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PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL TASK I

QUARTERLY TECHNICAL PROGRESS REPORT (I)

MARCH, 1978

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ABSTRACT

Major activities during this reporting period centered on process system properties, chemical engineering and economic analyses.

In analyses of process system properties, major efforts focused on properties of silicon tetrachloride which is the source material for several alternate processes under consideration for solar cell grade silicon production. The status and progress are reported for physical, thermodynamic and transport property data.

Experimental determination of gaseous thermal condutivity of silicon source materials was continued. Initial results for gas thermal conductivity of silicon tetrafluoride and trichlorosilane are reported in respective temperature ranges of 25 to 400 C and 50 to 400 C. There have been no previously reported experimental values for the thermal conductivity of trichlorosilane in this temperature range.

For chemical engineering analyses, the preliminary process design for the original silane process of Union Carbide was completed for Cases A and B, Regular and Minimum Process Storage. Two cases were presented because of the large recycle requirements for this process, necessitating considerable tankage for material storage. Included are raw material useage, utility requirements, major process equipment list, and production labor requirements. Seventy-six major pieces of process equipment are required for Case A versus fifty-eight for Case B.

The preliminary process design results for Cases A and B were used for economic analyses. Because of the large differences in surge tankage between major unit operations the fixed capital investment varied from $19,094,000 to $11,138,000 for Cases A and B, respectively. The product cost for Case A is $5.54/lb of silane versus $4.58/lb of silane for Case B.

For the silane process, Union Carbide engineering-research personnel revised their original flowsheet for a more optimum arrangement of major equipment, raw materials and operating conditions. The initial issue of the revised flowsheet (Case C) for the silane process indicated favorable cost benefits over the original scheme. This includes higher pressure silicon tetrachloride hyrogenation for incresed trichlorosilane yield with lower recycle requirements, higher pressure distillation not requiring expensive low temperature refrigeration, and improved raw materials not requiring hydrogen chloride purchase.

The revised silane process (Case C) should provide the following cost benefits:

- lower capital cost
- lower raw material costs
- lower operating labor costs

TABLE OF CONTENTS

I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

A. SILICON TETRACHLORIDE PROPERTIES

Analyses of process system properties of silicon source materials was continued during this reporting period.

Primary efforts were devoted to properties of silicon tetrachloride which is under consideration for solar cell grade silicon production in several alternate processes. Progress in areas of data collection, analysis, estimation and correlation are summarized below for those properties required in the performance of the chemical engineering analyses:

		Prior	Current
1.	Vapor Pressure, P_V	80%	90%
2.	Heat of Vaporization, ΔH_V	60%	70%
3.	Gas Heat Capacity, C_p^o	80%	90%
4.	Liquid Heat Capacity, Cp	45%	60%
5.	Density, ρ_L	45%	60%
6.	Surface Tension, σ_L	45%	60%
7.	Gas Viscosity, η_G	45%	60%
8.	Liquid Viscosity, η_L	45%	60%
9.	Gas Thermal Conductivity, λ_G	45%	60%
10.	Liquid Thermal Conductivity, λ_L	45%	60%
11.	Heat of Formation, ΔH_f°	45%	60%
12.	Gibb's Free Energy of Formation, ΔG_f°	45%	60%

B. THERMAL CONDUCTIVITY INVESTIGATION

During this reporting period the experimental determination of gaseous thermal conductivity of silicon source materials was continued.

The thermal conductivity of silicon tetraflouride (SiF_4) has been determined between 25° and 400°C (Table IB-3 and Figure IB-6). The values obtained in this study agree to within ±3% with previously reported experimental data for silicon tetrafluoride (ref. 40).

The thernal conductivity of trichlorosiland ($SiHCl_3$) has been determined between 50°C and 400°C (Table IB-4 and Fighre IB-7). There have been no previously reported experimental data for gaseous thermal conductivity of trichlorosilane.

TABLE IB-3

Preliminary Results for Gaseous Thermal Conductivity of Silicon Tetrafluoride

Temperature	Gaseous Thermal Conductivity		
°C	$mWcm^{-1}\ K^{-1}$	$Cal\ sec^{-1}cm^{-1}C^{-1}$	$Btu\ hr^{-1}ft^{-1}F^{-1}$
29	0.150	35.95×10^{-6}	8.69×10^{-3}
47	0.159	38.00×10^{-6}	9.19×10^{-3}
100	0.191	45.75×10^{-6}	11.06×10^{-3}
150	0.220	52.46×10^{-6}	12.68×10^{-3}
200	0.248	59.22×10^{-6}	14.32×10^{-3}
250	0.283	67.71×10^{-6}	16.37×10^{-3}
300	0.303	72.44×10^{-6}	17.51×10^{-3}
350	0.331	79.06×10^{-6}	19.11×10^{-3}
394	0.361	86.38×10^{-6}	20.88×10^{-3}

