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SEVENTH QUARTERLY PROGRESS REPORT

1 April to 30 June 1981

on

DEVELOPMENT OF A POLYSILICON PROCESS BASED ON CHEMICAL VAPOR DEPOSITION

(PHASE I)

prepared by

F. Plahutnik, A. Arvidson and D. Sawyer

May 5, 1982

JPL Contract 955533

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HEMLOCK SEMICONDUCTOR CORPORATION

a wholly owned subsidiary of Dow Corning Corporation

12334 Geddes Rd., Hemlock, Michigan 48626

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ABSTRACT

The goal of this program is to demonstrate that a dichlorosilane-based reductive chemical vapor deposition (CVD) process is capable of producing, at low cost, high quality polycrystalline silicon. Physical form and purity of this material will be consistent with LSA material requirements for use in the manufacture of high efficiency solar cells.

Four polysilicon deposition runs were completed in an intermediate size reactor using dichlorosilane fed from 250 pound cylinders. Results from the intermediate size reactor are consistent with those obtained earlier with a small experimental reactor.

Modifications of two intermediate size reactors were completed to interface with the dichlorosilane process demonstration unit (PDU).

PDU construction was completed in May, 1981. Start-up occurred June 1, 1981, on schedule. The PDU achieved a maximum DCS production rate of 47 pounds per hour with the 3" diameter redistribution reactor. No catalyst degradation has been observed during three weeks of operation.

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1.0 Summary

This report describes a process for the low-cost production of polycrystalline silicon from dichlorosilane (DCS) via reductive chemical vapor deposition (CVD) with hydrogen. The DCS is generated from the catalyzed redistribution of trichlorosilane. The by-product silicon tetrachloride may, if desirable, be converted to trichlorosilane via hydrogenation. Objectives of Phase 1 (the current contract) are to demonstrate the feasibility of using DCS as a CVD reactor feed material and to utilize base catalyzed redistribution of trichlorosilane to produce high purity DCS. Phases 2 and 3 of the program will demonstrate the technology readiness of the process at the EPSDU level.

Four polysilicon deposition runs were completed in an intermediate size production reactor using dichlorosilane fed from 250 pound cylinders. The DCS feed/intermediate reactor system worked well with no serious operational problems. Results from the intermediate size reactor are consistent with those obtained from earlier work done with the experimental reactor.

Modifications of two intermediate size reactors for accepting DCS/H_2 feed from the Process Demonstration Unit (PDU) were completed. Reactor integration with the PDU is essential for obtaining reliable reactor data.

PDU construction was completed in May, 1981. After safety audit approval was granted, the system was purged and leak tested. Minor leaks were found and corrected.

PDU start-up occurred on June 1, 1981, on schedule. After initial instrument adjustments were made, the PDU ran smoothly for the entire month achieving a maximum DCS production rate of 47 pounds/hour. No catalyst degradation occurred during this time period.

2.0 Introduction

2.1 Program Objectives

The objective of this program is to demonstrate that a chlorosilane based chemical vapor deposition (CVD) process can produce a low cost polycrystalline silicon in high volume. Product quality both in terms of purity and form should be comparable to material produced by the existing trichlorosilane (TCS) CVD process which meets or exceeds requirements for use in the manufacture of high efficiency solar cells.

The overall program covers a 42 month period and consists of a feasibility phase, which is the subject of the current contract, an EPSDU design phase, and an EPSDU construction/demonstration phase. The schedule for the program is shown in Figure 1. Specific Phase 1 project objectives include:

1. Characterization of dichlorosilane (DCS) as a feed stock material for an experimental CVD reactor including quantitative determination of reaction products. (CVD Reactor Feasibility)

2. Design and construction of a DCS CVD reactor which will demonstrate DCS performance in a larger size reactor. (Intermediate Dichlorosilane Reactor Development)

3. Design, construction, and operation of a laboratory scale redistribution reactor and a process development unit (PDU) to characterize the TCS-to-DCS redistribution process, determine product purity, and produce sufficient DCS to permit operation of a production sized reactor. (Dichlorosilane Process/Product Evaluation)

4. Conduct preliminary design of an EPSDU based on information collected in the areas previously described and

develop supporting information for an economic evaluation of a 1000 metric ton plant. (EPSDU Design)

The general approach taken in meeting the overall program objective for the 1000 MT/Y plant will consist of: the hydrogenation of silicon tetrachloride (STC) to produce TCS; synthesis of DCS via redistribution of TCS; high temperature decomposition of DCS to produce polycrystalline silicon; and recovery of decomposition by-products. STC, a major by-product of TCS redistribution and minor by-product of DCS decomposition, is recycled into the hydrogenation process.

The basic chemical nature of the various steps in the DCS-based low-cost silicon process have been described in previous Quarterly Reports.[1,2]

3.0 Technical Status

Phase 1 technical efforts are limited to the four areas discussed in Section 2.1. No effort is being expended in the area of STC hydrogenation due to JPL support of the Union Carbide program. Also, no effort is being devoted to the development of CVD reactor vent product recovery technology. This technology is closely aligned with recovery system technology currently employed in the TCS CVD process at Hemlock Semiconductor Corporation. Phase 1 efforts thus consist of the following:

CVD Reactor Feasibility
Intermediate Dichlorosilane Reactor Development
DCS Process/Product Evaluation
EPSDU Design
1000 Tonne Plant Preliminary Design
1000 Tonne Plant Economic Analysis

A milestone chart detailing work to be accomplished in these four general areas is shown in Figure 2. The four major areas of technical activity are discussed in Sections 3.1 through 3.5.

