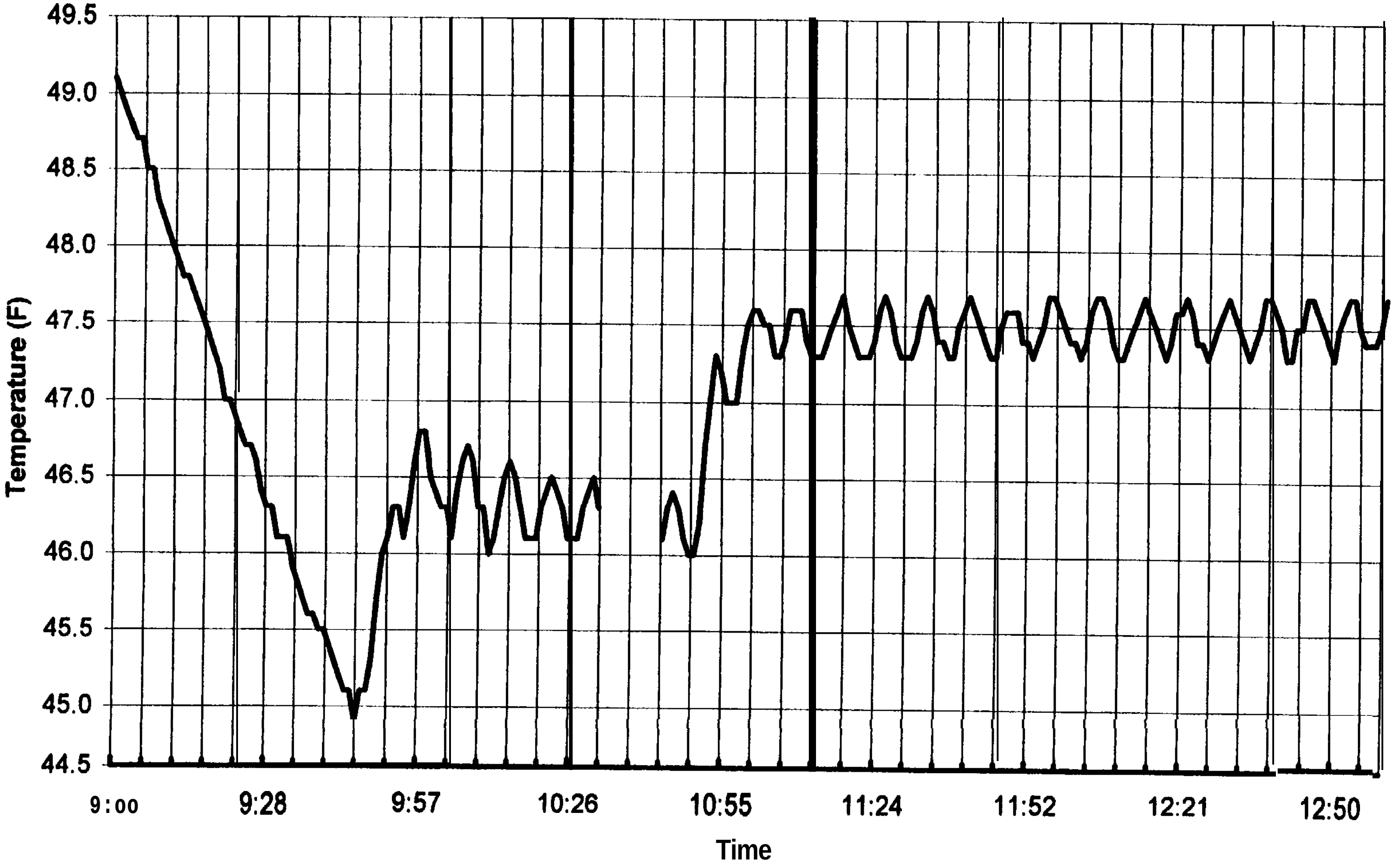


Figure 6-9: Startup System Response

resistance to heat flow, and increased the exit temperature from the heat exchanger to near the clathrate formation temperature.

Wash Column

During prior test series, and also in the first tests of this series, significant quantities of R141b were observed at the drains of the end boxes at the far end of the crystallizers. The settling of R141b at these locations was thought to be an indicator that the flow velocities in the tubes of the crystallizers should be higher to keep the R141b entrained. To increase the flow velocity the primary pump was replaced with a higher capacity pump, a second pump was placed in series, and the crystallizers were re-plumbed in a series-parallel configuration. These changes resulted in nearly tripling the flowrate over the design level when one pair of crystallizers was isolated. When both pairs were operating the per-unit flowrate was increased about 50%. The amount of R141b which collected in the crystallizer end boxes was reduced but not eliminated. The results confirm prior TESI experience that indicated upward vertical flow.is preferable to horizontal flow for keeping R141b in suspension.

During testing on February 3, 1998 the plant was cooled down with a shell-side flow seawater at about 3000 liters (800 gallons) per minute, and an indicated product side flow of about 190 liters (50 gallons) per minute. This flowrate was lower than anticipated from pre-operational tests. The flowmeter was misaligned somewhat, and when the proper alignment was restored the flowmeter indicated about 265 liters (70 gallons) per minute.

No R141b was added during this operation Free R141b was detected in the crystallizer ends and in several of the system tanks so the decision was made to operate with the R141b already in the system. After several hours of operation, the product side flow decreased substantially. A check of the drains on the CHX's revealed no flow from either the vent or drain at the far end of CHX-1, indicating that the tube side of the heat exchanger had frozen solid. A catch test indicated that the flowrate had decreased to about 58 liters (15 gallons) per minute.

Cooling flow was stopped for 2.5 hours to permit the clathrate in the CHX-1 tubes to melt. Samples were obtained from both the vent and drain at the far end of CHX-1, indicating that the tubes were again open. Cooling flow was restored at about 100 gallons per minute to prevent the unit from warming to ambient temperature overnight.

On February 4, 1998, cooling fiow was stopped for work on the plant. Cooling was restored at less than 388 liters (100 gallons) per minute at 12:00; the actual flow was insufficient to register on the flowmeter but was audible. The temperatures and flowrates are shown in Figure 6-10. At 13:30 the flow was increased to 388 liters (100 gallons) per minute, and was increased in stages to 1940 liters (500 gallons) per minute by about 15:30. Shortly thereafter, the product side indicated flow dropped to 163 liters (42 gallons) per minute. At the same time the product water exit temperature increased slightly. The flowrate recovered to about 182 liters (47 gallons) per minute and the temperature decreased. At about 16:25 the flowrate decreased to zero indicated, and the product water temperature increased by over 0.6°C (1°F). At about the same time, warmer water was introduced into the crystallizer inlet from the saline decanter. This led to an increase in product water exit temperature and melting of clathrate crystals in the heat exchanger. Product side flow recovered to nearly 208 liters (55 gallons) per minute. About 10.6 liters (2.8 gal.) of R141b were added between about 17:10 and 17:20, when the product side flow dropped to zero again.

