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FIFTH QUARTERLY PROGRESS REPORT

1 OCTOBER TO 31 DECEMBER 1980

on

DEVELOPMENT OF A POLYSILICON PROCESS BASED ON CHEMICAL VAPOR DEPOSITION

(PHASE 1)

prepared by

J. McCormick, K. Sharp, A. Arvidson and
D. Sawyer

March 1981

JPL Contract 955533

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HSC 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.

Experimental data generated by Hazards Research Corp. indicate that the ease of ignition and explosion severity of dichlorosilane (DCS)/air mixtures is substantially attenuated if the DCS is diluted with hydrogen. DCS/hydrogen mixtures will accordingly be transported in preference to transfer or storage of pure DCS.

Redesign of the PDU (Process Development Unit) to accommodate new safety-related information is complete. All major process equipment has been ordered, and construction of the facility is underway, with startup scheduled for June, 1981.

Similarly, a feed system to supply an intermediate sized reactor from purchased DCS cylinders has been extensively redesigned. System construction is in progress, with completion targeted for March, 1981.

Several different sources of trichlorosilane were used to generate a mixture of redistributed chlorosilanes via Dowex ion exchange resin. The unseparated mixtures were then fed to an experimental reactor in which silicon was deposited and the deposited silicon analyzed for electrically active impurities. At least one trichlorosilane source provided material of requisite purity for PDU integration with the existing HSC recovery system.

Silicon grown in the experimental reactor from commercically purchased DCS was converted to single crystal material and solar cells fabricated. Cell efficiencies met or exceeded baseline cell efficiencies.

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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 currrent 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.

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Silicon grown in the experimental reactor from commerically purchased DCS was converted to single crystal material and solar cells fabricated. Cell efficiencies met or exceeded baseline cell efficiencies.

2.0 Introduction

The following abbreviations will be used in this section: DCS for DCS; TCS for trichlorosilane; STC for silicon tetrachloride.

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 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 DCS as a feedstock 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 T/Y plant will consist of: the hydrogenation of STC to produce TCS; synthesis of DCS via redistribution of TCS; high temperature decomposition of DCS to produce polycrystalline silicon; and recovery of decomposition byproducts. STC, a major by-product of TCS redistribution and minor byproduct 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 efforts are being expended in the area of STC hydrogenation due to JPL support of the Union Carbide program. Neither are efforts 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 DCS is to be demonstrated in an intermediate sized reactor. This task includes the following specific goals:

- design and installation (or modification) of a reactor/feed system for safe handling of DCS from a 250 pound cylinder source.
- installation and checkout of a gas chromatographic analytical support system.
- collection and evaluation of operational data on the DCS/hydrogen fed reactor system.

3.2.2 Reactor and Feed System Design

3.2.2.1 Dichlorosilane-Hydrogen Vaporizer System

The system for feeding a mixture of DCS and hydrogen has been designed and is shown schematically in Figure 3. The vaporizer system consists of a 250 pound DCS cylinder from which liquid is withdrawn and fed to a packed bed countercurrent contactor for vaporization and mixing with preheated hydrogen. The resultant mixture is then fed to the polysilicon decomposition reactor. Because of the flammability and explosion hazards associated with pure DCS, the vaporizer system itself is located remotely from the reactor area and equipped with pneumatic control and safety valving so that its operation can be controlled or shut down from the reactor site. Personnel exposure is thus limited and a "safety buffer zone" will exist in the event of fire or explosion at the vaporizer site. As a further precaution this system will only be operated if the ambient temperature is below 47 ^{O}F (the boiling point of DCS).

Installation of this feed system is currently underway and scheduled for completion during the next quarter.

3.2.2.2 Reactor System

Work is currently underway to modify the polysilicon decomposition reactor for safe and efficient interfacing with the DCS-hydrogen vaporizer system. This modification is scheduled for completion during the next quarter, providing a suitable heat shield design can be developed. It has been determined that the present heat shield used with this reactor (see Section 3.5.2) is not of sufficient mechanical integrity to prevent personnel injury in the event of an explosion within the decomposition reactor.

3.2.2.3 Gas Chromatographic Support System

A Bendix gas chromatograph for feed and vent product analysis has been installed and integrated with an existing Perkin Elmer Sigma 10 data collection system for use with the polysilicon reactor employed in this task. Operation of this system will be checked out during the next quarter.

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 activities are reviewed in section 3.3.3.

3.3.2 PDU Support

General. The overall objective of the PDU Support laboratory program is to provide data to support the design and safe and efficient operation of the DCS mini-plant (PDU). The chemical reactions and principles of operation of this plant are described elsewhere[3]. Information is needed in the following general areas:

- Process Stream Analysis
- Catalyst Stability
- Redistribution Kinetics
- Product and Process Stream Safety

The experimental approaches to data acquisition in these areas have been discussed in previous Quarterly Reports.(1-3)

3.3.2.1 Process Stream Analysis

The gas chromatographic sampling and analytical techniques described in previous quarterly reports(1,2) has been used without difficulty throughout this quarter.

3.3.2.2 Catalyst Stability

No additional direct work was conducted this quarter on the stability of Dowex MWA-1 resin.

3.3.2.3 Redistribution Kinetics

No additional work was conducted during this quarter on liquid phase TCS redistribution kinetics. As reported previously, the second order kinetic rate constant (k_o/a) was found to be ca 0.20 min-1 at 77°C and and linear flow rates greater than 25 ft/hr. This value is considerably higher--and more favorable--than originally anticipated. The rate constant k_o/a is defined as the open tube kinetic rate constant divided by the void volume fraction of the catalyst particles. A void volume fraction of 0.5 was used for Dowex resin.

3.3.2.4 Purity of Polysilicon from Laboratory Rearranger

The purity of material from the laboratory scale liquid phase rearranger was evaluated during this quarter by collecting a 6 liter sample of redistributed chlorosilanes, and using these as feedstock for depositing a silicon rod by chemical vapor deposition. A 100 inch bed of Dowex MWA-1 resin was used for redistribution. The deposited silicon rod was analyzed for boron, donor (phosphorus) and aluminum by a multipass zone refining technique and for carbon by Fourier transform infrared (FTIR) analysis. Results are shown below.