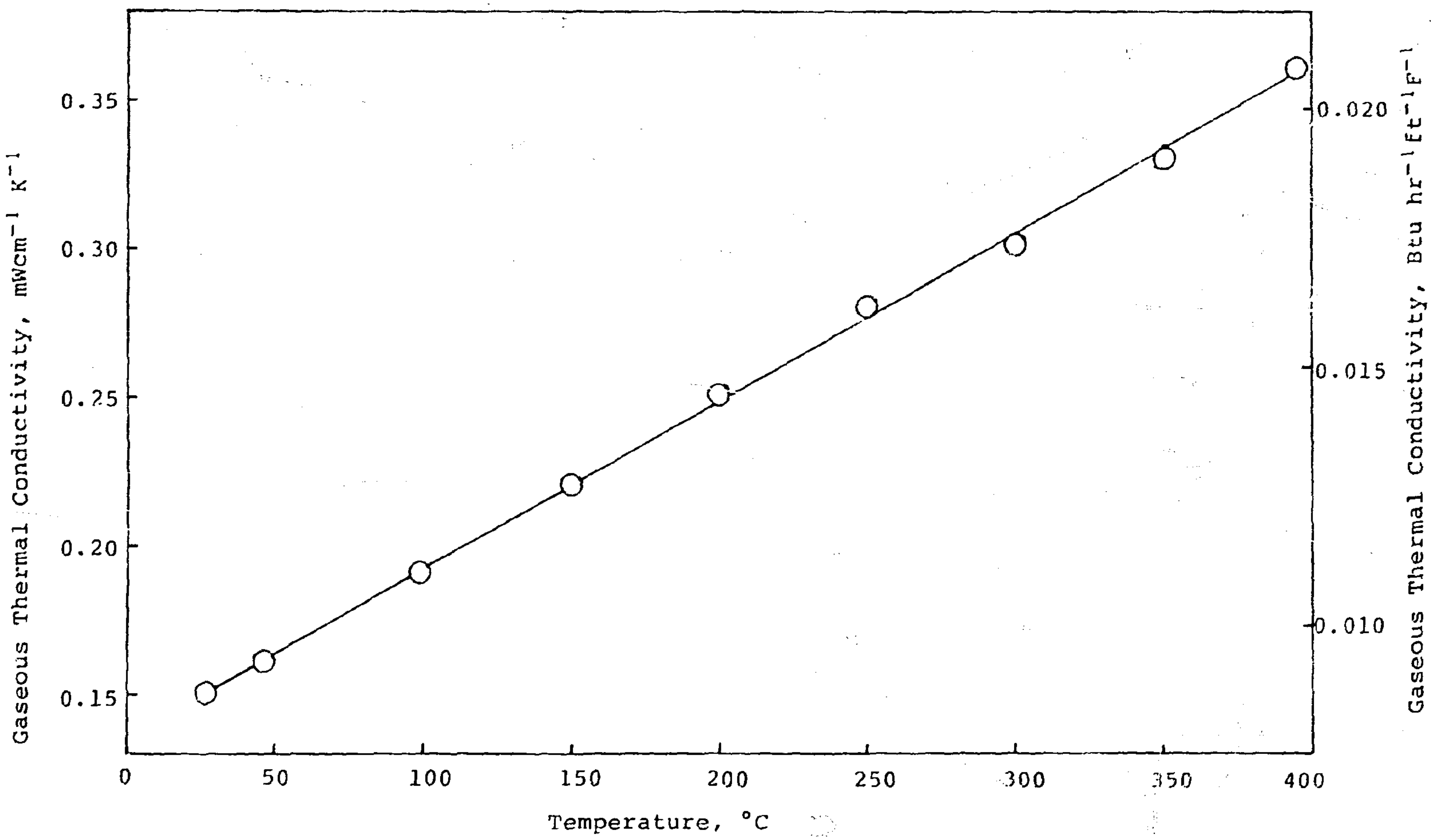


Figure IB-6. Thermal Conductivity of Silicon Tetrafluoride

TABLE IB-4

Preliminary Results for Gaseous Thermal Conductivity of Trichlorosilane

Temperature	Gaseous Thermal Conductivity		
C°	$MWcm^{-1}\ K^{-1}$	$Cal\ sec^{-1}cm^{-1}C^{-1}$	$Btu\ hr^{-1}ft^{-1}F^{-1}$
48°	0.093	22.28×10^{-6}	5.39×10^{-3}
100°	0.111	26.05×10^{-6}	6.41×10^{-3}
150°	0.129	30.76×10^{-6}	7.44×10^{-3}
200°	0.148	35.30×10^{-6}	8.53×10^{-3}
250°	0.167	39.84×10^{-6}	9.63×10^{-3}
300°	0.188	44.81×10^{-6}	10.83×10^{-3}
350°	0.207	49.40×10^{-6}	11.94×10^{-3}
394°	0.226	54.11×10^{-6}	13.08×10^{-3}