The flow indications and temperature response indicated that the crystallizer heat exchangers had been severely occluded or blocked with clathrate crystals so operation was terminated at about 19:00. As previously, the cooling flow was restored to a minimal level to prevent the CHXs from warming to ambient temperature overnight.

On February 5, 1998, substantial quantities of clathrate crystals were again made, and again there was evidence of occlusion of the CHXs. The temperature and flow data from this operation are given in Figure 6-11. Manually recorded data captured product water flowrate and CHX exit temperature between 15:30 and 18:25. The data logger captured a larger set of data from about 18:25 until midnight.

The intial cooling of the system occurred with a cooling flow of approximately 580 liters (150 gallons) per minute. During the cooldown, which began at about 15:00, the product side flowrate was somewhat erratic at about 230 liters (60 gallons) per minute. The product exit temperature from the CHXs decreased steadily until 16:21, at which point the temperature began to increase. The temperature increased for about 16 minutes and then began to decrease again, indicating the release of the heat of fusion as clathrate crystals were formed. The flowrate through the system decreased as the temperature increase occurred, indicating an accumulation of clathrate crystals in the crystallzers. The temperature decrease signaled the depletion of available R141b for forming clathrate.

R141b was again added to the system at about 17:11. Again, there was a small temperature

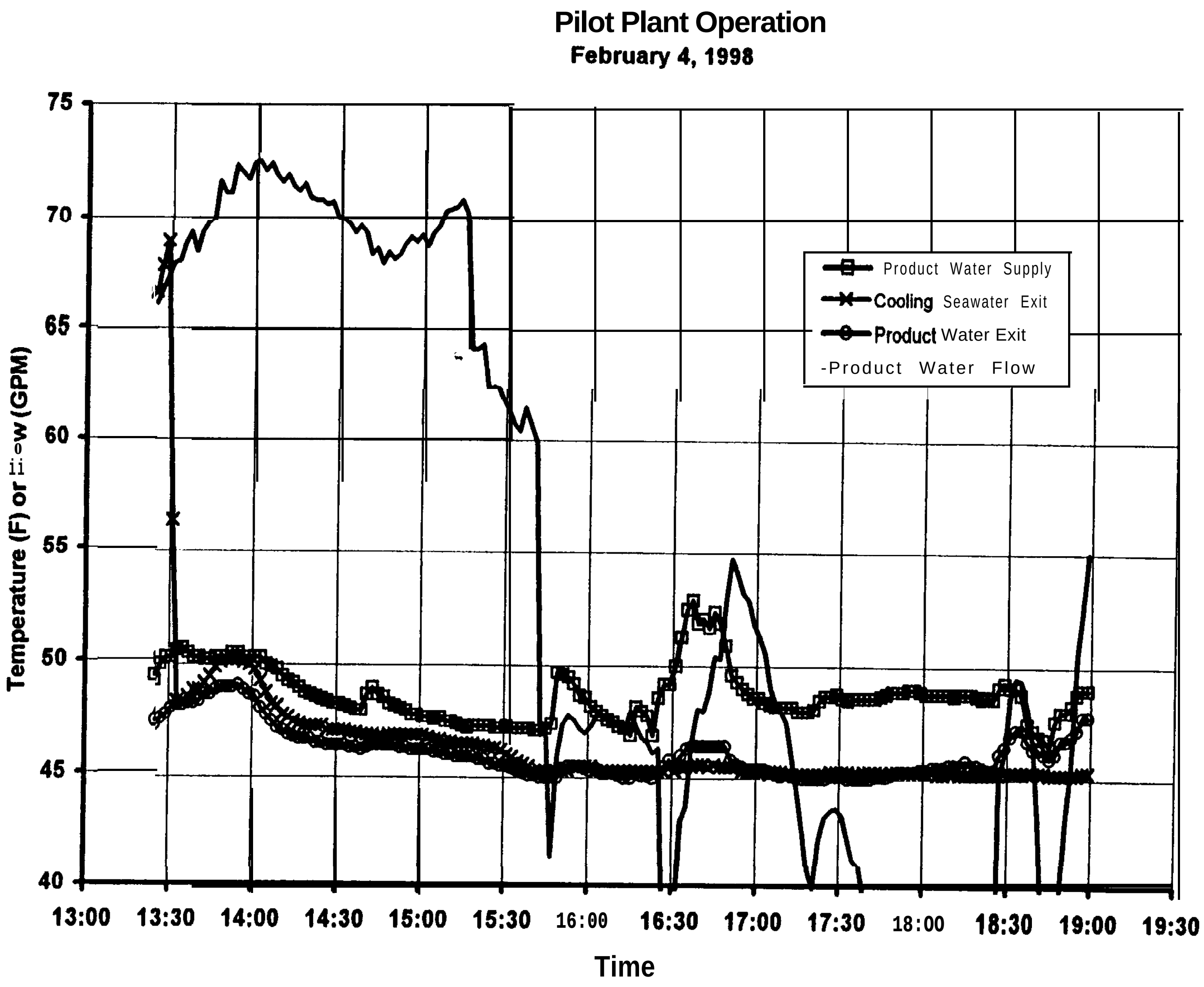


Figure 6-10: Pilot Plant Operation on Feb. 4, 1998

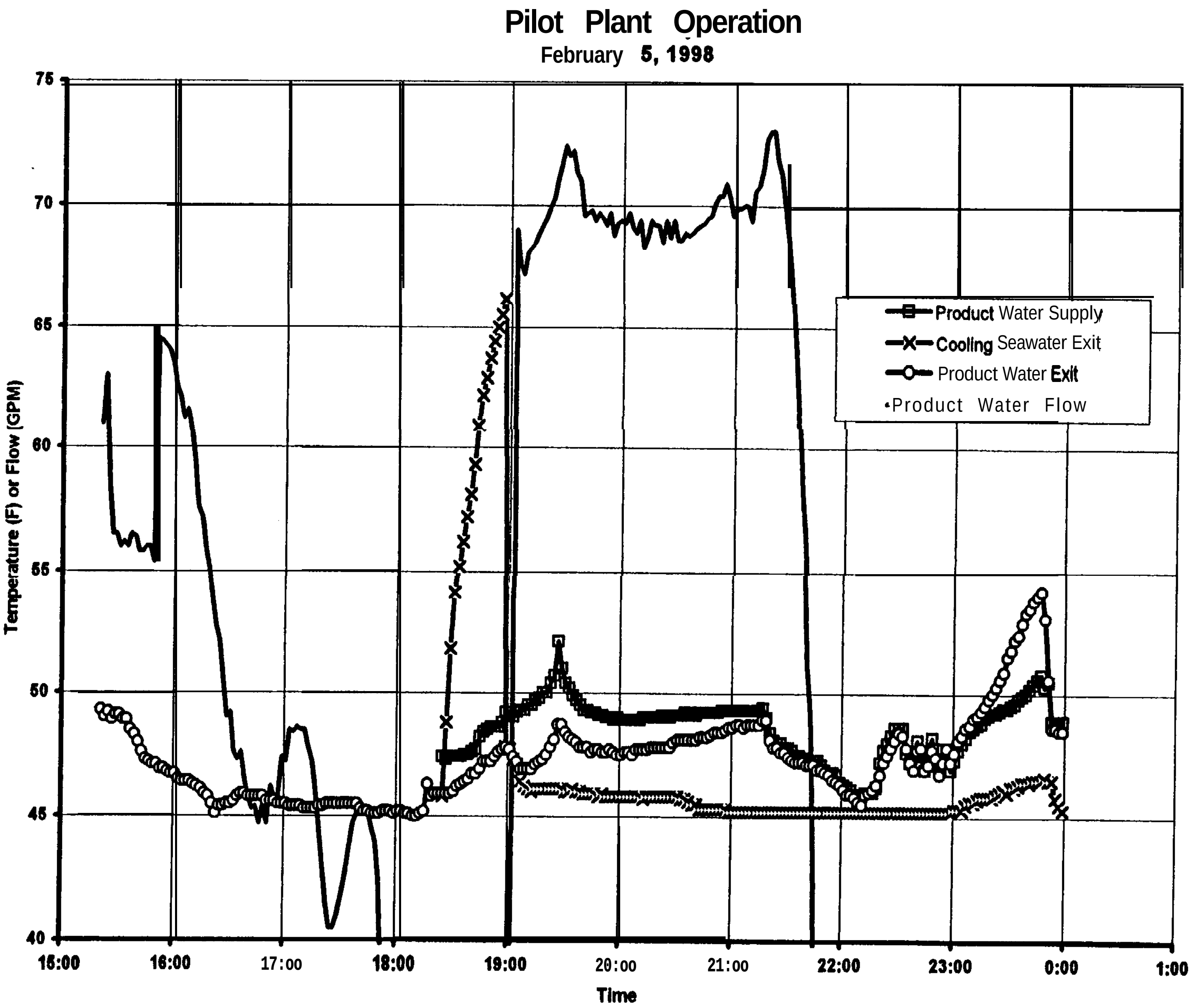


Figure 6- Pilot Plant Operation on Feb. 5 998

increase, followed by a temperature decrease. The indicated flowrate again decreased, dropping to zero at about 18:00. Because of the apparent freezing that had occurred the night before, severe occlusion of the CHXs was suspected. Consequently, it was decided to shut off the cooling water at about 18:25. With the cooling seawater flow shut off the indicated exit temperature in the stagnant section quickly increased to about 18.3°C (65°F). The system valve lineup was changed to permit placing the startup pump in the product loop.