3.1 CVD Reactor Feasibility

This task has been successfully completed; results are described in detail in previous reports.[1-3]

3.2 Intermediate Reactor Development

3.2.1 Objectives

The safe and efficient production of polycrystalline silicon from commercially purchased dichlorosilane, as well as the dichlorosilane produced by the process demonstration unit (PDU) installed at HSC, is to be demonstrated in an intermediate sized reactor. This task includes the following specific goals:

1. Design and installation of a reactor/feed system for safe handling of DCS from a 250 pound cylinder source.
2. Installation and checkout of a gas chromatographic analytical support system.
3. Integration of the intermediate sized reactor with the dichlorosilane PDU. The reactor/reactor feed system using purchased dichlorosilane contained in 250 lb. cylinders provides data to establish baseline conditions for integrated reactor/PDU operation.
4. Collection and evaluation of operational data on the DCS/hydrogen reactor system.

3.2.2 Reactor Feed System Design

The design and operating principles of this system were reported in the previous quarterly report[4].

The piping schematic of this system is presented in Figure 3.

3.2.3 Experimental Results

3.2.3.1 Reactor Performance

Four poly reactor deposition runs were made during the quarter with dichlorosilane fed from 250 pound cylinders via the feed system shown in Figure 4. Data relating to feed rate, deposition rate, conversion efficicncy, and power consumption from these runs are presented in Table 1 for the intermediate size reactor. Selected data at similar temperatures and feed conditions for the experimental reactor are presented in Table 2 for comparison.

Also contained in Table 1 are the trichlorosilane fed segments of the runs. Run segments 324-409-2 and 324-410-2 were judged most successful in terms of rod surface quality, smooth operation of the reactor, and limited silicon build-up on the interior bell jar wall. Run 324-409-5 experienced some difficulty in temperature control with a resultant rough rod surface. Segment 324-410-3 was carried out at low rod temperature which resulted in relatively poor performance in terms of deposition rate, conversion efficiency, and power consumption without reducing bell jar deposition problems.

Experimental reactor runs for the Intermediate reactor were segmented similarly to those for the experimental reactor. Segmentation[5] has been used successfully to minimize dichlorosilane purchase.

A comparison of Experimental Reactor (E) and Intermediate Reactor (I) performance with dichlorosilane feed is presented in Table 3. The Intermediate reactor performance with silicon feed rates of 4-5 g/h-cm is an average of three run segments (324-409-2, 324-410-2, 324-410-3).

Good agreement exists in all areas with somewhat higher conversion efficiency being observed in the intermediate size reactor being expected due to the longer residence times in the larger reactor and larger rod diameter. Power consumption in the intermediate size reactor is somewhat less due to a higher deposition rate.

A comparison of Intermediate Reactor results with dichlorosilane and trichlorosilane feed at corresponding diameters and temperatures is presented in Table 4.

Reducing the silicon deposited, conversion and power consumption values to unity for trichlorosilane (TCS) feed, Table 4 becomes:

Feed Type	Silicon Fed (gh-1cm-1)	Silicon Deposited (TCS = 1)	Conversion (TCS = 1)	Power Consumption (TCS = 1)
DCS	4-5	1.95	2.22	0.76
DCS	7-8	2.23	2.11	0.55
All DCS	4-8	2.03	2.19	0.70

Results with the production scale Intermediate Reactor indicate dichlorosilane to be superior to trichlorosilane as a feed material in the production of polycrystalline silicon. This type of performance is consistent with that required to meet the overall program objectives.

Deposition of silicon on the walls of the reactor vessel occurred in three of the four runs made with dichlorosilane feed. Reactor operating parameters were defined which should reduce, or eliminate this unwanted deposit without adversely affecting the deposition rate, conversion, and power consumption advantages of dichlorosilane.

3.2.3.2 Gas Chromatography Data

During intermediate reactor operation, feed and vent streams were sampled and analyzed by means of the Bendix gas chromotography - Perkin Elmer Sigma 10 data collection system. Gas chromotography data is presented in Table 5. The product silicon in this table is the calculated amount of silicon deposited within the reactor system which is determined by silicon and chlorine balances of the reactor feed and vent streams. This value, in effect, represents the conversion efficiency of the DCS reactor deposition system.

Comparison of the conversion and the conversion determined by actual rod weight (Table 1) is presented below:

	Conversion (Mole %)	
Run	GC Conversion	Rod Wt. Conversion
324-409-2	40.5	35.0
324-409-5	49.3	33.4
324-410-2	42.2	41.1
324-410-3	38.9	27.2

As mentioned previously, deposition of silicon on the walls of the bell jar occurred in three of the four runs that were made. This is confirmed by the data from runs 324-409-2, 324-409-5, and 324-410-3. Also, the data on run 324-410-2 indicates very little wall deposition of silicon (<3%). The gas chromotography results from this agree with visual inspection of the reactor vessel that very little silicon was deposited on the bell jar wall.

3.2.4 Integrated Reactor System

The temporary DCS feed system was dismantled for Reactor 324. Modifications of Reactor 324 (Model 8D intermediate reactor) feed system and integration with the DCS-PDU were completed and all systems checked.

In addition, a second polycrystalline silicon reactor (Model 8D) was modified for operation using DCS-H_2 feed supplied by the PDU. Two reactor operation is necessary for continuous operation of the DCS-PDU. Otherwise, large swings from full feed to total reflux conditions would require excess operating control time.

3.3 Dichlorosilane Process/Product Evaluation

3.3.1 General

The objectives of this task are:

1. Establish purity of DCS produced via catalyzed redistribution of TCS.

2. Permit characterization and optimization of the redistribution process through design, construction, and operation of a laboratory rearranger unit (PDU support) and a process development unit (PDU), and provide design information for the EPSDU and 1000 metric ton plant.