Ref No	Silicon Rod Feed Source	Boron (ppba)	Donor (ppba)	Aluminum (ppba)	Carbon (ppma)
022	77 °C Dowex	Bed Conditioning Run - No Rod Grown			
023	Control	0.19	1.1	0.30	0.5
024	24 °C Dowex	0.48	1.4	0.09	0.5
025	Control	0.15	1.7	0.18	0.3
026	77 °C Dowex	0.69	1.2	0.06	0.4

These results indicate a slight degree of boron contamination, with respect to semiconductor grade silicon, caused by redistribution using Dowex. While such boron levels would have no impact on the utility of the product for solar grade silicon, they do pose a problem for PDU integration with the HSC recovery system.

3.3.3 PDU Design and Construction

This section describes PDU safety related design features, system operation and construction status.

3.3.3.1 PDU Design

Based on the potential hazards of DCS, several design changes have been incorporated into the final design. These changes are:

1. No DCS storage will be included in the facility.

2. Minimal DCS hold-up in process equipment has been designed.

3. Mixtures of hydrogen and DCS rather than pure DCS will be transported through the production facility. The design eliminates the need for a DCS vaporizer in the production area.

4. The redistribution reactor and distillation column still will be located remotely from HSC production facilities.

5. The system will be operated with controls that are sufficiently remote to minimize operator exposure to the system while DCS is present during normal operating or maintenance conditions.

6. The overpressure relief system will vent at a point remote from the distillation column.

An additional design change that is not safety related is that the TCS/STC mixture from the bottom of the still will be returned to the HSC distillation train.

Figure 4 represents the final design for the PDU. TCS from the feed source is heated in the preheater to 80^{O}C prior to entering the redistribution reactor. In the redistribution reactor the TCS is converted to a near-equilibrium mixture which contains about 10% DCS, 80% TCS and 10% STC.

This mixture is fed to the distillation column. Feed flow is controlled with feed forward control from the overhead product take off, and with feed back trim from column differential temperature.

DCS is removed as vapor from the column overhead on a demand basis to satisfy the ratio control for hydrogen/DCS. Hydrogen flow is initiated by low pressure in the feed header line to the decomposition reactors.

The bottoms flow from the still is established by level control in the reboiler. High level initiates flow of the TCS/STC mixture from the still to the HSC distillation train.

3.3.3.2 PDU Construction

Construction of the PDU was initiated in this quarter. Grading and drainage provisions for the site are complete, as is concrete work including footings and equipment pads. All major equipment has been ordered, and final design of the piping and instrumentation has been initiated.

The next quarter will be the major construction period for the PDU. Completion of construction is targeted for May, 1981, as indicated in the milestone charts.

3.3.4 PDU Evaluation

No effort in the area during this reporting period.

3.4 Preliminary EPSDU Design

This section summarizes the general objectives of the EPSDU and reviews status of the preliminary design.

3.4.1 Objectives

The following have been established as general objectives for an EPSDU:

1. Demonstrate technology readiness.

2. Substantiate technology claims of low electricity consumption, 40% conversion of DCS to silicon, and a deposition rate of 3 kg/hr per reactor.

3. Evaluate the equipment and procedures to produce silicon via chemical vapor decomposition of DCS, and to produce DCS via redistribution of TCS.

3.4.2 EPSDU Status

No design changes have occurred in the EPSDU since the last reporting period. Efforts relating to the EPSDU currently include evaluation of factors, such as power supply and raw material availability, that impact Hemlock Semiconductor Corporation. No new design concepts are anticipated for the EPSDU irrespective of the final scale of the facility.

3.4.3 Recovery Design Considerations

When vent gas composition data from the decomposition reactors are obtained from the PDU, the EPSDU recovery design will be reviewed. This review will not change the overall design concept, although it may change some of the sizing details.

3.5 SAFETY-RELATED DATA ON DICHLOROSILANE

As discussed in a previous quarterly report[3], DCS has been reported to undergo combustion that is easily initiated and unusually violent. Accordingly, Hazards Research Corporation (HRC) was engaged to conduct a series of tests relating to the flammability and hydrolytic characteristics of DCS. Following is a review of that work and final experimental values reported by HRC.

3.5.1 Test Methods

Autoignition Temperature Determination

Autoignition temperature determination was by methods slightly different than the standard ASTM. Experimental details are described in the previous Quarterly Report.

Explosion Severity Scan

Explosion severity provides an indication of combustion rate by observation of pressure-time characteristics of combustion in a closed vessel. Details of the test method are reported in the previous Quarterly Report.

Hydrolytic Behavior

Explosive Output Trials

Test methods for the hydrolytic and explosive output tests are described in the previous Quarterly Report.

Results and Discussion

Autoignition Temperature (AIT)

Material	AIT
DCS	58 ±5°C
DCS/H_2	255 ±5°C
Redistributed TCS (10% DCS)	130 ±5°C

Irrespective of the precise value of the AIT of DCS in dry air, it is clear that the thermal ignition requirements of such mixtures are very small.

Explosion Severity

Material	Expt.	$(dP/dt)_{max}$ psi/sec	P(static) psig	P(dynamic) psig
DCS	1	1.1×10^{6}	112	280
	2	1.1×10^{6}	125	280
	3	0.8×10^{6}	125	225
DCS/H_2 10/90		5.4×10^{4}	90	
H_2		2.4×10^{4}		
Redistributed TCS		1.2×10^{3}	84	

Figure 9 is a plot of maximum pressure rise (psi/sec) vs. concentration of DCS in air.

From this test it was established that a detonation can occur with DCS. More detailed discussion of detonation characteristics is contained in the previous Quarterly Report.

These results are obtained from equipment that confines the combustion products. It is possible that the high peak rate of pressure rise is affected by confinement; however, it must be assumed that partially confined or unconfined vapor clouds of DCS could detonate under some conditions. The PDU design and location is based on such an assumption.

Hydrolytic Tests

These experiments were summarized in the previous Quarterly Report.

Explosive Output Studies

Analysis of slides from the first test (see 4th Quarterly Report) and movies of the second explosion test indicated that the second test produced less energy than the first. In the first test one Bikini gage was blown off its support and only one section of sixteen of the plastic enclosure was left in place. In the second test only two of the sixteen sections of plastic were removed by the blast, although the others burned in a secondary fire. The Bikini gages were not dislodged. One can postulate that the second trial, with liquid loading of DCS to the cube did not provide complete vaporization and mixing of the DCS and air. From this series of tests it can not be concluded that an unconfined vapor cloud detonation is impossible. Factors such as initial temperature, partial confinement, and efficiency of mixing seem to effect the combustion process significantly.

Summary of Results From Hazards Research Corp.