It was unclear whether the flowrate had actually decreased, or whether the product side flowmeter was indicating inaccurately. Several manipulations of system pumps and valves were made to determine which was the case. Observations of flows led to the conclusion that the flowmeter was incorrectly indicating zero flow, at just about the same time that the indicated flowrate began to increase. During this time period the product exit temperature increased steadily.

At about 18:56 the cooling flow was restored to 1940 liters (500 gallons) per minute, and the product side temperature began to decrease. At the same time the flowmeter showed an increase in flowrate from zero to about 270 liters (70 gallons) per minute. The product side CHX exit temperature decreased until makeup water (for crystals in wash column) at ambient temperature was added at about 19: 15. The product water inlet to the CHXs and the outlet temperature increased almost simultaneously. Subsequently, the CHX exit temperature increased until the startup chiller was placed in operation. Again the CHX exit temperature followed the inlet temperature almost instantaneously, as shown in the period 22:30 to 23:00.

It appeared that the crystallizers had been short circuited somehow, perhaps by an internal structural failure in the crystallizer ends. Subsequent inspection did reveal damage to the CHX-4 inlet end box baffle, and extensive occlusion of the tubes by clathrate crystals.

The clathrate crystals accumulated in the wash column were left in the wash column overnight. No cooling was provided.

On February 6, 1998, the system was again operated. A substantial quantity of clathrate crystals remained in the wash column from the operation on the previous day.

The design of the wash column provides over 900 holes of 3 millimeter (0.125 inch) diameter for draining water out of the ice pack. In prior tests the drain capacity of these holes was much less than needed for continuous operation and formation of a clathrate crystals pack in the center tank. As the clathrate slurry was introduced into the column, the slurry partially obscured the drain holes, reducing their flow capacity. The reduction was marked, and led to filling and overflowing the inner tank of the wash column. This resulted in seawater and clatbrate slurry entering the product water tank