3. Provide sufficient DCS at a reasonable cost from the PDU to permit regular operation of an intermediate size CVD reactor at high feed rates.

These objectives will be met through dual tasks designed to provide a data base for PDU design and operation, and actual PDU design, construction, and optimization. The PDU support effort is discussed in Section 3.3.2 while PDU design-construction-operation activities are reviewed in Section 3.3.3.

3.3.2 PDU Support

No effort was expended in this area during this quarter.

3.3.3 PDU Design

The Sixth Quarterly Report contains the process diagram, equipment description, and control scheme explanation. No design changes have occurred.

3.3.3.1 PDU Construction

PDU construction was completed in early May. The final safety audit as well as a technician training program were also completed in May. Figures 5 - 11 show various phases of construction and components of the PDU, specifically:

Figure 5 - PDU Steel Tower and Transfer Piping.
Figure 6 - Completed PDU Facility.
Figure 7 - Ground Level View of the PDU Showing the Preheater, and Bottom of Distillation Column.
Figure 8 - Second Deck of PDU Showing the Filter Assembly and Reboiler Steam Control Valve.
Figure 9 - Top Deck of PDU Where the Condenser, Column Pressure Control Valve, and Relief Valve Assembly are located.
Figure 10 - Top Deck of PDU Where the DCS Take-Off Line and Hydrogen Line are Located.
Figure 11 - DCS-PDU Control Panel.

3.3.3.2 Standard Operating Procedures

The purpose of the standard operating procedure (SOP) is to provide steps and precautions necessary to safely manufacture and purify DCS for use in decomposition reactors to produce silicon. The SOP has been modified to eliminate reference to specific HSC buildings and safety policy numbers and forwarded to the Jet Propulsion Laboratory under separate cover.

3.3.4 PDU Start-Up

The PDU was purged and pressure checked prior to start-up. Start-up of the PDU occurred on 1 June 1981 as planned after minor problems were corrected. After instrument adjustments were made, the system ran smoothly and reliably with two decomposition reactors on line.

3.3.5 PDU Evaluation

No catalyst degradation has been observed in 3 weeks of operation. Laboratory kinetic evaluation represents pilot scale operation based on flow rate and temperatures required to achieve 10 mole percent conversion of TCS to DCS. Efforts to characterize the redistribution reactor are planned for the next quarter.

Pressure drop through the catalyst bed and temperature of the TCS feed required to achieve 10 mole percent DCS in the redistribution reactor effluent limit DCS production to 47 lb/hr using the 3 inch diameter redistribution reactor. Replacement of the 3 inch reactor with a 5 inch reactor is planned for next quarter so that a proper distillation evaluation can be obtained.

No distillation evaluation has occurred due to the limitation of 47 lb/hr DCS production in the redistribution reactor. No problems with distillation are apparent up to the 47 lb/hr DCS rate.

Pressure drop evaluations are incomplete because excessive pressure drop is periodically observed. When pressure drop is excessive, a procedure of back blowing the catalyst bed, which includes the containment screens, results in a return to normal pressure drop. The problem and corrective action are typical of plugging or blinding of the containment screens. This problem is probably the result of fine catalyst particles and dirt in the new system, and will subside with system operation. This phenomenon is a common occurrence in the start-up of new facilities.

3.4 Preliminary EPSDU Design

3.4.1 Safety, Description, and Design Status

No changes have occurred to the EPSDU design presented in the previous quarterly report.

3.4.2 Future Work

During the next quarter, mass and energy balances as well as equipment sizing for the EPSDU will be calculated for steady state operation by use of ASPEN. ASPEN (Advanced System for Process Engineering) developed at Massachusetts Institute of Technology under the auspices of the U. S. Department of Energy is a steady state process simulator. Advanced versions of ASPEN can do, in addition to mass-energy balances and equipment sizing, preliminary cost estimates, operating cost and profitability analysis. No activity is currently planed where the advanced versions of ASPEN would be used.

3.5 1000 Tonne/Yr. Plant Design

No design changes have been made, or are expected, for the 1000 tonne/yr plant design presented in the Sixth Quarterly report.

Future work will include sensitivity analysis with respect to mole percent, DCS in reactor feed, percent conversion of DCS in feed to silicon, and electrical power consumption per kilogram. The mole percent DCS and percent conversion effect the capital expenditure required for a 1000 tonne/yr facility. The electrical power consumption effects the direct manufacturing cost.

4.0 Conclusions and Recommendations

The DCS feed/intermediate reactor system worked well. Few operational problems were encountered while feeding the intermediate size reactor. Four polysilicon deposition runs were completed in the intermediate size reactor using dichlorosilane fed from 250 pound cylinders. Results from the intermediate reactor are consistent with those obtained from the experimental reactor. Data collected will provide the basis for establishing the conditions for PDU/Intermediate reactor operating conditions for Phase 2.

PDU construction was completed in May, 1981. PDU start-up occurred June 1, 1981. After initial instrument adjustments were made, the PDU ran smoothly for the month of June. No catalyst degradation was observed during this time.

5.0 Program Schedule/Plans

The program is proceeding according to plan. Efforts planned in the various task areas in accordance with the Program Schedule shown in Figure 2 are summarized below:

1. Experimental Reactor Feasibility/Optimization (3.1)

   Task completed - Mixed feed and reactor cooling will be investigated with the intermediate size reactor.

2. Intermediate Dichlorosilane Reactor Development (3.2)

   The task requiring characterization of intermediate size reactor with DCS from cylinders has been completed. Modifications to two intermediate size reactors for accepting DCS/H_2 feed from the PDU have been completed. Reactor evaluation will commence during the next reporting period.