The HRC experimental program has confirmed the suspicions that DCS/air mixtures are both very easily ignited, and that the explosive potential from such ignitions is unusually high. Alternative designs and procedures for the PDU and intermediate and production reactors have accordingly been formulated and are discussed in their respective sections.

Information indicates that DCS can be safely handled. Safety features such as equipment location or explosion confinement must be carefully considered and implemented as required for each system containing DCS, to assure personnel safety.

3.5.2 Jar Explosion Tests

Because of the extreme fire and explosion hazard associated with pure dichlorosilane an explosion test of the reactor system planned for use in the "Intermediate Reactor Development" phase of this program (See Section 3.2) was considered necessary to insure that the system would maintain sufficient integrity in the event of explosion within the reactor to prevent personnel injury and minimize equipment damage. The reactor system consists of a support table, quartz reaction jar and a heat shield. Of primary concern is the containment of quartz fragments by the heat shield.

The explosion test was conducted late in this quarter and consisted of ignition of a mixture of dichlorosilane (3.5 mole %); hydrogen (31.2 mole %) and air (65.0 mole %) contained within the quartz reaction jar at atmospheric pressure. This mixture was calculated to give the most severe explosion and the approx. 1:10 dichlorosilane to hydrogen ratio was selected to reflect the feed ratio planned for use with the intermediate reactor. Data on the explosion itself was collected by a high speed pressure recording system and by video cameras.

Although analysis of the data is still in progress the following preliminary conclusions can be made:

- the heat shield did contain broken quartz fragments
- several parts of the heat shield were blown off at sufficient velocity to injure personnel
- the explosion was a deflagration and not a detonation

Although the reactor system cannot be deemed of sufficient integrity at this time for use in the "Intermediate Reactor Development" phase of this project, sufficient data was collected from this initial explosion test to enable proper design of this system, particularly the heat shield. The new heat shield should be ready and a second explosion test conducted during the next quarter.

3.6 1000 Ton/Year Plant Design

Line identification tables relating to Figures 5 through 8, HSC Process Flow Diagrams, were not included in the 4th Quarterly Progress Report. Figures 5 through 8 summarize the overall HSC process while line identification is contained in Table 1. No effort was spent in this area during the present quarter.

4.0 Conclusions and Recommendations

Extensive redesign and revision of operating characteristics of the Process Development Unit (PDU) and intermediate reactor feed systems necessary to accommodate new safety-related information were successfully concluded during this reporting period.

The construction phases of both of the above systems were initiated during this quarter. Progress on PDU construction included site preparation and pouring of a concrete pad for process equipment and a structural steel tower. All major equipment for the PDU has been ordered.

The feasilibility of generating high quality silicon from dichlorosilane via CVD was further established during this reporting period by fabrication of solar cells from (commercially supplied) DCS-grown silicon. Cell efficiencies met or exceeded the baseline cell efficiency.

Polycrystalline silicon was also grown from redistributed trichlorosilane without any intervening purification. The product was evaluated by electrical measurements and Fourier transform infrared techniques on a zone refined sample. Boron, phosphorus and carbon concentrations were essentially at the levels required by the semiconductor industry.

5.0 Program Schedule/Plans

Except in the areas of intermediate reactor DCS feed and PDU construction, as noted above, the program is proceeding according to or exceeding 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)
   Has been completed.

2. Intermediate Dichlorosilane Reactor Development (3.2)
   Complete construction; start up first quarter '81.

3. Dichlorosilane Process/Product Evaluation (3.3)
   Continue PDU construction completion scheduled in May 1981.

4. Preliminary EPSDU Design (3.4)
   Identify plant, reactor locations.
   Identity/select alternatives for interaction with HSC recovery system.
   Modify Process Flow Diagrams to indicate changes in PDU design.

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, September, 1980.

APPENDICIES A

TABLE 1.

FLOW AND MOLE % FOR SYSTEM SHOWN ON (Fig. 5)

Line Number	lb/hr Flow	Weight % H2	HCl	MCS	DCS	TCS	STC	Si	Comment
101	321							100	Metallurgical Grade
102	6.8								Copper
103	-								
104	-								
105	327.8							99	1% Copper
106	-								
107	327.8							99	1% Copper
108	35.5	100							
109	-								
110	-								
111	327.8							99	1% Copper H_2 not shown
112	180	100							
113	18500	1.16					98.8		Si not shown
114	18710	1.				25	74		
115	67512				0.6	25.1	74.3		
116	86222	1.			0.6	25	73.4		
117	18530	1.			0.6	25	73.4		
118	18530	1.			0.6	25	73.4		
119	182					25.5	74.5		Si not shown in composition
120	182					25.5	74.4		
121	18350				0.6	25.1	74.3		
122	180	100							
123	180	100							
124	180	100							
125	18350				0.6	25.1	74.3		
126	18264						100		
127	17907						100		
128	18284						100		
129	326.4						100		

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TABLE 1 (Continued)

FLOW AND MOLE % FOR SYSTEM SHOWN ON (Fig. 6)

Line Number	lb/hr Flow	Weight % H2	HCl	MCS	DCS	TCS	STC	Si	Comment
201	2070				12.1	55.2	32.7		
202	18350				0.6	25.1	74.3		
203	-								
204	-								
205	-								
206	18350				0.6	25.1	74.3		
207	17907						100		
208	-								
209	-								
210	-								
211	-								
212	28666				10.8	89.2			
213	28666				8.7	91.3			
214	26153			0.2	7.5	78.5	13.8		
215	26153					100			
216	-								
217	-								
218	-								
219	2508			2.8	91.5	5.7			
220	2508			2.8	91.5	5.7			
221	2508			2.8	91.5	5.7			

TABLE 1 (Continued)

FLOW AND MOLE % FOR SYSTEM SHOWN ON (Fig. 7).