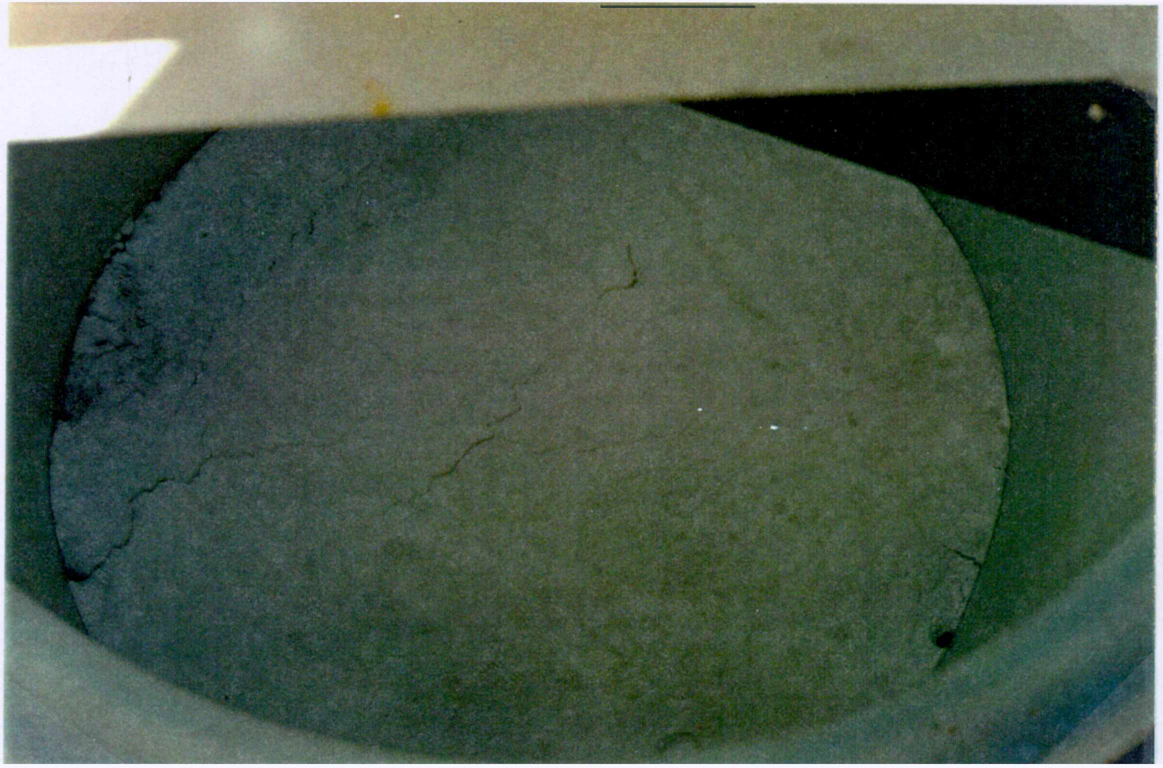
It was thought that greater quantities of clathrate slurry would facilitate formation of a coherent clathrate ice pack in the inner tank of the wash column, which would collect the clathrate crystals and prevent them from partially plugging the drain holes. To further promote the formation of the clathrate ice pack a removable screen, with approximately the same diameter as the inner tank was placed at the bottom of the tank and withdrawn as clathrate ice accumulated.

The operation with fresh water in the product side of the loop produced greater quantities of clathrate crystals slurry, and the removable screen accomplished some of its intended function. The drain holes again were occluded, though, and the inner tank began to fill with the slurry. In this instance, as the level in the wash column approached the top of the inner tank, the flow into the inner tank was reduced substantially by bypassing the wash column with much of the flow. When this was done the clathrate crystals settled and began to form a nearly coherent permeable pack.

Although the clathrate ice pack appeared on visual inspection to be coherent, when flow was restored, fissures in the pack appeared and the slurry flowed through the pack and again filled the inner tank until it was necessary to reduce the flow into the wash column. The pack did not appear to move vertically, but grew thicker as additional material was deposited on top of the ice pack. This process was repeated several times until the pack was estimated to be about 0.6 meters (2 feet) thick. The pack remained permeable and drained readily, changing in appearance from translucent to snow-like (see Fiie 6-12) as it drained. Catch tests done at 9: 15 and 9:42 established the drain rate as about 7.2 liters (1.9 gallons) per minute, substantially less than the design value of 94.6 liters (25 gallons) per minute, indicating the degree to which the drain holes were occluded. It is not known if increasing the number of drain holes would suffice to make the wash column operate as intended, but the lack of upward movement seems to indicate that it is unlikely.

After evaluation of the drain flow from the wash column the crystallizer inlet end box covers were removed to permit inspection of the baffles and tubes. As was mentioned previously, the joint on one side of the baffle in the CHX-4 end box had broken. This could have

Figure 6-12: “Ice Pack” formed in Wash Column



permitted substantial flow bypassing the heat exchanger. In addition, RTV adhesive seals on the baffles weakened and had been forced out of the CHX. This material was recovered from the product side of the system in several locations. Perhaps more importantly, all but a few of the crystallizer tubes were blocked completely with clathrate ice. It is likely that the evident short circuiting of the crystallizers had several causes, including:

- clathrate crystals blockage of all but one or two tubes in the CHXs, increasing the velocity and decreasing the residence time
- absence of the RTV seals on the center baffles
- leakage at the broken PVC baffle/end box thermal weld

The combination of these effects was substantial, and created a partial short-circuit of the crystallizer heat exchangers.

6.6 Lessons Learned

6.6.1 Compatibility of materials with R141b.

Despite considerable attention to compatibility issues, several instances of incompatibility arose that affected plant operation. The first instance was the R141b flowmeter. The flowmeter was exposed to pure R141b and incompatible internal parts began to disintegrate immediately. Although the flowmeter failure did not lead to a shutdown of operations, it made it necessary to use an alternative means of measuring R14 1 b input that did not provide continuous flow measurement data. Finding a flowmeter that was compatible with the clathrate former was difficult because of the relatively small flow and the nature of R141b.

Several small valves failed either open or shut because internal components swelled to the point that the valves could not be moved to the other position and had to be replaced. Again operations were not halted but required special procedures to accomplish some functions. Those procedures were more difficult and generally took longer to accomplish.

The FRP pump impellers in two pumps swelled and delaminated. The impellers swelled sufficiently to remove clearance in the impeller/housing bearing and prevent the pump from starting. In these instances the inability to operate the pumps did prevent operation of the plant. ,

No damage to the PVC piping was observed but repairs to pumps and other components led to an observation that the solvent-bonded PVC joints did not seem as strong as joints in piping not exposed to R141b. This was a subjective observation by an individual with considerable experience with solvent-bonded PVC piping and suggests that testing of this type of joint should be done before committing to a long-term installation.

6.6.2 Heat transfer conditions

There were several differences from the design conditions actually experienced that adversely affected the performance of the crystallizer heat exchangers. Among these were that the cold water temperature was never below 6°C (42.8°F), versus an expected cold water temperature of 5.6°C (42°F). In addition, the cooling flow during several of the tests was substantially lower than expected. In July operations, for example, the flowrate seldom exceeded 27 10 liters (700 gallons) per minute with the consequence that the cooling water exit temperature was higher than design conditions.

The actual freezing temperature of the clathrate crystals in seawater was lower than the design basis. This, combined with the higher cooling inlet and outlet temperatures reduced the heat transfer capability of the crystallizers. It is likely that the capacity of the heat exchangers is half the design heat transfer value. However, since the heat exchangers had a 34 percent safety margin the capacity of the CHX's, given these effects, was greater than 50 percent of the design value.

The pressure drop in the shell side of the crystallizers and connecting piping was higher than anticipated. This contributed to the lower flow through the shell side and could not be overcome by raising the system pressure because NELH's other customer's piping could not withstand higher pressures. This can be overwme either by placing pairs of CHX's in parallel, or by eliminating the expansion loops at each end of the CHX's and directly connecting the end boxes. This latter concept was considered impradical because of the difficulty of making the necessary precise alignments for direct connection.