3. Dichlorosilane Process/Product Evaluation (3.3)

   PDU construction has been completed. PDU start-up occurred June 1, 1981. Evaluation of e 3" diameter and 5" diameter redistribution reactors will occur during the next reporting period. Polysilicon product will be analyzed for boron, donor, and carbon.

4. Preliminary EPSDU Design (3.4)

Preliminary design complete. No further effort in this task area prior to start of detailed design.

5. Phase 2

A Phase 2 work statement will be completed during the next reporting period which will include a modified program plan and milestone schedule.

6.0 New Technology

No new technology was developed during this quarter.

7.0 References

1. Hemlock Semiconductor Corp., "First Quarterly Report", Low-Cost Silicon Solar Array Project , DOE/JPL Contract No. 955533, January, 1980.

2. Hemlock Semiconductor Corp., "Second Quarterly Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 955533, May, 1980.

3. Hemlock Semiconductor Corp., "Third Quarterly Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 955533, August, 1980.

4. Hemlock Semiconductor Corporation, "Sixth Quarterly Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 955533, May, 1980, Section 3.1.3.

5. Hemlock Semiconductor Corp., "Second Quarterly Report", Low-Cost Silicon Solar Array Project, DOE/JPL Contract No. 955533, May, 1980, Section 3.1.3.

FIGURE 1. SCHEDULE OF EFFORT BY PHASES

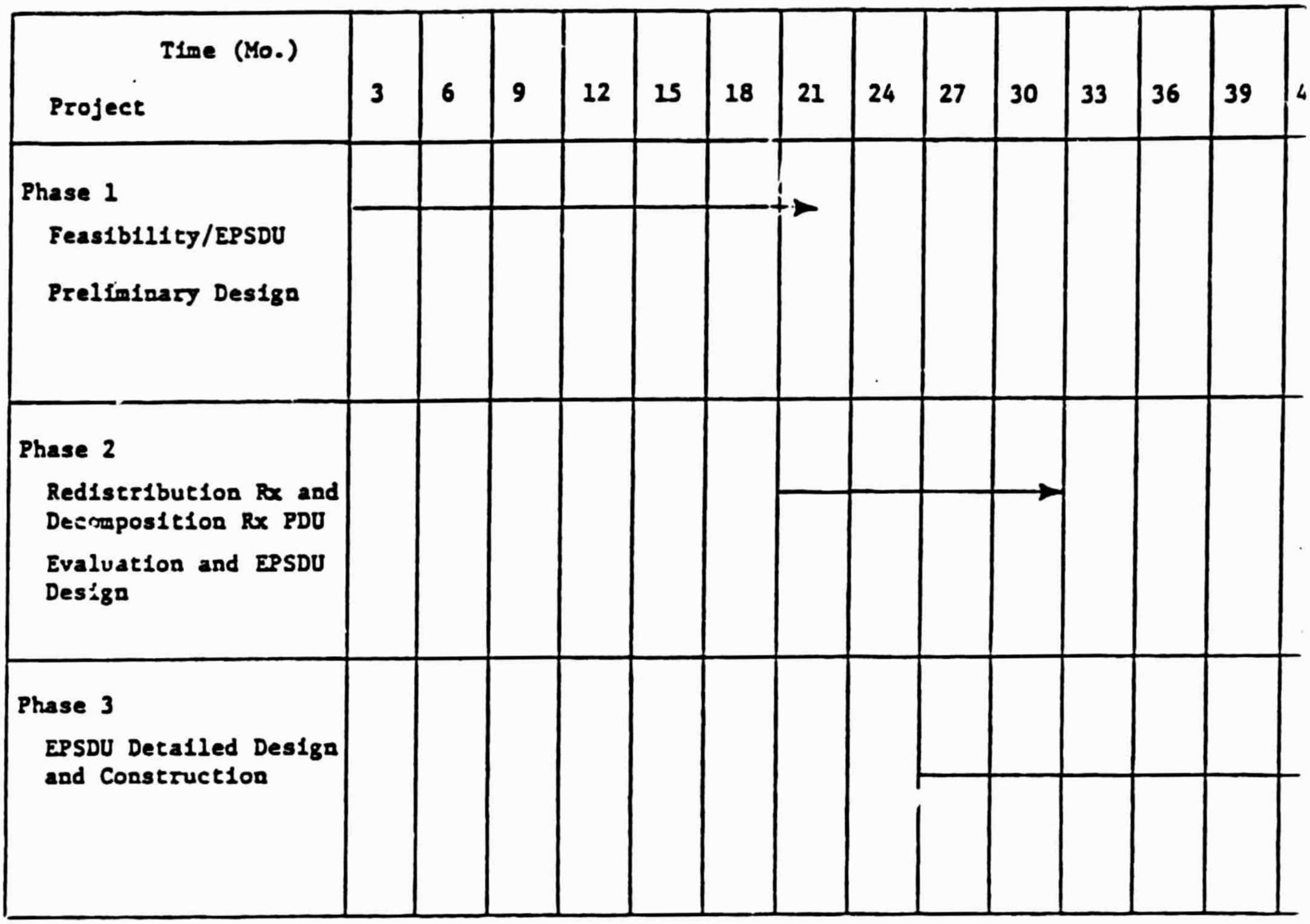

FIGURE 2. PROGRAM PLAN/MILESTONE SCHEDULE

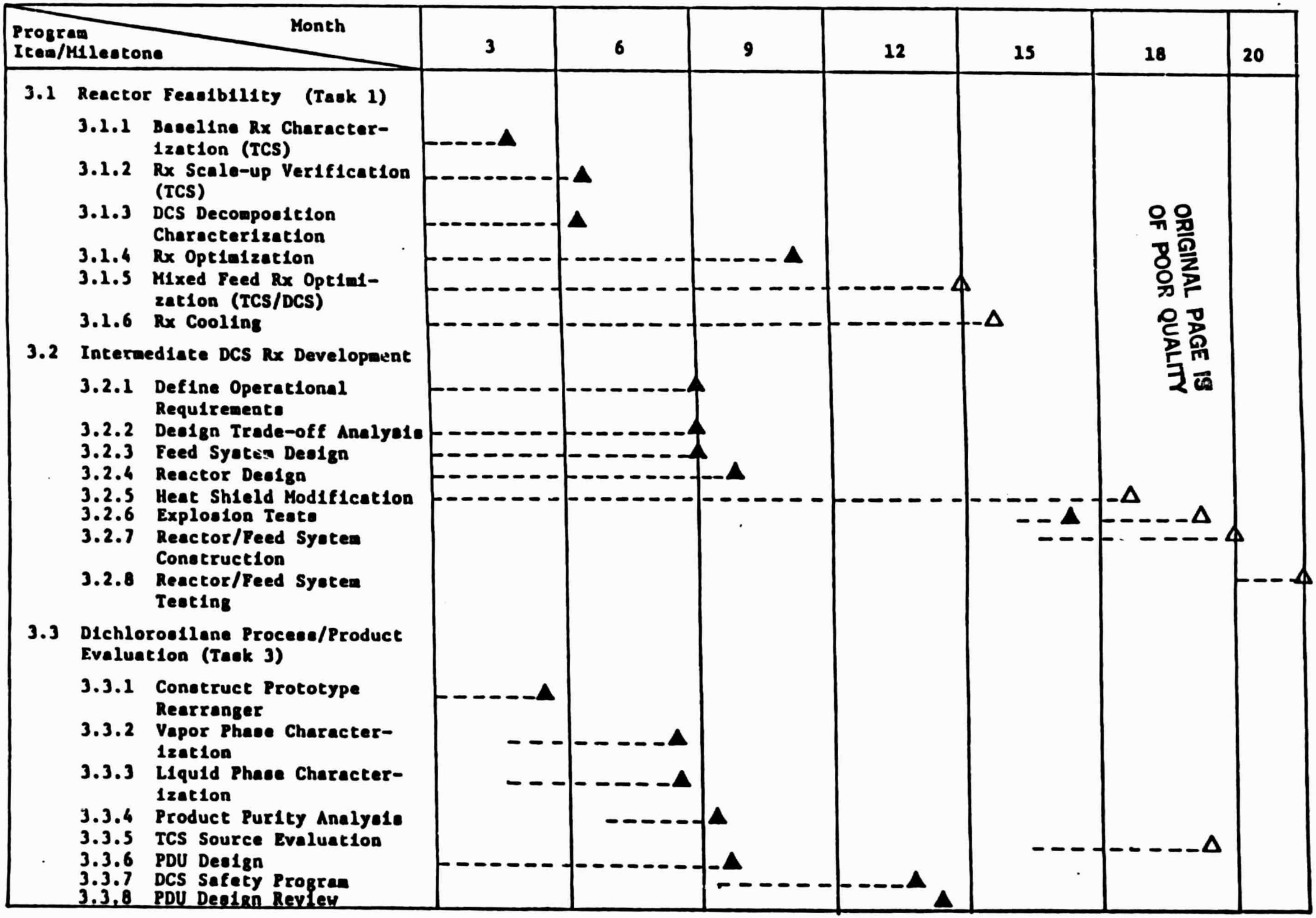

FIGURE 2. PROGRAM PLAN/MILESTONE SCHEDULE (Continued)