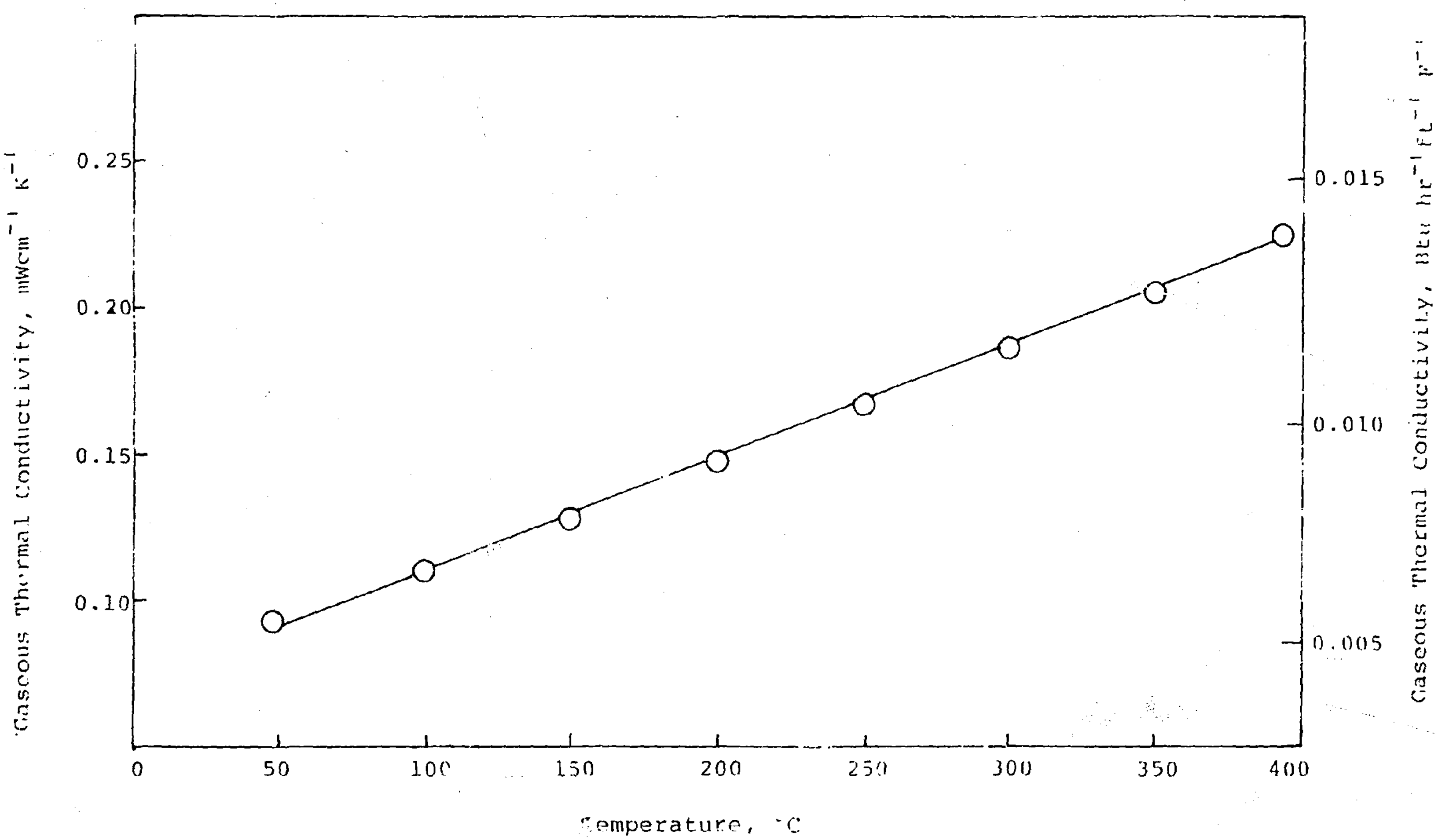


Figure IB-7. Thermal Conductivity of Trichlorosilane

II. CHEMICAL ENGINEERING ANALYSES (TASK 2)

A. SILANE PROCESS (UNION CARBIDE)

The completion of the preliminary process design of the Union Carbide Silane Process as characterized by the original flowsheet received was a major accomplishment this reporting period. Since the amount of recycle required for this process is large, the regular surge tankage requirements required for recycle material can effect the plant investment significantly. Therefore, two cases for the preliminary process design were considered for the original flowsheet:

Case A - Regular Process Storage

Case B - Minimum Process Storage

In additional activities for the silane process, Union Carbide engineering-research personnel revised their original flowsheet. The revised process (Case C) involves a more optimum arrangement of major process equipment, raw material requirements and operations conditions:

Case C - Revised Process

Initial review of the revised flowsheet suggests favorable improvements over the original scheme.

Each of these Cases (A, B and C) are discussed separately in the following sections.

1. CASE A - Regular Process Storage

A summation of the salient features of Case A is presented in a tabular format as follows:

CASE A

Process......................................Silane (Union Carbide)
Plant Size...................................1270 MT/year of Silane
Process Flowsheet............................Original received from Union Carb-
Process Chemistry and Equilibrium...............From Union Carbide
Intermediate Product Storage Considerations......Regular
Major Process Equipment..........................76 pieces of process equipment

The status of preliminary process design activities involving Case A, including progress since the last reporting period, is given below for key items:

	Prior	Current
. Process Flow Diagram	100%	100%
. Material Balance	100%	100%
. Energy Balance	100%	100%
. Property Data	85%	100%
. Equipment Design	85%	100%
. Production Labor	75%	100%

The detailed status sheet is shown in Table IIA-1.0A, and is representative of the various subitems that make up the preliminary design activity. The flowsheet used for the design is shown in Figure IIA-1.0A. This flowsheet was received from Union Carbide.

The results from the preliminary process design are presented in a tabular format similar to previous design results for alternate processes to produce silicon. Note that in this process results are per pound of silane versus other processes represented as per kilogram of silicon. The silane plant size assumes a 90% conversion of silane to silicon.

The guide to the tables for Case A is given below:

. Base Case Conditions..................Table IIA-1.1A
. Reaction Chemistry....................Table IIA-1.2A
. Redistribution Equilibrium............Figure IIA-1.1A
. Raw Material Requirement..............Table IIA-1.3A
. Utility Requirements..................Table IA-1.4A
. Major Process Equipment...............Table IIA-1.5A
. Production Labor Requirements.........Table IIA-1.6A