To accomplish compatability with seawater, the CHXs were constructed of low conductivity plastic materials. Although this was appropriate for the shells of the heat exchangers, it resulted in poor heat transfer through the tubes and large heat exchangers. A different tube material should be considered for a wmmercial-sized plant.

6.63 Wash column

In retrospect, the wash column design seems to have some flaws. The inlet at the bottom of the inner tank is abrupt. A conical section that would permit the flow to decelerate more uniformly would seem to provide a better chance of establishing a coherent ice pack before the small drain holes plug.

The wash column has worked in other applications but another technology may be more appropriate for this clathrate crystal in seawater. The clathrate crystals are more dense than seawater and must be carried upward by the vertical velocity. The design analysis indicates that this should occur but it was not demonstrated in the pilot plant wash column.

An alternative approach suggested by Austin McCormack would be a variation on that method which makes use of gravity to accomplish separation of the clathrate crystals from the seawater while washing the surface of the crystals. This concept has been informally discussed but no specific design has been suggested, although it does seem that such equipment would be more complex than the wash column. Further, no analysis or research on this suggestion has been done. Nevertheless, a design concept that would let natural forces accomplish separation and washing has intuitive appeal.

6.6.4 Leakage

Seemingly small leak rates are unacceptable in the desalination plant. Continuous operation with small leak rates can result in large losses of product. Internal leak paths in the crystallizers might normally be unimportant, but they become conduits for bypass flows when the crystallizer tube pressure drops increase as clathrate crystals are made in the tubes. In the last stages of operation, after the crystallizer pressure drop was increased by clathrate occlusion, the data made it clear that much, if not most, of the flow was bypassing the crystallizer heat exchangers through undetected and unintended internal leak paths.

6.6.5 Ambient Heat Load

The ambient heat load on the crystallizer heat exchangers was estimated from a test with no flow on the product side. The temperature rise of the cooling flow was small, making it difficult to measure with accuracy, but the data indicate that the ambient heat load (including insolation) exceeded 1054 megajoules (100,000 BTU) per hour. This heat load is a significant, but not determinative factor, in the performance of the system. During operation, the cooling flowrate seldom exceeded 3 100 liters (800 gallons) per minute. This was about

20% lower than expected and had roughly the same magnitude of effect as the ambient heat load. In comb&ion, the ambient heat load and reduced cooling resulted in a net reduction of crystallizer performance of about 40%. For future testing, the cooling flow could be restored to the design levels 3875 liters (1000 gallons) per minute by placing the shell side of the crystallizers in a fully parallel or series-parallel arrangement. The ambient heat load could be reduced by placing pumice fill over the crystallizers to function as insulation, or by providing external piping insulation.

7. CAPITAL COST ESTIMATE

The pilot plant design was constrained by the availabiity of funds and the practical need to use the cold seawater pumped by NELH to remove the heat of fusion from the clathrate crystals formed. The pilot plant needed a small fraction of the water normally pumped by NELH for other lessees hosted by NELH. Consequently, the capital cost of the pilot plant is not an appropriate model for providing insight into the likely cost of a commercial plant. The estimated capital cost of a commercial plant was provided in the report cited previously. This-was reevaluated in light of lessons learned in the pilot plant program. The cost estimate was revised somewhat to make it consistent with the design of a commercial plant configuration that reflects the lessons learned.

7.1 Summary of Capital Costs for Commercial-sized Plants

The total capital costs for two commercial-sized plants are [redacted]ized in Table 7.1. One plant would produce 13.6 million liters (3.6 million gallons) per day or 4,000 acre-feet/year and the second would have twice that capacity. The estimates assume a location similar to NELH in that deep-ocean cold water would be available close to the shore. In each instance, the cost estimate assumes a stand-alone plant that would require installation of a large-diameter deep-water pipeline.

In Table 7.2 each of the equipment items comprising the complement of plant equipment for the 13.6 million liter per day, or 13.6 thousand cubic meters (m^3), (3.6 MG/day) plant are identified with cost of equipment, freight, and installation shown separately. The cost estimates are generally derived from the prior report with adjustments to reflect prices obtained for similar pilot plant equipment, and differing quantities based on pilot plant experience. These estimates are generally based on the complement of equipment described in the mechanical equipment list in Table 10.1 of the clathrate desalination plant study report.

The cost of the large diameter deep-sea pipeline is based on locating the wmmercial plant where an ocean depth of 610 to 915 meters (2,000 to 3,000 feet) is reasonably close to shore without a difficult undersea terrain for the pipeline. For the purposes of this estimate a pipeline length of 1220 meters (4,000 feet) was assumed. When a specific location for the

Table 7-1 Capital Cost Summary		
Capacity	**13.6 Million liters/day (3.6 MG/day)**	**27.2 Million liters/day (7.2 MG/day)**
Cost Element	Amount ($000)	Amount ($000)
Equipment cost	12,784	23,650
Deep water pipe installation	1,250	2,313
Engineering and environmental cost	480	720
Site related costs	160	240
Project development and management	300	450
Insurance	75	137
Working capital, interest during construction	1,488	2,753
Project cost before contingency	16,537	30,594
Contingency @ 5%	827	1,530
Total estimated cost	17,364	32,123

Table 7-2: Equipment Capital Cost Estimate

EQUIPMENT CAPITAL COST ESTIMATE
3.6 Million gallon per day capacity

Equipment Item	Equipment description	Equipment cost	Freight cost	Installation cost	Total cost
Salt water slurry pump	14,000 gpm @ 30 psig, 350 Hp	125	5	10	140
R141 b injection pump	740 gpm @ 100 psig, 60 Hp	8	1	2	11
Wash column	Four Units, 15 fl dia x 54 ft, c.steel w/HDPE liner	800	4	240	1044
Wash column rinse pump	250 gpm@20 psig 4 Hp	8	1	2	11
Wash column rinse water cooler	3.1 million BTU/hr	15	1	3	19
Ice melter	50,000 gal tank w/heat coil, 250 million BTU/hr	100	10	35	145
Fresh water decanter	500,000 gal, 3 hr residence	265		25	290
Fresh water tank	Two 500,000 gal tanks, 50 ft dia x 32 fl high	530		50	580
R141 b storage tank	500,000 gal, c. steel	280		25	305
Solar steam generator pump	500 gpm @ 30 psig, 15 Hp	12	2	3	16
Solar steam generator	250,000 BTU/hr, 15 psig sat. steam	500	50	250	800
Initial R141 b charge	500,000 gal	5665	142	0	5807
Equipment Total		8308	215	645	9167
Refrigerant recovery system		2500	100	1000	3600
Pipelines					
Salt water slurry	4000 ft x 24" HDPE	417	3	40	460
Waste water	4000 ft x 24" HDPE	417	3	40	460
R141b	4000 ft x 6" HDPE	12	2	3	17
Total pipelines		846	7	83	937
Total Costs		11654	316	1648	12784

commercial plant is selected, costs can be adjusted to reflect the specific site since undersea conditions have a significant effect on the cost.