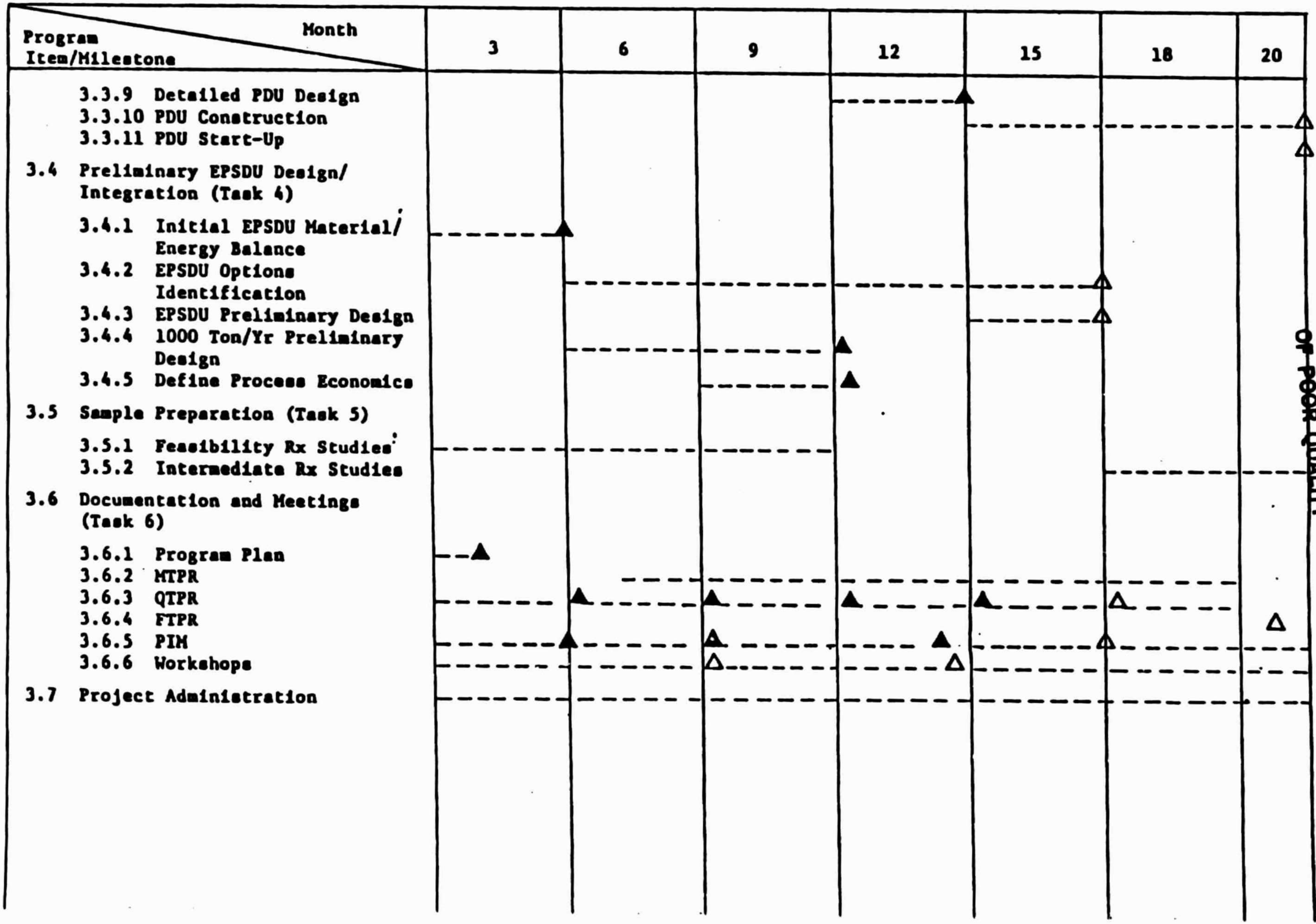

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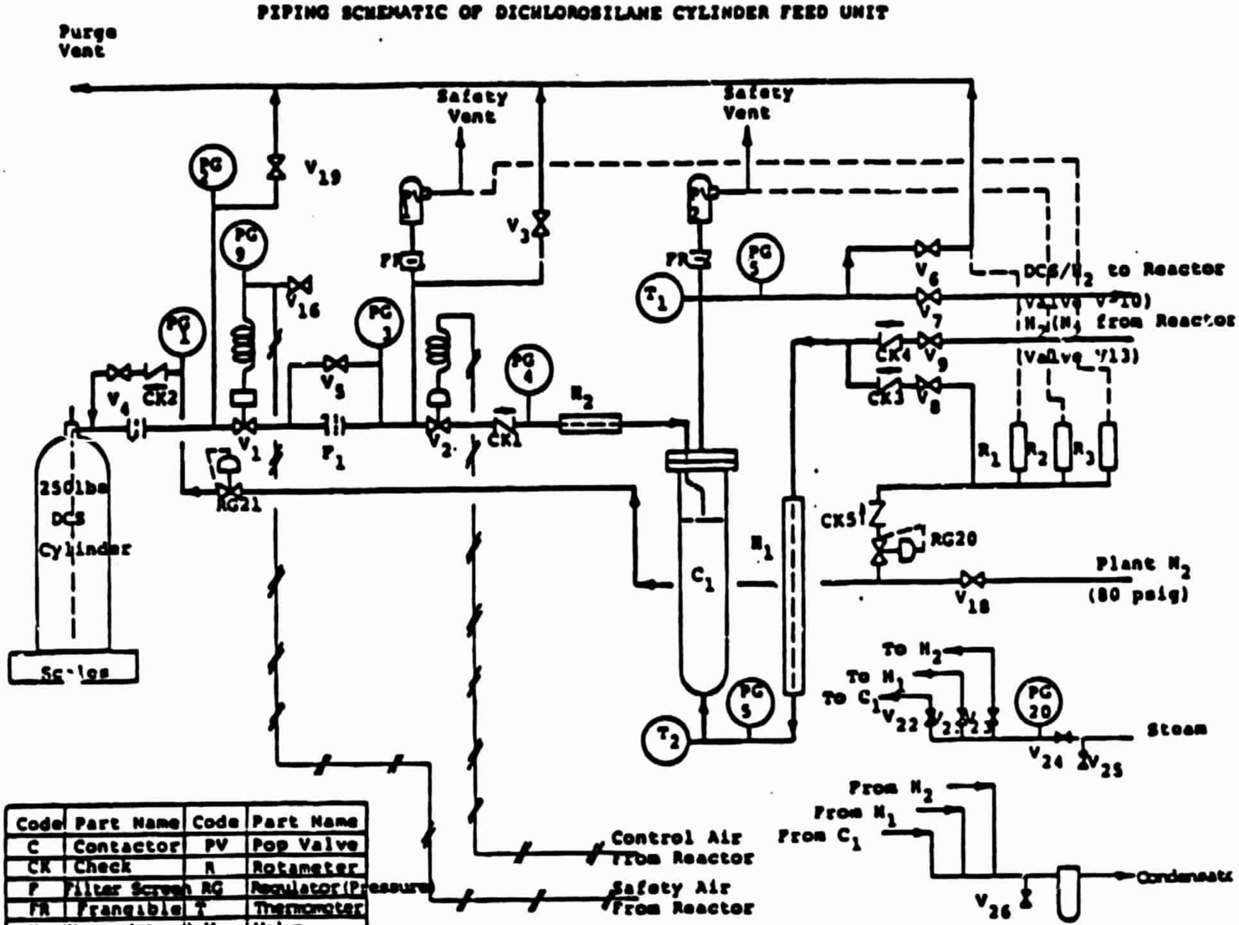
FIGURE 3.
PIPING SCHEMATIC OF DICHLOROSILANE CYLINDER FEED UNIT