Line Number	lb/hr Flow	Weight % H2	HCl	MCS	DCS	TCS	STC	Si	Comment
301	2508			2.8	91.5	5.7			
302	-	100							
303	2508			2.8	91.5	5.7			
304	2197		5.8		11.4	52.1	30.7		
305	-								
306	-								Recovery Flows
307	-								Proprietary
308	-								
309	2070				12.1	55.2	32.7		
310	127		100						
311	-								
312	32.8								Produced in Decomposition Reactor
313									Water
314									Aqueous HCl Waste
	279								Silicon Produced

APPENDICIES B

FIGURE 1. SCHEDULE OF EFFORT BY PHASES

Time (Mo.) Project	3	6	9	12	15	18	21	24	27	30	33	36	39	42
Phase 1 Feasibility/EPSDU Preliminary Design	—	—	—	—	—	—	→							
Phase 2 Redistribution Rx and Decomposition Rx PDU Evaluation and EPSDU Design							—	—	—	→				
Phase 3 EPSDU Detailed Design and Construction									—	—	—	—	—	→

FIGURE 2. PROGRAM PLAN/MILESTONE SCHEDULE

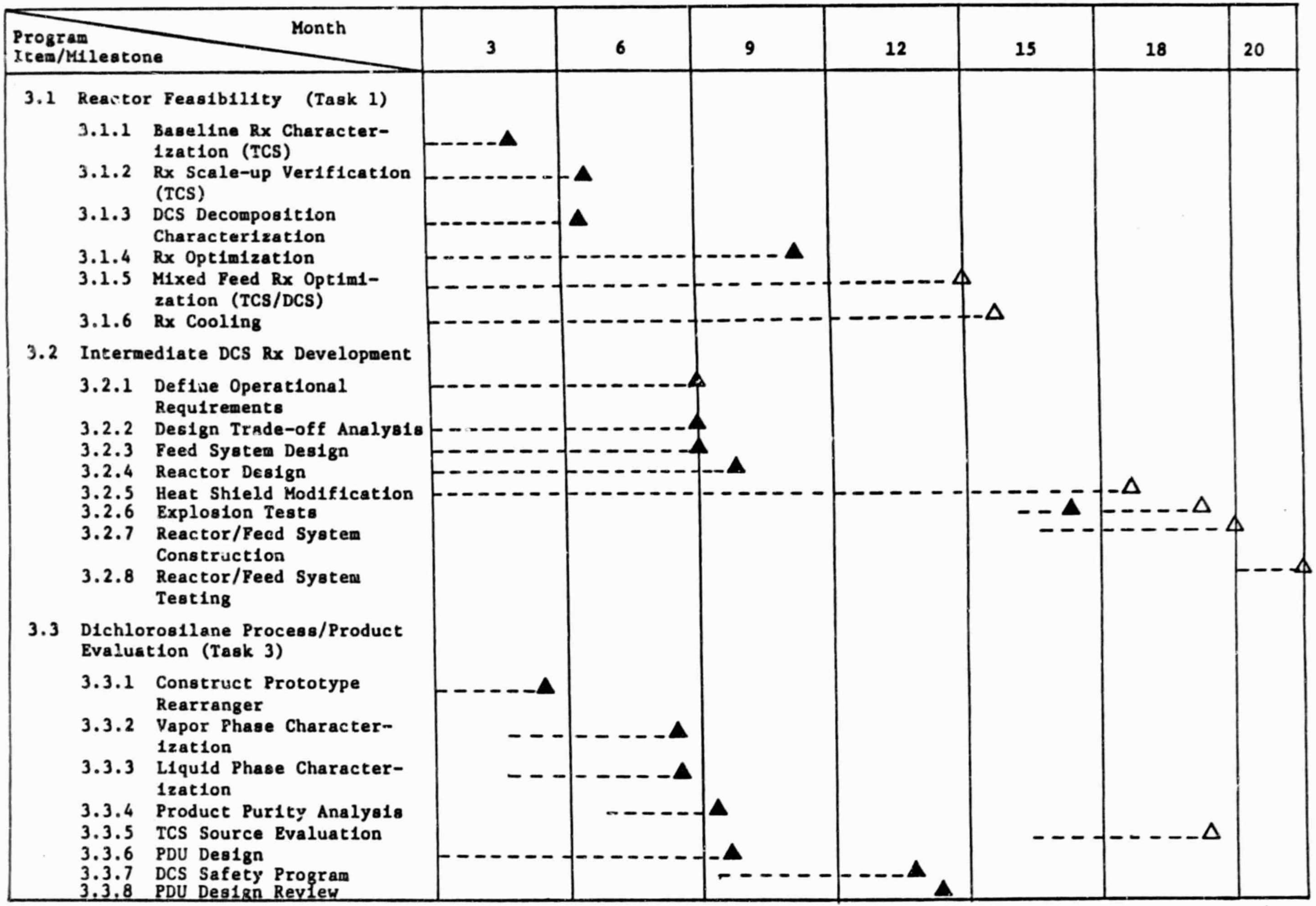


FIGURE 2. PROGRAM PLAN/MILESTONE SCHEDULE (Continued)