The engineering design of the commercial plant is not especially complicated., and will be based mostly on sizing and layout of commercially available equipment. The environmental costs assume the preparation of an environmental impact report without interveners having strenuous objections that lead to extra costs associated with meeting environmental requirements. For example, the 34.1 million liters (9 million-gallons) per day desalination plant at Santa Barbara California was built without delay from environmental concerns, and the plants estimated here are 40 to 80 percent as large. Engineering and environmental costs are estimated at about 3 percent of the total project cost.

The site related costs are based on a seacoast location that does not require extensive development, costly buildings, or environmental remediation. Aesthetic considerations are assumed to require minimal expenditures, such as screening the site from view with berms and trees. These costs are estimated at 1 percent of the total project cost.

Project development and managementisbased on an architect-engineer-constructor managing the project with technical support from TESI. The same firm that designed the plant to minimize costs would conduct the project management. Project development and management is estimated at 3 percent of total project cost.

Insurance was estimated at approximately 0.5 percent of total project costs since no high pressure equipment is involved in the process and the working fluid is not toxic or hazardous. The working capital, interest during construction, reserves, and fees are based on commercial considerations estimated at 9 percent of the total project cost. The contingency funds were set at 4.5 percent of the total project cost before contingency.

The estimate for a larger commercial plant, designed to produce 27.3 million liters per day (7.2 million gallons/day or 8,000 acre-w) was estimated by scaling the estimate for the deep sea pipeline and process equipment costs estimated for the smaller commercial plant using a scaling factor of 1.85. It is expected that two 122-cm (48-inch) pipes would support the flow requirements for the larger commercial plant. The cost of the pipeline material is small compared to the cost of laying the pipes. Laying two pipes in the same vicinity with the same equipment would reduce the unit costs. Similarly, dual process equipment lines would be necessary to handle flow requirements and the cost of installation of two identical process lines would reduce costs. In some cases larger size equipment could serve both process lines to further reduce costs.

Engineering and environmental costs were estimated at 50 percent higher for the larger plant compared to the smaller plant. This is also true for the site-related costs, project development and management costs, and insurance costs. Working capital, interest during construction, reserves, and fees were more than doubled for the larger plant while the contingency was increased by a factor of 1.80. The total project cost for the larger plant was a factor of 1.85 higher than the smaller plant, indicating some economies of scale.

8. OPERATING COST ESTIMATE

The estimated annual operating costs for the 13.6 and 27.2 thousand m^3 (3.6 and 7.2 million gallon) per day commercial plants are summarized in Table 8-1. The annual operating costs were estimated primarily on the basis of electrical power required for system pumps.

Table 8-1 Annual Operating Cost ($000)

	Plant Capacity	
Cost Element	**13,600 m^3 ML/day (3.6 MG/day)**	**27,200 m^3/day (7.2 MG/dav)**
Electric power at $0. 10/kWhr	387	774
Makeup R141b at 1.10/lb	11	22
Carbon for liquid-phase carbon adsorbers at $1.00/lb	205	410
Management and labor		
Plant manager	98	112.5
Operations supervisor	91	101
Maintenance supervisor	78	90
Plant operators	458	528
Maintenance technicians	140	336
Total operating cost	$1,467	$2,374

The electric energy consumption for the two plant sizes was based on the electric motor list given as Table 8-2. The energy consumption assumes continuous operation of the plants, and a duty cycle on the motors that averages overall about 82 percent. The primary electrical power requirement is for pumping power but an allowance of 10 percent was added for lighting, air conditioning, control systems, and other miscellaneous small loads.

The evaluation of pumping power requirements indicated that injecting the R141b clathrate former at the 2,000-foot [610-m] depth would consume 300,000 kilowatt-hours/year and

Table 8-2: Electric Motor List for Commercial Plant

ELECTRIC MOTOR LIST FOR COMMERCIAL PLANT

Electric Motor	Flow (gpm or cfm)	Delta Pressure (psi)	Required (HP)	Required (KW)	Power Consumptio (KW-HR)	Power (HP)	Annual Consumption (KW-HR)	Connected Power (HP)	Annual cost ($000)
Commercial Plant					353028				
HCFC **R141B** injection pump	740gpm	100	54	40.3	299,738	60	299,738	60	30
Ice slurry pump	**14,000gpm**	30	306	228	**1,701,217**	350	**1,701,217**	350	170
Wash column rinse pump	250gpm	15	3	2	15,189	4	15,189	4	2
Wash column ice scraper				42	312,453	75	312,453	75	31
Wash column paddle				8	62,491	15	62,491	15	6
Solar steam generator pump	**600gpm**	30	11	8	62,491	15	62,491	15	6
Feed pump - fresh water air stripper	**2,750gpm**	25	50	37	278,473	60	278,473	60	28
Air blower - fresh water air stripper	24,000cfm	0.1%		42	312,453	75	312,453	75	31
Process air blower	**24,000cfm**			28	208,302	50	208,302	50	21
Drying air blower	**24,000cfm**			28	43,396	50	43,396	50	4
Liquid-phase adsorber feed pump	**2,750gpm**	20	40	30	222,778	40	222,778	40	22
Steam condensate transfer pump	**6gpm**	20	0.1	0.1	304	0.25	304	0.25	0
Totals				493.4	3519285	794.25	**3,519,285**	794	352
	Miscellaneous plant loads								35
	Total plant load								387

pumping the crystals slurry from the 2,000-foot depth consumes 1,700,000 kilowatt-hours/year. These two pumping systems account for 57 percent of the annual energy consumption of 3,520,000 kilowatt-hours by all motors. The allowance for house loads results in estimated total annual energy consumption of 3,872,000 kilowatt-hours. The energy was priced at $0.10 per kilowatt-hour, a relatively high value that is more representative of Hawaii than other locales.

The desalination process recovers 99.997 percent of the R141b used in the plant. This reduces make-up requirements to an annual consumption of about 3,875 liters (1,000 gallons). The addition of the carbon adsorbers to the process increases recovery from 99.87 percent with a relatively small operating cost reduction (about $30,000) that partially offsets the cost of the activated carbon. This economy was ignored in the cost evaluation.

The the liquid-phase carbon adsorbers require 20 times as much carbon as the mass of R14 1 b collected. This results in the consumption of 93,100 kilograms (205,376) pounds of carbon per year, at a cost of $205,000.