CASE A

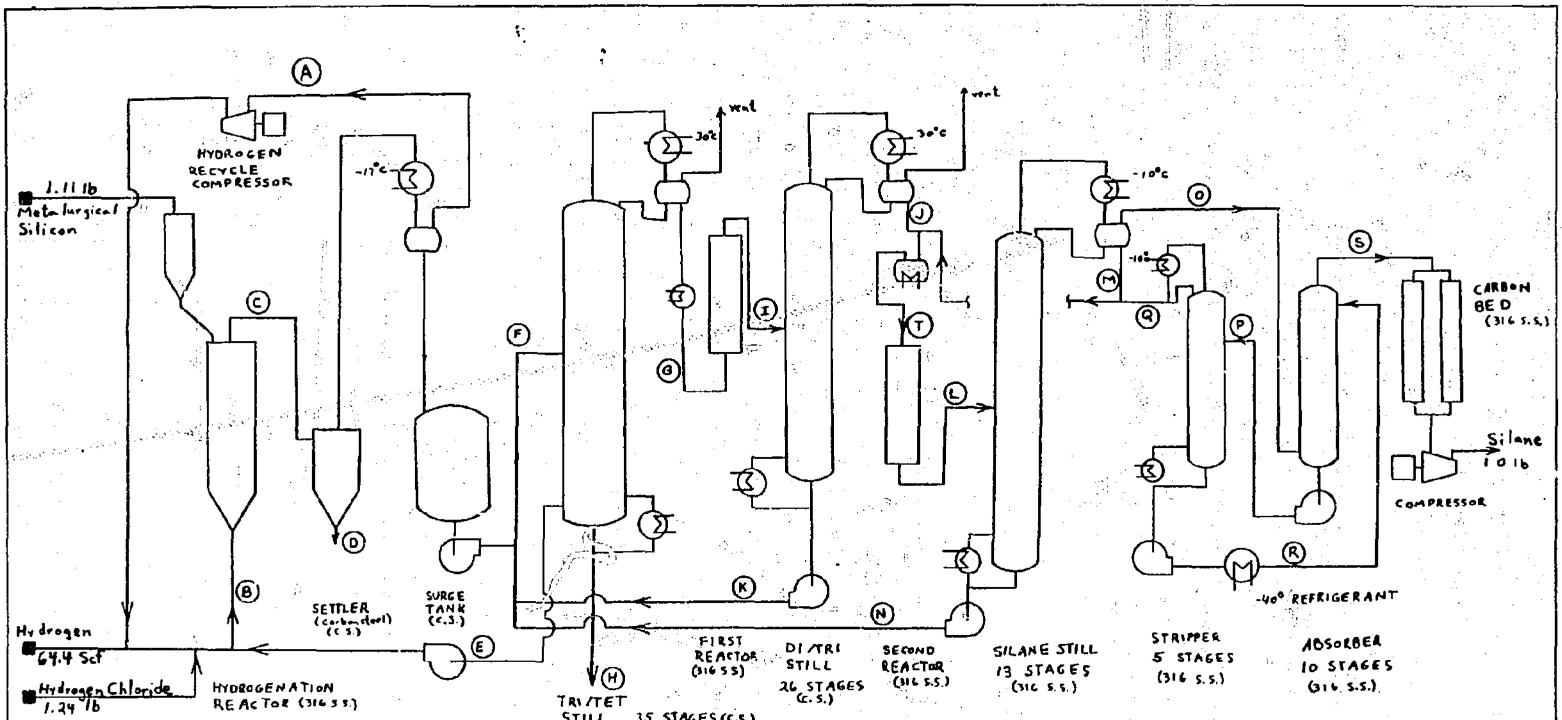
TABLE IIA-1.0A CHEMICAL ENGINEERING ANALYSES: PRELIMINARY PROCESS DESIGN ACTIVITIES FOR SILANE PROCESS - CASE A (UNION CARBIDE)

Prel. Process Design Activity	Status
1. Specify Base Case Conditions	●
1. Plant Size	●
2. Product Specifics	●
3. Additional Conditions	●
2. Define Reaction Chemistry	●
1. Reactants, Products	●
2. Equilibrium	●
3. Process Flow Diagram	●
1. Flow Sequence, Unit Operations	●
2. Process Conditions (T, P, etc.)	●
3. Environmental	●
4. Company Interaction (Technology Exchange)	●
4. Material Balance Calculations	●
1. Raw Materials	●
2. Products	●
3. By-Products	●
5. Energy Balance Calculations	●
1. Heating	●
2. Cooling	●
3. Additional	●
6. Property Data	●
1. Physical	●
2. Thermodynamic	●
3. Additional	●

Prel. Process Design Activity	Status
7. Equipment Design Calculations	●
1. Storage Vessels	●
2. Unit Operations Equipment	●
3. Process Data (P, T, rate, etc.)	●
4. Additional	●
8. List of Major Process Equipment	●
1. Size	●
2. Type	●
3. Materials of Construction	●
8a. Major Technical Factors (Potential Problem Areas)	● ●
1. Materials Compatibility	●
2. Process Conditions Limitations	●
3. Additional	●
9. Production Labor Requirements	●
1. Process Technology	●
2. Production Volume	●
10. Forward for Economic Analysis	●

O Plan
◐ In Progress
● Complete

CASE A



Ref. Point	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	
Temp., °C	-17	550	550	110	124		80	124	80	25	85	80	-5	85	-5	-5	-5	-40	-40	80			
Press., psig	50	50	45	45	50	50	50	50	50	50	50	45	45	50	45	35	35	45	45	45			
Flow, lb/hr.	6.6	139	140	.16	131	268	136	1.3	136	10.2	125	20.7	9.3	9.0	2.3	2.85	1.3	1.6	1.03	20.7			
H_2, mole %	94.7	49.4	45.9	-	-	-	-	-	-								-	-	-				
$SiCl_4$, mole %	3.6	49.2	43.3	80	99.0	42.8	-	99.5	11.5		12.8					35.4	-	97.5	.2				
$HSiCl_3$, mole %	1.7	.3	10.8	20	1.0	57.2	99.0	.5	78	1	87.2	29	4.4	91.2		-	-		-	2.5			
H_2SiCl_2, mole %	-	-	-		-	-	1.0		10	99		35	67.8	8.3	15.1	27.8	44.3		-	80.2			
H_3SiCl, mole %	-	-	-		-	-			0.5			16	18.4		16.8	31	47.6	2.5	.7	12.2			
SiH_4, mole %	-	-	-		-	-			-			20	9.4		67.6	5.1	8.1		99.1	5.1			
HCl, mole %		1.1	-		-	-																	

REF. DWG.

CHEMICALS AND PLASTICS
UNION CARBIDE CORPORATION
SISTERSVILLE, WEST VIRGINIA

Silicon to Silane Process Flowsheet

DR: WCB | CH | AP | SCALE | DATE: 12-10-76 | C-4334-2

Figure IIA-1.0A Process Flow Sheet for Silane Process-Case A
(Revised, Provided by Union Carbide)

CASE A

TABLE IIA-1.1A

BASE CASE CONDITIONS FOR SILANE PROCESS - CASE A (Union Carbide)

1. Plant Size
   - Allow for 10% losses of silane in production of silicon
   - 1270 metric tons/year of silane
   - Solar cell grade silicon