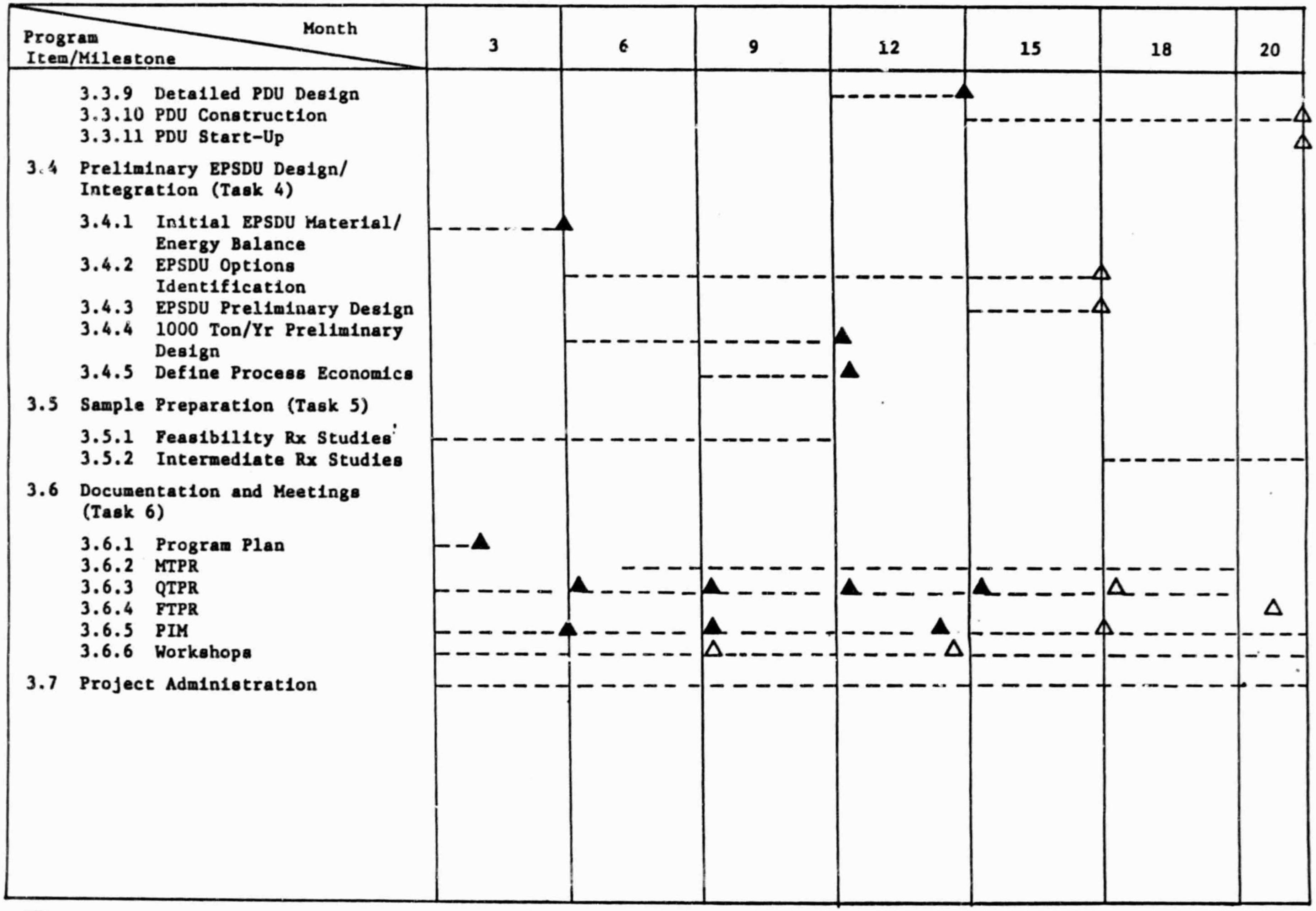
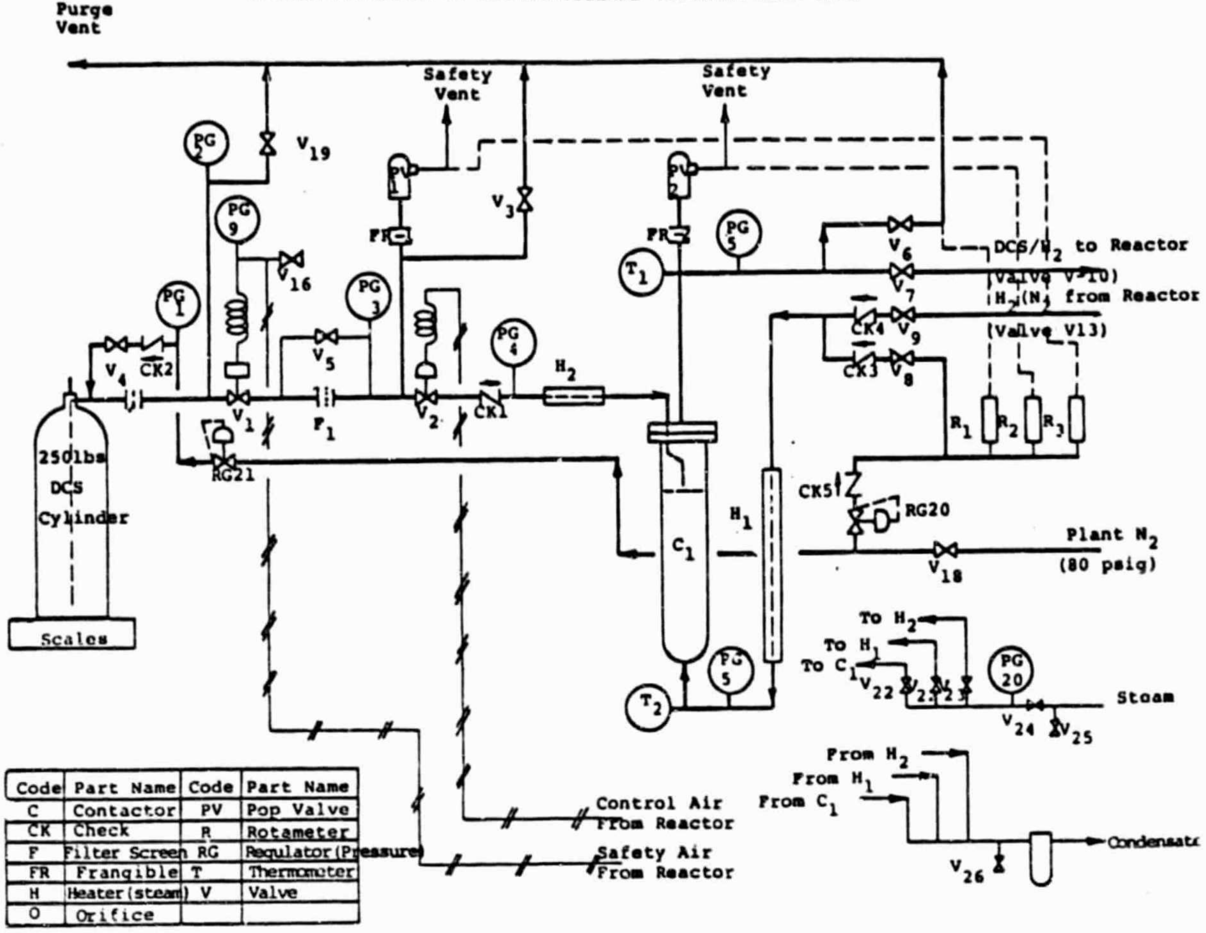


FIGURE 3.

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		

3/2/81

FIGURE 4.