The management and labor costs represent slightly more than 60 percent of the annual operating costs. The 14-person management and labor staff consists of one plant manager, two supervisors, eight operators, and three maintenance technicians. The total work force compensation cost amounts to $865,000, or roughly 60 percent of annual operating costs.

The larger plant energy and consumables costs were estimated as double that for the smaller plant. It is expected that the same number of management and operations personnel could operate the plant but that additional maintenance personnel would be required for the larger facility. The maintenance staff was increased to six technicians. Because of the increased responsibilities of the staff, annual compensation was increased for all personnel. The 17-person management and labor work force consists of one plant manager, two supervisors, eight operators, and six maintenance technicians. The management and labor costs estimated on this basis amount to about $1,168,000, and would represent about 50 percent of the annual operating costs.

9. COST OF FRESH WATER

9. I Financing Considerations

The costs of producing fresh water from the commercial clathrate desalination plants were estimated for two sets of assumptions regarding the financing of the plants. One approach assumed that bonds issued by a municipal or district water authority would finance the plants. The bond issues were assumed to include one year of operating expenses to provide working capital. The interest on the bonds is set at 6 percent with payback over a 30-year life of the plant.

The structure of the private financing was assumed to be a mix of 18 percent equity and 82 percent debt; raised via common stocks and bonds in the capital markets. As in the public financing option, the amount raised included one year of operating costs to provide working capital. Again, the bonds were amortized over the 30-year life of the plant. The interest on the bonds was set at 9 percent. The total equity return before income taxes was set at 15.65 percent with a 42.5 percent federal and state income tax rate. This would provide a 9 percent return after taxes.

9.2 Amounts Financed

The two publicly financed desalination plants producing 13.6 or 27.2 thousand m^3 (3.6 or 7.2 million gallons) per day of fresh water would be entirely financed with bonds. The entire capital cost plus the first year of operation would be financed and would require the amounts shown in Table 9-l. The annual operating cost for the first year would be provided by equity funds. The operating cost for the 13.6 and 27.2 thousand m^3 (3.6 and 7.2 million gallons) per day plants would be 1.47 million and 2.37 million, respectively

Table 9-l Amounts Financed ($000)

	Public Financing		Private financing	
Plant Capacity	13,600 m^3/day (3.6 MG/day)	27,300 m^3 /day (7.2 MG/day)	13,600 m^3 /day (3.6 MG/day)	27,200 m^3 /day (7.2 MG/day)
Equity funds	0	0	3,126	5,782
Bond funds	17,364	32,123	14,239	26,341
Total financed	$17,364	$32,123	$17,364	$32,123

9.3 Cost of Water Produced

The annual capital and operating costs, the water production, and the unit cost of water produced is summarized in Table 9-2. The unit cost of water varies with the size of plant, and the type of financing obtained. The least expensive financing, with municipal bonds, results in the lowest cost of capital. With this option the unit cost of water for the smaller plant is about $0.53/m^3 ($2.00 per 1000 gallons). Because the larger plant results in operating cost economies of scale, the unit cost is somewhat lower, about $1.74 per 1000 gallons. These costs are at or below the target price of $0.53/m^3 ($2 per 1,000 gallons) set by a public water authority in Southern California to compete with surface water. The costs for the privately financed plants are higher but remain near the competitive target. For comparison, the San Diego County Water Authority is negotiating to purchase conserved Colorado River water in much larger quantities at prices of about ($1.10 per 1,000 gallons). Larger desalination plants would produce additional economies of scale that would make the unit cost of the water produced by the clathrate desalination plant even more attractive.

Table 9.2: Summary of Annual Costs, and Unit Cost of Water ($000)

	Public Financing		Private Financing	
	Capacity		Capacity	
	13,600 m^3 /day (3.6 MG/day)	27,200 m^3 May (7.2 MG/day)	13,600 m^3 /day (3.6 MG/day)	27,200 m^3 /day (7.2 MG/day)
Annual cost of capital				
Return on equity	0	0	489	905
Amortization of debt	1,155	2,161	1,386	2,564
Total annual cost of capital	1,155	2,161	1,875	3,469
Annual operating cost	1,467	2,374	1,467	2,374
Total annual cost	2,622	4,535	3,342	5,843
Annual water production (million cubic meters)	4.93	9.87	4.93	9.87
Annual water production (million gallons)	1,304	2,607	1,304	2,607
Unit cost of water ($/cubic meter)	0.53	0.46	0.68	0.59
Unit cost of water ($/1000 gallons)	2.00	1.74	2.57	2.24

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APPENDICES

Appendix A: Glossary Of Terms

Clathrate:	A lattice-like structure in which molecules of one substance are enclosed within the crystal structure of another substance. In this application the clathrate, formed with R14 1 b, is an ice like structure with about seventeen water molecules surrounding a single molecule of R141b or other non-water molecule.
Clathrate former:	A hydrocarbon or other non-water molecule that will form a clathrate with water crystals at elevated temperatures above the normal freezing point of water; there are a multitude of clathrate formers that will form clathrate crystals at various pressures and temperatures, in both liquid and gaseous form, both as inorganic or organic compounds, with various degrees of toxicity, flammability, and other characteristics. The clathrate former used in the pilot plant is dichloromonofluoroethane (CCl_2FCH_3), also known as Refrigerant 141b.
R141b:	A specific hydrocarbon clathrate former used as the primary agent in this research report that will form "ice" crystals in water at 8.6°C (47.5°F), and in seawater at about 7.5°C (45.5°F).
Latent Heat of Fusion:	The heat energy absorbed or released respectively, when a solid melts, or a liquid is solidified. In pure materials, the heat energy is absorbed or released at a constant temperature that is a property of the material.

Appendix B: Abbreviations/Acronyms

BTU	British Thermal Unit
C	Centigrade
cm	Centimeter
m^3	Cubic meter
F	Fahrenheit
kg	**Kilogram**
km	Kilometer
m	Meter
NELHA	Natural Energy Laboratory of Hawaii Authority
cm^2	Square centimeter
km^2	Square kilometer
m^2	Square meter

Appendix C: Units Conversion Factors

Multiply	By	To Obtain
LENGTH		
Angstroms	$10^{(-10)}$	Meters
Micron	$10^{(-6)}$	Meters
Inches	2.54	centimeters
Feet	0.3048	Meters
Miles	1.609344	Kilometers
AREA		
Acre	4,046.9	Square meters
Square Miles	2.5899	Square Kilometers
VOLUME		
Acre-feet	1,233-S	Cubic meters
Cubic feet	0.0283 168	Cubic meters
Gallons (U. S.)	3.78533	Liters
Thousand Gallons	3.78545	Cubic meter
WEIGHT		
Moles	1	Gram Molecule
Pounds	0.453592	Kilograms
FORCE/AREA		
Atmospheres	1.033513	Kilograms per square
centimeter		
Pounds per square inch	0.070307	Kilograms per square
centimeter		
Pounds per square inch	6.8947572 (10^3)	Newtons/per square meter or Pascals
MASS/VOLUME (DENSITY)		
Pounds per cubic foot	16.0185	Kilograms per cubic meter
MASS/CAPACITY		
Pounds per gallon	119.829	Grams per liter