Code	Part Name	Code	Part Name
C	Contactor	PV	Pop Valve
CK	Check	R	Rotameter
F	Filter Screen	RG	Regulator(Pressure)
FR	Frangible	T	Thermometer
H	Heater(steam)	V	Valve
O	Orifice		

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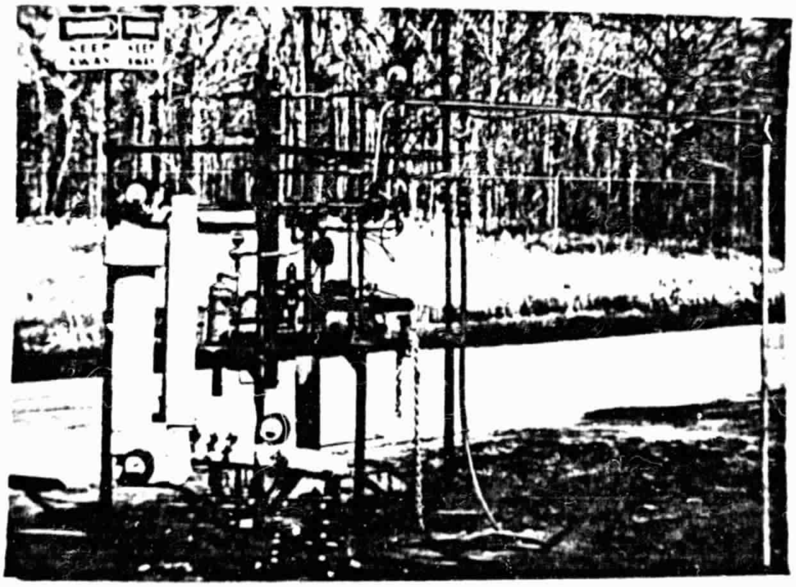