DCS-PDU FLOW DIAGRAM

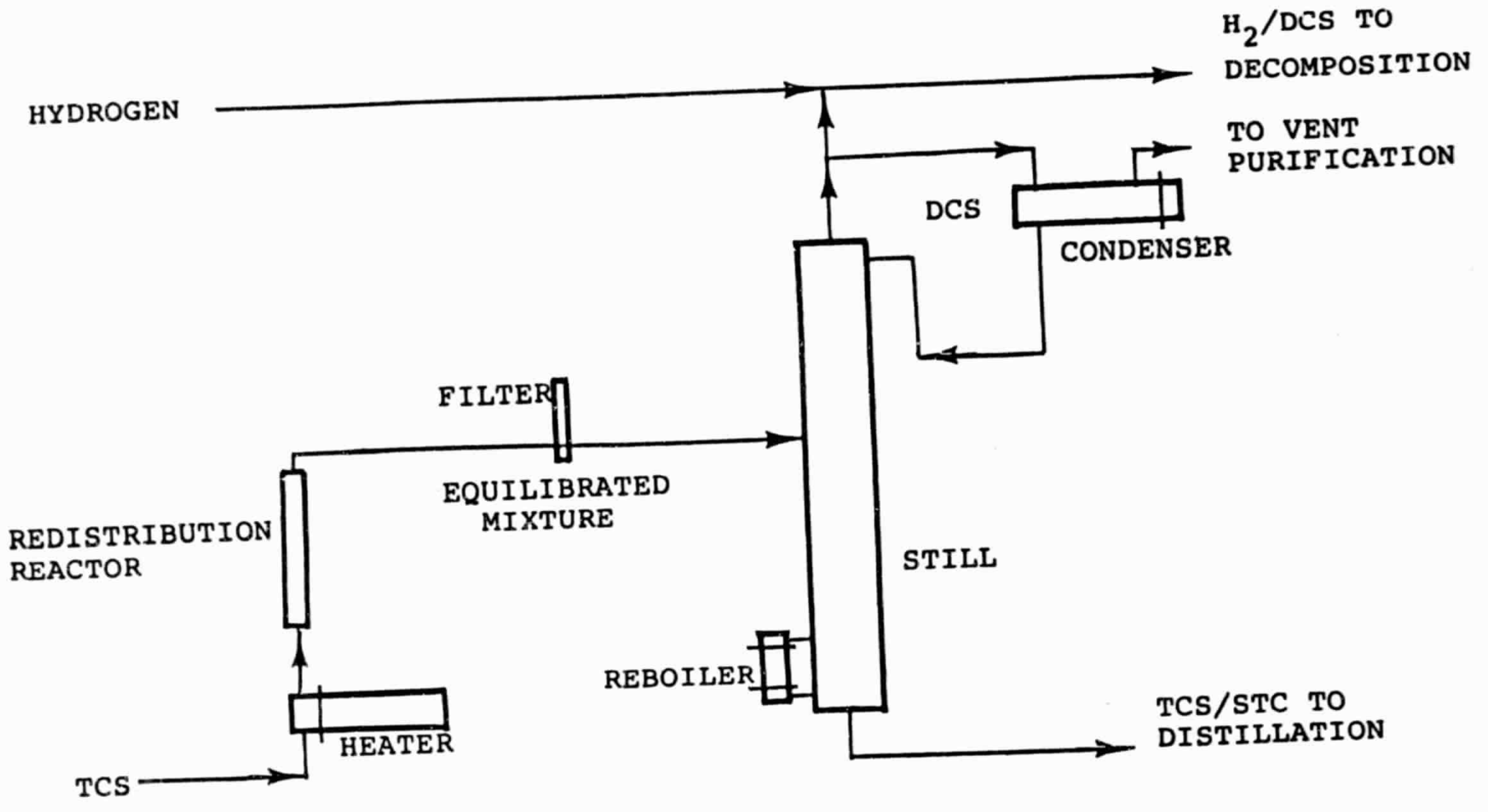


FIGURE 5.

HSC PROCESS

SILICON TETRACHLORIDE
HYDROGENATION SYSTEM

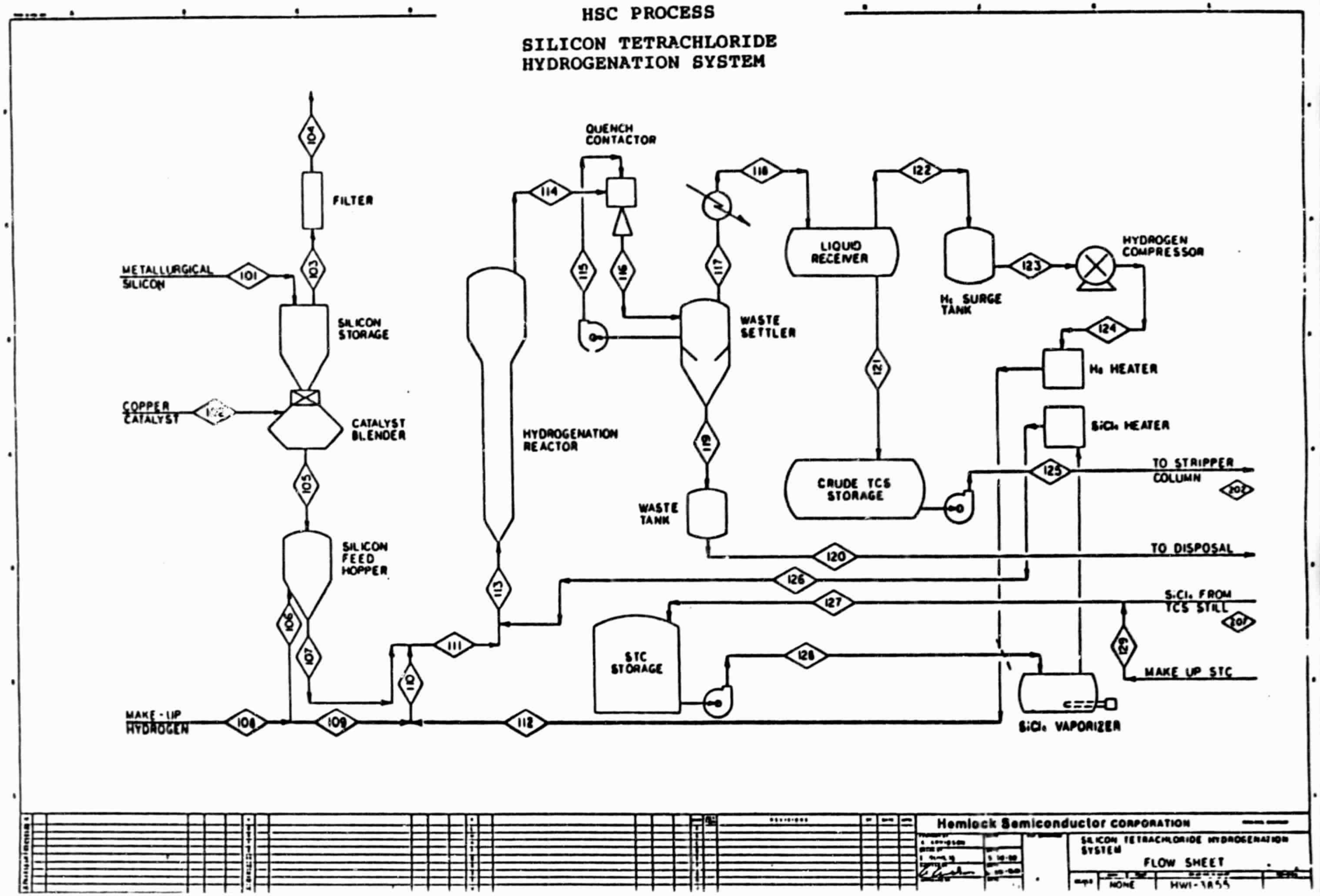


FIGURE 6.