2. Hydrogenation Reaction
   - Metallurgical grade silicon, hydrogen, to produce trichlorosilane (TCS) make-up hydrogen chloride used and recycle silicon tetrachloride (TET)
   - Copper catalyzed
   - Fluidized bed
   - 550°C, 50 PSIG
   - 15.8% conversion of $SiCl_4$ (Union Carbide flowsheet)
3. TCS Redistribution Reaction
   - TCS from hydrogenation produces dichlorosilane (DCS)
   - Catalytic redistribution of TCS with tertiary amine ion exchange resin.
   - Liquid phase 50 PSIG, 80°C.
   - Conversion a function of inlet concentration per Figure IIA-2 (Union Carbide equilibrium)
   - Conversion from pure TCS feed is about 10% to DCS (example)
4. DCS Redistribution Reaction
   - DCS produces SiH_4 (silane)
   - Catalytic redistribution of DCS with tertiary amine ion exchange resin.
   - Gas phase 60-80°C
   - Conversion a function of inlet concentration per Figure IIA-1.1 (Union Carbide equilibrium)
   - Conversion from pure DCS feed is about 14% to Silane (example)
5. Recycles
   - Unreacted chlorosilanes separated by distillation and recycled

6. Silane Purification
   - Chlorosilanes removed by absorption in -40°C $SiCl_4$ (Tet)
   - Trace contaminants removed by carbon adsorption

7. Operating Ratio
   - Approximately 90% utilization
   - Approximately 7880 hour/year production

8. Storage Considerations
   - Feed materials (two week supply)
   - Product (two week supply)
   - Process (several days)

CASE A

TABLE IIA-1.2A

REACTION CHEMISTRY FOR SILANE PROCESS - CASE A (UNION CARBIDE)

1. Hydrogenation Reaction

$$3\ SiCl_4 + Si + 2H_2 \rightleftarrows 4SiHCl_3$$

2. Trichlorosilane Redistribution Reaction

$$2\ SiHCl_3 \rightleftarrows SiH_2Cl_2 + SiCl_4$$

3. Dichlorosilane Redistribution Reaction

$$3\ SiH_2Cl_2 \xrightarrow{\text{Distillation}} 2SiHCl_3 + SiH_4$$

Note

1. Reaction 1 Product contains H_2, $SiCl_4$, $SiHCl_3$, SiH_2Cl_2 (trace), other trace chlorides

2. Reaction 2 Product contains $SiHCl_3$, $SiCl_4$, SiH_2Cl_2, SiH_3Cl

3. Reaction 3 Product contains SiH_2Cl_2, $SiHCl_3$, $SiCl_4$, SiH_3Cl, SiH_4

CASE A

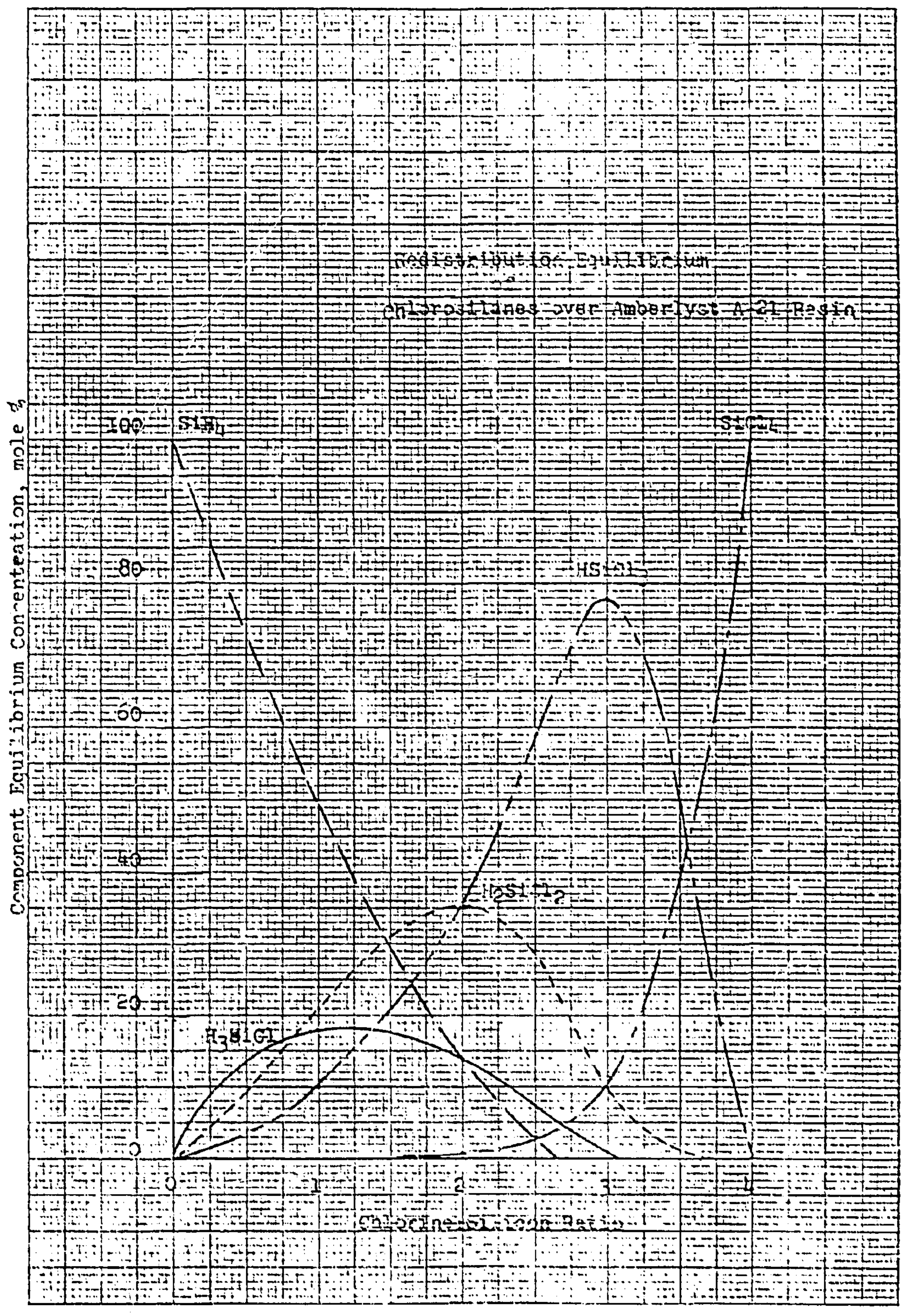


Figure IIA-1.1A Redistribution Equilibrium For Silane Process - CASE A
(Provided by Union Carbide)