Figure 4 Photograph of Dichlorosilane Cylinder Feed Unit

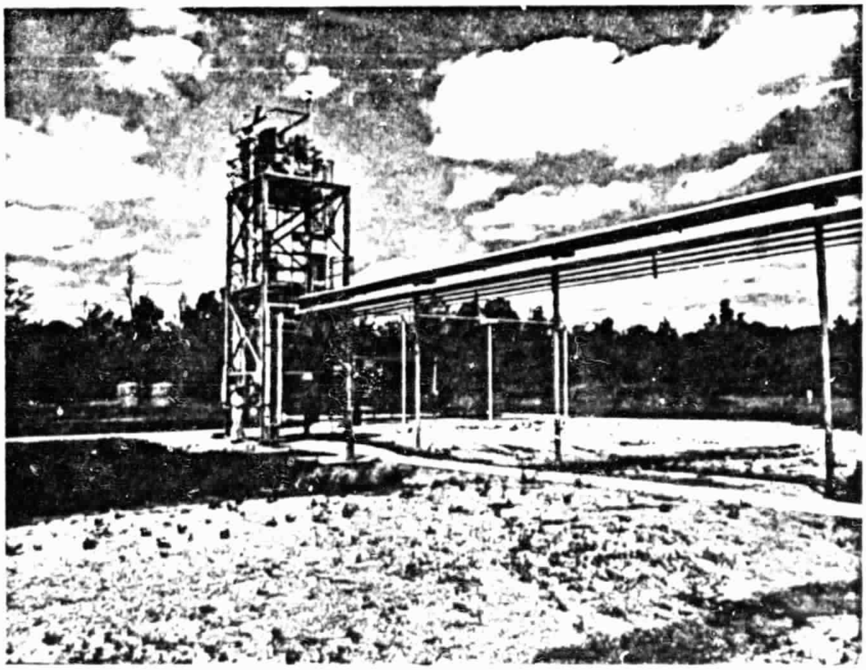

Figure 5 PDU Steel Tower and Transfer Piping

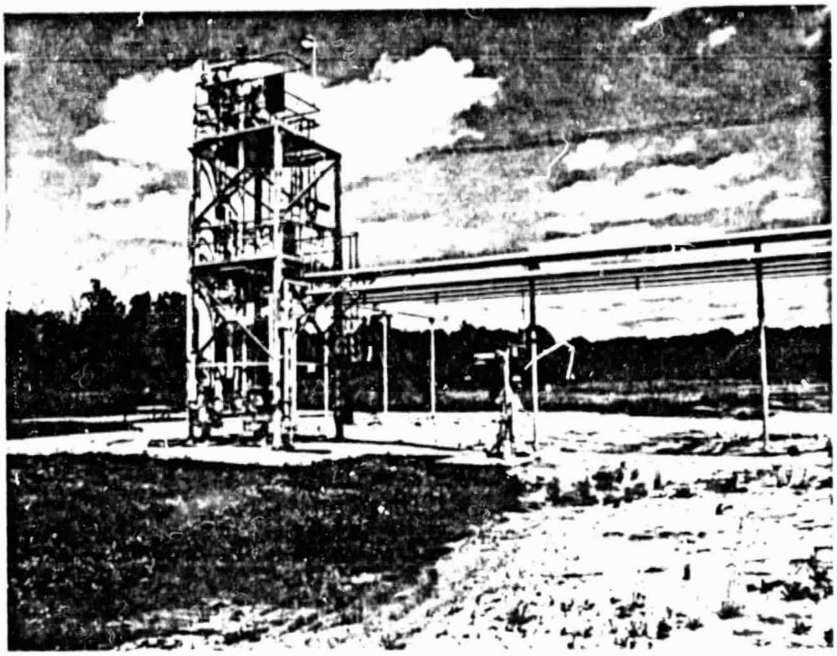

Figure 6 Completed PDU Facility

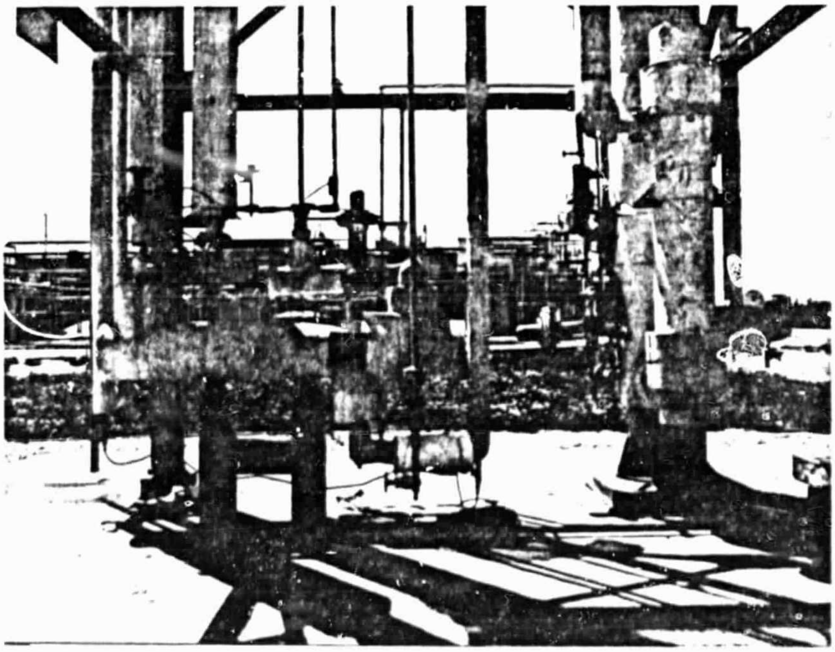

Figure 7 Ground Level View of the PDU
Showing the Preheater and
Bottom of Distillation Column

Table 1. Summary of Intermediate Reactor Performance

Run Number	Feed Type	Run Time (Hrs.)	Rod Diameter (mm)	Silicon Fed (gh^{-1} cm^{-1})	Silicon Deposited (gh^{-1} cm^{-1})	Conversion (Mole %)	Consumption (kWh/kg)
324-409-1	TCS	35.0	7 - 33.9	4.62	0.58	12.5	133
324-409-2	DCS	13.2	33.9 - 47.7	4.43	1.56	35.2	88
324-409-3	TCS	12.0	47.7 - 54.3	5.35	1.03	19.2	151
324-409-4	TCS	12.0	54.3 - 60.9	7.33	1.16	15.8	135
324-409-5	DCS	7.9	60.9 - 67.5	7.49	2.59	33.4	74
324-410-1	TCS	35.0	7 - 37.5	4.79	0.71	14.8	N.A.
324-410-2	DCS	14.1	37.5 - 51.9	4.06	1.67	41.1	101
324-410-3	DCS	12.5	51.9 - 59.6	4.62	1.26	27.2	136

Table 2. Experimental Reactor Performance - Selected Data

Run Number	Feed Type	Run Time (Hrs.)	Rod Diameter (mm)	Silicon Fed (gh^{-1} cm^{-1})	Silicon Deposited (gh^{-1} cm^{-1})	Conversion (Mole %)	Consumption (kWh/kg)
394-063-6	DCS	4.0	35.8 - 40.4	4.56	1.603	35.2	96.3
394-067-3	DCS	9.5	31.2 - 39.1	4.56	1.069	23.5	114.1
394-063-5	DCS	8.0	31.6 - 35.8	5.66	0.648	40.7	165.8