HSC PROCESS

DICHLOROSILANE PRODUCTION
AND PURIFICATION

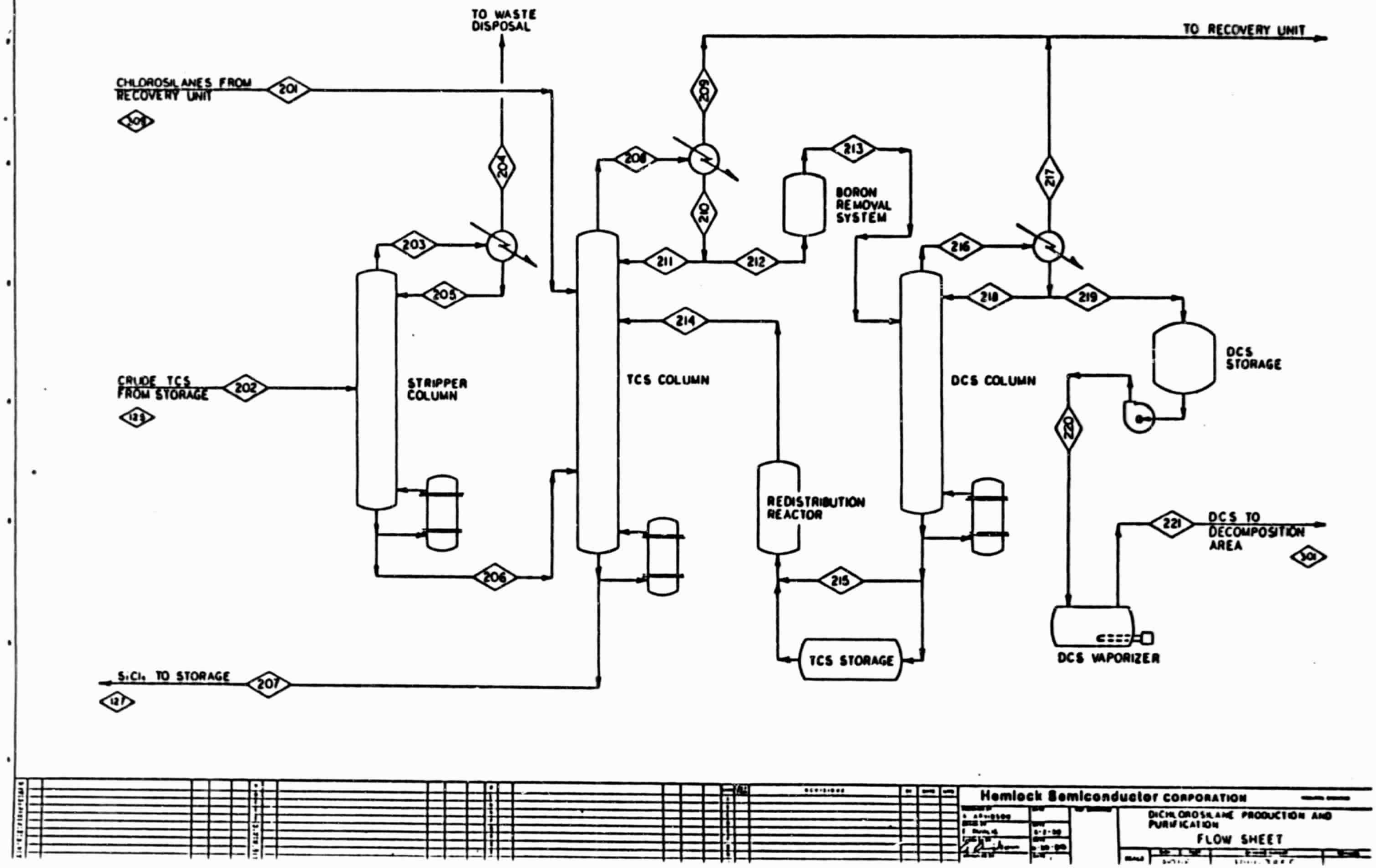
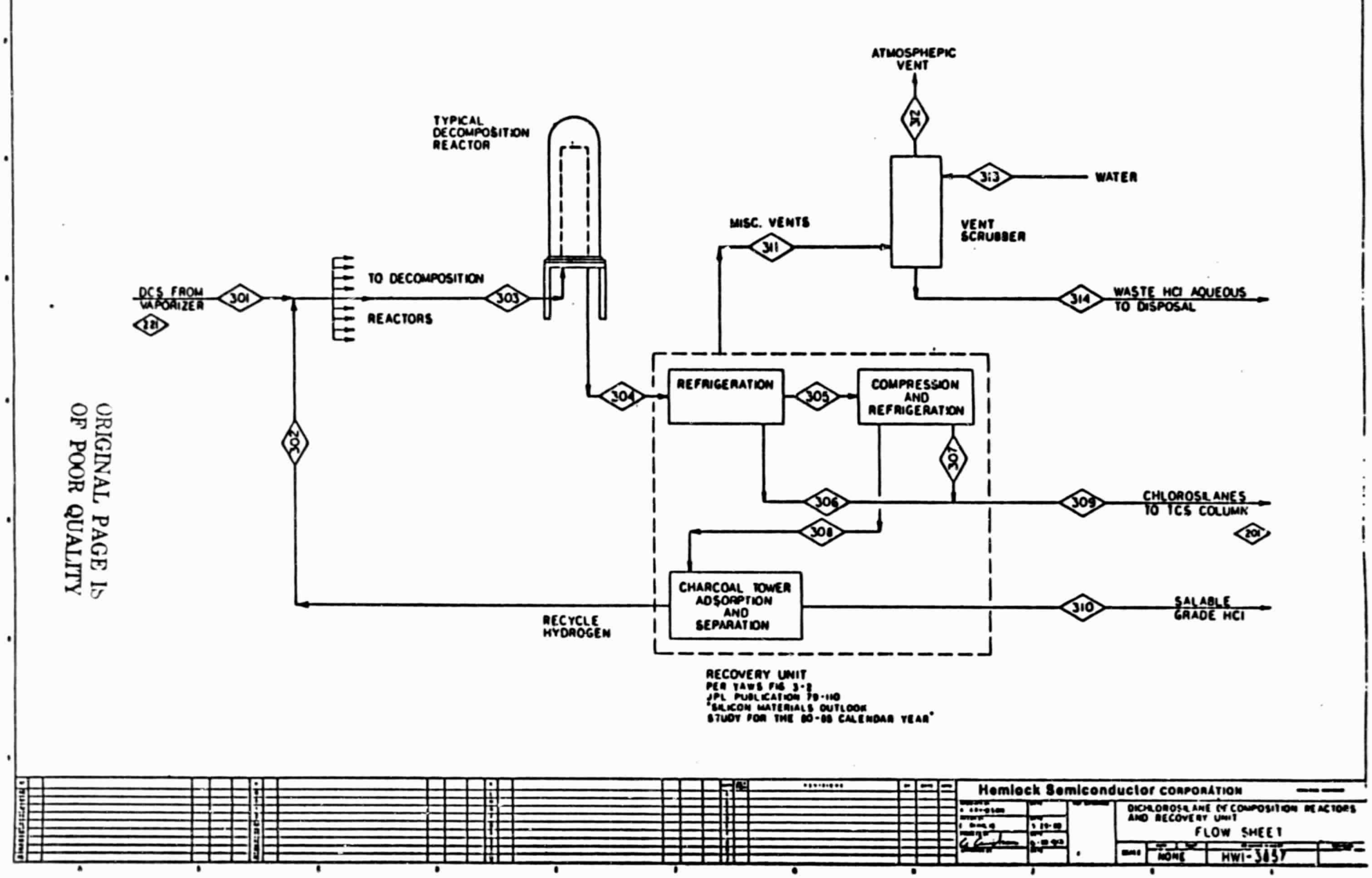