CASE A

TABLE IIA-1.3A

RAW MATERIAL REQUIREMENTS FOR SILANE PROCESS - CASE A (UNION CARBIDE)

	Raw Material	Requirement lb/lb of Silane
1.	Anhydrous HCl	1.239
2.	Hydrogen	.362
3.	Caustic (50%)	2.448
4.	M.G. Silicon	1.11

CASE A

TABLE IIA-1.4 A

UTILITY REQUIREMENTS FOR SILANE PROCESS - CASE A (UNION CARBIDE)

Utility/Function		Requirements/lb of Silane Product
1. Electricty		.253 KW-HR
1. All pump and compressor motors (24)	(.253)	
2. Steam 250 Psia		190.34 lbs
1. #1 Distillation Column Preheater	(6.96)	
2. #1 Distillation Column Reboiler	(81.18)	
3. #2 Distillation Column Reboiler	(91.77)	
4. #2 Redistribution Reactor Preheater	(3.0)	
5. #3 Distillation Column Preheater	(3.62)	
6. #3 Distillation Column Reboiler	(3.29)	
7. #4 Distillation Column Reboiler	(.41)	
8. Waste Treatment	(.11)	
3. Cooling Water (10-120°F)		168.12 gallons
1. #1 Distillation Column Condenser	(146.12)	
2. #2 Distillation Column Condenser	(22.09)	
4. Process Water (90°F)		8.22 gallons
1. Waste Treatment	(8.22)	
5. Refrigerant (23°F)		27.1 BTU
1. #4 Distillation Column Feed Tank	(27.1)	
6. Refrigerant (5°F)		79.1 BTU
1. #3 Distillation Column Overhead Receiver	(79.1)	
7. Refrigerant (-7°F)		26.4 BTU
1. #4 Distillation Column Overhead Receiver	(26.4)	
8. Refrigerant (-20°F)		2303.2 BTU
1. #3 Distillation Column Condenser	(2058.0)	
2. #4 Distillation Column Condenser	(245.2)	

CASE A

TABLE IIA-1.4A (Continued)

9.	Refrigerant (-30°F)		30788.0 BTU
	1. TCS Reactor Recycle Gas Condenser	(30788.0)	
10.	Refrigerant (-40°F)		280.6 BTU
	1. #2 Redistribution Reactor Condensate Receiver	(192.2)	
	2. Silane Product Storage	(88.4)	
11.	Refrigerant (-50°F)		3503.2 BTU
	1. #2 Redistribution Reactor Gas Condensor	(2986.0)	
	2. Product Silane Condenser	(137.9)	
	3. Absorbent Cooler	(379.3)	
12.	High Temperature Heat Exchange Fluid		3.324×10^4 BTU
	1. TCS Reactor Recycle Gas Heater	(6.591×10^3)	
	2. HCl Vaporizer	(4.466×10^2)	
	3. Tet Vaporizer	(2.464×10^4)	
	4. Heat Nitrogen to Regnerate Char. Adsorbers	(70.95)	
	5. TCS Reactor	(1.491×10^3)	
13.	Nitrogen		5.54 SCF
	1. Regenerate Charcoal Adsorbers	(5.54)	

CASE A

TABLE IIA-1.5 A

LIST OF MAJOR PROCESS
EQUIPMENT FOR SILANE PROCESS - CASE A (UNION CARBIDE)

	Type	Function	Duty	Size	Materials of Construction
1.	(T1) M.G. Silicon Storage Hopper	Raw Material Storage	2 weeks storage	1.363×10^4 gallons	CS
2.	(T2) Hydrogen Storage Tank	Raw Material Storage	8 hours backup for pipeline failure	9.161×10^4 gallons 250 PSIA (spherical)	CS
3.	(T3) Liquid HCl Storage TAnk	Raw Material Storage	2 weeks storage	1.612×10^4 gallons 250 PSIA, $-50^{\circ}F$ (spherical)	Nickel Steel
4.	(T4) Recycle TET Storage	For TCS Reactor Feed	2 days storage	1.985×10^5 gallons 65 PSIA	CS
5.	(T5) TCS Reactor Off-Gas Flash Tank	Phase Separation		1 ft. diameter by 4 ft. long, 65 PSIA, $0^{\circ}F$ 65 PSIA	CS
6.	(T6) TCS/TET Storage	Feed Distillaton Column #1	2 Days hold-up	1.966×10^5 gallons 65 PISA	CS
7.	(T7) #1 Distillation Column Condensate Accumulator	Reflux feed; column Control	20 minutes hold-up	4.88×10^3 gallons 65 PSIA	CS
8.	(T8) #1 Redistribution Reactor Feed Tank	Hold-up and feed Reactor	2 days hold-up	2.266×10^5 gallons 65 PSIA	CS
9.	(T9) #1 Redistribution Reactor Product Tank	Hold-up and feed #2 Distillation Column	2 days hold-up	2.21×10^5 gallons 65 PSIA	CS

CASE A

TABLE IIA-1.5A (continued)