394-067-4	DCS	10.0	39.1 - 44.7	5.66	0.859	54.1	177.3
394-069-2	DCS	7.5	26.8 - 32.4	5.66	0.822	51.4	167.8
394-063-4	DCS	4.0	28.8 - 31.6	8.21	0.774	33.7	138.8
394-068-3	DCS	8.0	31.5 - 38.5	8.21	1.121	49.2	133.9
394-067-2	DCS	7.0	26.4 - 31.2	8.39	0.723	31.7	143.6
394-068-4	DCS	9.0	38.5 - 43.1	8.39	0.753	33.0	162.0
394-068-2	DCS	5.0	26.9 - 31.5	8.39	0.983	43.2	131.7

Table 3. Experimental and Intermediate Size Reactor Performance Comparison

Reactor	Silicon Fed ($gh^{-1}cm^{-1}$)	Silicon Deposited ($gh^{-1}cm^{-1}$)	Conversion (mole %)	Power Consumption (kWh/kg Si)
E	4-5	1.34	29.4	105
E	5-6	0.78	48.7	170
E	8-9	0.87	38.2	142
I	4-5	1.50	34.5	108
I	7-8	2.59	33.4	74

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Table 4. Comparison of DCS and TCS Reactor Performance

Feed Type	Silicon Fed (gh-1cm-1)	Silicon Deposited (gh-1cm-1)	Conversion (mole %)	Power Consumption (kWh/kg Si)
DCS	4-5	1.50	34.5	108
TCS	4-5	0.77	15.5	142
DCS	7-8	2.59	33.4	74
TCS	7-8	1.16	15.8	135
All DCS	4-8	1.77	34.2	98
All TCS	4-8	0.87	15.6	140

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Table 5. Gas Chromotography Data from Intermediate Reactor

Run Number	Feed Type	Products (Moles/100 Moles Silicon Fed)				
		Si	HCl	DCS	TCS	STC
324-409-2	DCS	40.5	8.8	6.0	34.0	17.3
324-409-5	DCS	49.3	27.7	1.5	28.1	21.1
324-410-2	DCS	42.2	12.5	6.0	34.1	17.1
324-410-3	DCS	38.9	6.2	8.5	35.3	16.4