FIGURE 7.

HSC PROCESS

DICHLOROSILANE DECOMPOSITION REACTORS AND RECOVERY UNIT



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OF POOR QUALITY

FIGURE 8.

HSC PROCESS

DCS PRODUCTION UNIT WITH HIGH AND LOW PURITY TCS STREAMS

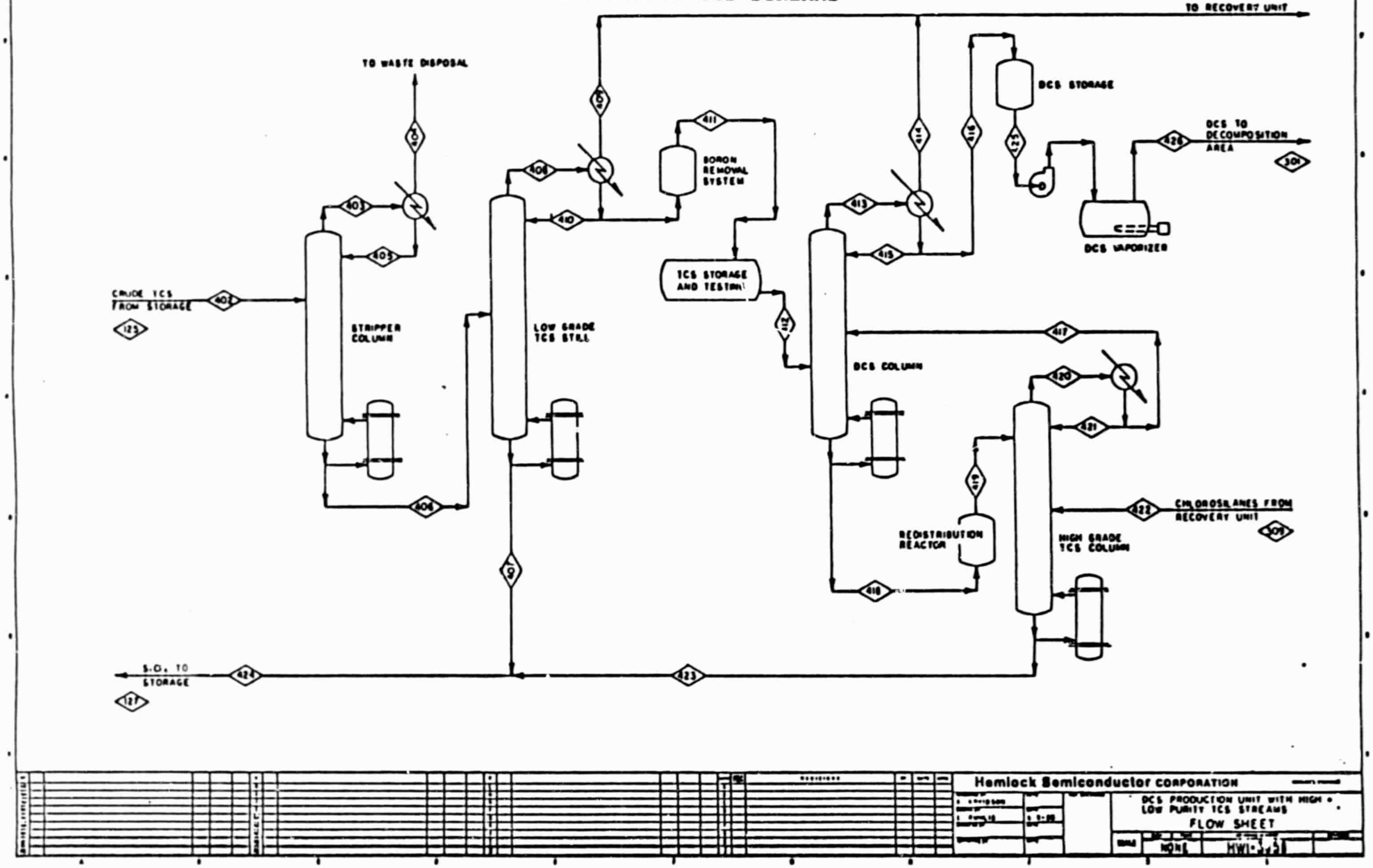

FIGURE 9. PRESSURE RISE RATE FOR DCS EXPLOSIONS IN 10 LITER SPHERE