10.	(T10)	#2 Distillation Column Condensate Accumulator	Reflux feed; column Control	20 minutes hold-up	746 gallons 65 PSIA	SS
11.	(T11)	#2 Redistribution Reactor Feed Tank	Hold-up and feed Reactor	2 days hold-up	1.891×10^4 gallons 65 PSIA	SS
12.	(T12)	#2 Redistribution Reactor Product Tank	Hold-up and feed #3 Distillation Column	2 days hold-up 6.8×10^4 BTU/hr	3.46×10^4 gallons $-40^{\circ}F$, 60 PSIA	SS
13.	(T13)	#3 Distillation Column Condensate Accumulator	Reflux feed; phase Separation; column control	20 minutes hold-up	194 gallons $5^{\circ}F$, 60 PSIA	SS
14.	(T14)	#3 Distillation Column Condensate Tank	Hold-up and recycle feed to #2 Redistribution Reactor	2 days hold-up 2.81×10^4 BTU/hr	1.7×10^4 gallons 60 PSIA, $5^{\circ}F$	SS
15.	(T15)	#4 Distillation Column Feed Tank	Surge between absorber and distillation	2 days hold-up 9.63×10^3 BTU/hr	4.69×10^3 gallons 60 PSIA	SS
16.	(T16)	#4 Distillation Column Condensate Accumulator	Reflux feed; column control	20 minutes hold-up	18 gallons 50 PSIA, $-7^{\circ}F$	SS
17.	(T17)	#4 Distillation Column Condensate Tank	Hold-up and recycle to #2 Redistribution Reactor	2 days hold-up 9.4×10^3 BTU/hr	2.55×10^3 gallons 50 PSIA, $-7^{\circ}F$	SS
18.	(T18)	Waste Tank	Collect waste for Treatment and disposal	2 week storage	1.378×10^4 gallons 65 PSIA	CS
19.	(T19)	Absorber Feed Tank	Feed TET to absorber	2 days storage	2.44×10^3 gallons 50 PSIA	SS
20.	(T20)	Silane Storage	Final Product storage	1 week storage 3.14×10^4 BTU/hr	1.522×10^4 gallons $-40^{\circ}F$, 250 PSIA	SS

CASE A

TABLE IIA-1.5A(continued)

21.	(T21)	Caustic Storage	Raw Material Storage	2 weeks storage	2.304×10^4 gallons	SS
22.	(H1)	TCS Reactor Recycle Gas Heater	Heat Recycle gas and Hydrogen to 550°C	2.342×10^6 BTU/hr	752 ft^2 65 PSIA	CS
23.	(H2)	HCl Vaporizer	Heat Reactant to 550°C	1.587×10^5 BTU/hr	34 ft^2 65 PSIA	CS
24.	(H3)	TET Vaporizer	Heat Reactant to 550°C	8.755×10^6 BTU/hr	2381 ft^2 65 PSIA	CS
25.	(H4)	TCS Reactor Recycle Condenser	Phase separation; Recycle hydrogen	1.094×10^7 BTU/hr	1882 ft^2 65 PSIA	CS/SS
26.	(H5)	#1 Distillation Column Preheater	Preheat distillation feed to bubble point	2.044×10^6 BTU/hr	164 ft^2 250 PSIA	CS
27.	(H6)	#1 Distillation Column Condenser	Provide Reflux to Column	1.296×10^7 BTU/hr	3189 ft^2 65 PSIA	CS
28.	(H7)	#1 Distillation Column Reboiler	Provide vapor to Column	2.382×10^7 BTU/hr	2818 ft^2 250 PSIA	CS
29.	(H8)	#2 Distillation Column Condenser	Provide Reflux to column	1.96×10^6 BTU/hr	956 ft^2 65 PSIA	CS/SS
30.	(H9)	#2 Distillation Column Reboiler	Provide Vapor to Column	2.693×10^7 BTU/hr	2514 ft^2 250 PSIA	CS
31.	(H10)	#2 Redistribution Reactor Feed Vaporizer	Vaporize Reactants for Reactor	8.81×10^5 BTU/hr	78 ft^2 250 PSIA	CS/SS
32.	(H11)	#2 Redistribution Reactor Product Condenser	Condense Vapor for hold-up storage	1.06×10^6 BTU/hr	306 ft^2 60 PSIA	CS/SS
33.	(H12)	#3 Distillation Column Preheater	Vaporize and preheat feed to column	1.06×10^6 BTU/hr	66 ft^2 250 PSIA	CS/SS

CASE A

TABLE IIA-1.5A(continued)

34.	(H13)	#3 Distillation Column Condenser	Provide Column Reflux (Partial Condenser)	7.312×10^5 BTU/hr	593 ft^2 60 PSIA	CS/SS
35.	(H14)	#3 Distillation Column Reboiler	Provide Vapor to Column	9.64×10^5 BTU/hr	84 ft^2 250 PSIA	CS/SS
36.	(H15)	Silane Condenser	Condenser Final Product for storage	4.9×10^4 BTU/hr	53 ft^2 250 PSIA	CS/SS
37.	(H16)	#4 Distillation Column Condenser	Provide Reflux	8.71×10^4 BTU/hr	84 ft^2 50 PSIA	CS/SS
38.	(H17)	#4 Distillation Column Reboiler	Provide Vapor to Column	1.2×10^5 BTU/hr	13 ft^2 250 PSIA	CS/SS
39.	(H18)	Absorber Precooler	Cool TET for absorption column	1.35×10^5 BTU/hr	35 ft^2 60 PSIA	CS/SS
40.	(H19)	Nitrogen Heater	Heat Nitrogen to regenerate Charcoal Adsorbers	2.52×10^4 BTU/hr	14.1 ft^2	CS
41.	(P1)	TCS Reactor Off Gas Recycle Compressor	Circulate Recycle Gas to Reactor	1.36×10^3 SCFM	26.5 Horsepower 75 PSIA Discharge	CS*
42.	(P2)	#1 Distillation Column Feed Pump	Feed Column	136.5 gpm	106 PSI; 14.5 BHP	CS*
43.	(P3)	#1 Distillation Column Overheads Pump	Provide Reflux and remove overhead product	244 gpm	92.3 PSI; 22.5 BHP	CS*
44.	(P4)	#1 Distillation Column Bottoms Pump	Remove Bottoms Product to TET storage tank	69 gpm	106 PSI; 7.3 BHP	CS*

CASE A

TABLE IIA-1.5A (continued)