Multiply	By	To Obtain
FLOW		
Acre-feet per year	1,233.5	Cubic meters per year
Cubic Feet per minute	0.4719	Liters per second
Gallons per minute	0.06309	Liters per second
Gallons per day	3.7854	Liters per day
Pounds per hour	0.4536	Kilograms per hour
Ounces per hour	0.02835	Kilograms per hour
Thousand gallons per year	3.7854	Kiloliters per year
Million gallons per day	3,785.4	Kiloliters per day
WORK AND ENERGY		
British Thermal Units (BTU)	0.252	Kilogram-calories
British Thermal Units (BTU)	1054	Joules
BTU per pound	2.326	Joules per gram
BTU per cubic foot	35.314725	BTU per cubic meter
Kilowatt-hours	0.001	Megawatt-hours
POWER		
Horsepower	745.500	Watts
British Thermal Units per hour	0.29307	Watts
Kilowatts	0.001	Megawatts
Kilowatt-hours per year	1	Kilowatt-hours per year
Kilowatt-hours per thousand gallons	0.26417	Kilowatt-hours per kiloliter
TEMPERATURE		
Degrees Fahrenheit	5/9 (after subtracting 32)	Degrees Celsius
REFRIGERATION		
Ton-hours	3.5168	Kilowatts
Pounds per ton-hour	0.128379	Kilograms per kilowatt
Cubic feet per ton-hour	8.051865	Cubic meters per watt
Gallons per ton-hour	1.076376	Liters per kilowatt
MONETARY		
Dollars per acre-foot	810.7×10^{-6}	Dollars per cubic meter
Dollars per acre-foot	3.07×10^{-3}	Dollars per 1000 gallons
Dollars per thousand gallons	0.264178	Dollars per kiloliter

Multiply	By	To Obtain
Cents per kilowatt-hour	10	Dollars per Megawatt-hour
Dollars per kilowatt-hour	1	Dollars per kilowatt-hour
Dollars per pound	2.204	Dollars per kilogram
Dollars per gallon	0.264178	Dollars per liter
Dollars per British Thermal Unit	3.968	Dollars per kilogram-calorie

Appendix D: ***Pilot Plant Design Analysis***

Table D-l: Properties of R141b And Water

Property	Units	R141b	Water
Molecular Formula		CCl_2FCH_3	H_2O
Molecular Weight		116.9	18
Ozone Depletion Potential		0.11	0
Freezing Point of ice	°C (°F)	11.6 (52.9)	0 (32.0)
Adjusted Freezing Point of ice	°C (°F)	8.6 (47.5)	0 (32.0)
Boiling Point	°C (°F)	32 (89.6)	100 (212.0)
Density			
	Pounds/foot^3	76.9	62.4
Density	grams/cc	1.23	1.0
	Pounds/gallon	10.3	8.3
Energy Density of Crystal	Joules/kg	335 x 10^3	318 x 10^3
	BTU/pound	137.0	144.0
Energy Density of Crystal	Joules/cc	346.7	305
	BTU/foot^3	9,305	8,216

Table D-2: Characteristics of R141b and Clathrate Crystals

Name of clathrate former	HCFC - R141 B
Manufacturer	Allied Signal
State material available	Liquid
Container size available	55 gal drum
Common uses	Foam blowing agent, solvent, vapor degreaser
Year use highly controlled	2005
Characteristics of Clathrate Crystals	
Mole ratio water to clathrate former	17.2
Clathrate density - lb cuft	65.53
Clathrate specific gravity, water = 1 .O	1.05
Clathrate formation temperature - F	47.5
Clathrate heat of fusion - btu/lb	137
Clathrate particle size	
Clathrate particle shape	
Clathrate entrainment velocity	
Clathrate depession temperature - F	46
Pysical Properties of Clathrate Former	
Molecular formula	CCl_2FCH_3
Chemical name	1, 1-Dichloro-1-floroethane
Molecular weight	116.95
Liquid specific gravity @ temp F	1.24 @ 70 F
Liquid density @ temp F - lb/gal	10.58 @ 45 F
Liquid viscosity @ temp F - Centapoise	0.5 @ 50 F
Normal boiling point @ pres psia - F	89.7 F @ 14.7 psia
Vapor pressure @ temp F - psia	9.95 psia @ 70 F
Vapor density @ temp F & pres psia - lb/cuff	4.1 times air
Solubility in water @ temp F & pres psia - ppm wt	1360 ppm @ 77 F & 14.7 psia
Water solubility @ temp F & pres psia - ppm wt	420 ppm @ 77 F & 14.7 psia
Physical appearance	Clear, colorless liquid
Odor	Faint sweet, ethereal
Flammability	
Liquid flammabiliy	Nonflammable
Vapor flammability in air mixture	
Lower limit @ temp - vol % in air	7.6% @ < 200 F
Higher limit @ temp - vol % in air	17.7% @ < 200 F
Ignition energy	Difficult to ignite, 5 times propane
Rate of combustion	Slow
Flash point	None
Environmental	
Liquid waste per RCRA	Non hazardous
Ozone depletor	Yes
Transportation - US DOT Hazard class	Not regulated

Table D-2: Characteristics of R141b and Clathrate Crystals

Safety	
Permissible exposure limit - ppm	500
Toxicity	Non-toxic, >5 gm/kg body wt
Stability	Similar to other HCFCs decomposes > 500 F
Biodegradability	Minimal
Material compatabilty	
Plastics	Polyethylene, polypropylene, polyvinylidene-fluoride, polytetrafluoro-ethylene
Metals	Carbon steel, stainless steel, brass, aluminum
Elastomers	Perfluorelastomers "o" rings polytetrafluorethylene gaskets
Reactivity	Rust, alkali metals, strong alkaline compounds

The following appendices are proprietary and have not been included in this document. If the reader wishes to obtain information contained in these appendices he/she is encouraged to contact the authors at the address shown on the title sheet.

Appendix D - Pilot Plant Design Information

D-3 Heat and Mass Balance (pages D-5 and D-6)
D-4 Equipment Sizing Calculations (pages D-7 through D-14)

Appendix E - Summary of Alternative Clathrate Formers (pages El through E12)