45.	(P5)	Process Water Feed Pump	Feed Process Water to Waste Treatment	48.6 gpm	82.5 PSI; 4 BHP	CS*
46.	(P6)	Caustic Feed Pump	Feed Raw Material to waste treatment	1 gpm	118 PSI; ¼ BHP	SS
47.	(P7)	#1 Redistribution Reactor Feed Pump	Feed TCS to Reactor	79 gpm	106 PSI, 8.4 BHP	SS
48.	(P8)	#2 Distillation Column Feed Pump	Feed TCS/DCS still	76.6 gpm	92.3 PSI; 7.1 BHP	SS
49.	(P9)	#2 Distillation Column Overheads Pump	Provide Reflux and Remove Overhead Product	37.3 gpm	92.3 PSI; 3.4 BHP	SS
50.	(P10)	#2 Distillation Column Bottoms Pump	Remove Bottoms Product to TCS/TET storage tank	66.7 gpm	106.3 PSI; 7.1 BPH	SS
51.	(P11)	#2 Redistribution Reactor Feed Pump	Feed DCS to Reactor	13.4 gpm	130 PSI; 1.7 BHP	SS
52.	(P12)	#3 Distillation Column Feed Pump	Feed Silane Still	12 gpm	87.3 PSI; 1 BHP	SS
53.	(P13)	#3 Distillation Column Overhead Pump	Provide Reflux; Remove Overhead Product	9.7 gpm	87.3 PSI; 1 BHP	SS
54.	(P14)	#3 Distillation Column Bottoms Pump	Remove Bottoms Product to TCS/TET Tank	5.2 gpm	106.3 PSI; ½ BHP	SS
55.	(P15)	#4 Distillation Feed Pump	Feed TET Stripper	1.6 gpm	77.3 PSI, ¼ BHP	SS

* Includes incremental higher cost for special purity requirements.

CASE A

TABLE IIA-1.5A (continued)

56.	(P16)	#4 Distillation Column Overhead Pump	Provide Reflux, Remove Overhead Product	1 gpm	77.3 PSI; ¼ BHP	SS
57.	(P17)	#4 Distillation Column Bottoms Pump	Remove Bottoms Product to Absorber Feed Tank	1 gpm	91.3 PSI; ¼ BHP	SS
58.	(P18)	#4 Distillation Condensate Re-cycle Pump	Recycle Condensate back to #2 Redistri-bution Reactor	1 gpm	106.3 PSI; ¼ BHP	SS
59.	(P19)	Silane Product Compressor	Liquefy Silane for Storage	66 SCFM	250 PSIA Discharge 6.5 HP	SS
60.	(P20)	Waste Feed Pump	Distillation Wastes to Waste Treatment	1 gpm	76.3 PSI; ¼BHP	CS
61.	(P21)	TCS Reactor Feed Pump	Feed TET to Reactor	69 gpm	92.3 PSI; 6.4 BHP	CS*
62.	(P22)	#3 Distillation Condensate Re-cycle Pump	Recycle Condensate back to #2 Redistribu-tion reactor	5.9 gpm	92.3 PSI; ½ BHP	SS
63.	(P23)	Waste Collection Pump	Distillation Wastes to Waste Tank	1 gpm	87.3 PSI; ¼ BHP	CS
64.	(P24)	Absorber Feed Pump	Feed Cold TET to Absorption Column	1 gpm	87.3 PSI; ¼ BHP	SS
65.	(C1)	#1 Distillation Column	Separate TET from TCS	95,220 lb/hr of feed	7.56 ft. diameter 100 ft. tall, 50 trays	CS

CASE A

TABLE IIA-1.5A (continued)

66.	(C2)	#2 Distillation Column	Separate TCS from DCS	48, 321 lb/hr of feed	10.6 ft. Diameter 136 ft. tall, 68 trays	CS
67.	(C3)	#3 Distillation Column	Separate Silane from other Chlorosilanes	7344 lb/hr of feed	2.01 ft. Diameter 29 ft. tall, 29 trays	SS
68.	(C4)	#4 Distillation Column	Strip TET for use in absorber	1007.7 lb/hr of feed	1.04 ft. Diameter 28.5 ft. tall, 38 trays	SS
69.	(C5)	Silane Absorber	Absorb Chlorosilane from Silane	819.3 lb/hr of vapor feed	0.823 ft. Diameter 12 ft. tall, 16 trays	SS
70.	(C6)	Charcoal Adsorber	Activated Carbon Adsorbtion of Silane to remove Trace Chlorosilane	366 lb/hr of vapor feed	1 ft. Diameter 7 ft. tall (2), 623 lbs of carbon	SS
71.	(R1)	TCS Fluidized Bed Reactor	Produces TCS from TET M.G.Silicon, and H_2		6.26 ft. in diameter 26.5 ft. tall, 481 tubes 1", 16' long	SS
72.	(R2)	#1 Redistribution Reactor	Redistribute TCS to DCS		2' Diameter by 15 ft. tall 1042 lbs catalyst	SS
73.	(R3)	#2 Redistribution Reactor	Redistribute DCS to Silane		2.34' Diameter by 35 ft.tall 1667.2 lbs catalyst	SS
74.	(A1)	Fines Separator	Remove Silicon Fines carried over with TCS Reactor Off-gas		Standard design 30" Diameter	SS
75.	(A2)	Waste Treatment	Discharge innocuous effluent		1 column for absorption + 1 heat exchanger to vaporize feed	SS
76.	(A3)	Hydrogen Flare	Dispose of Hydrogen from Waste Treatment		30 ft. stack 6" Diameter	CS

CASE A

TABLE IIA-1.6A

PRODUCTION LABOR REQUIREMENTS FOR
SILANE PROCESS-CASE A (UNION CARBIDE)

	Unit Operation	Type	Skilled Labor, Man Hours Per Day	Per lb. Silane	Semiskilled Labor Per Day	Per lb. Silane
1.	TCS Production	B	65	.0085		
2.	Hydrogen Recycle	C	18	.0023		
3.	Raw Material Vaporization	C	50	.0065		
4.	TCS Condensation	C	50	.0065		
5.	TCS/TET Separation	C	62	.0081		
6.	#1 Redistribution Reactor	C	49	.0064		
7.	DCS/TCS Separation	C	52	.0068		
8.	#2 Redistibuiton Reactor	C	32	.0042		
9.	Silane Distillation	C	32	.0042		
10.	Silane Absorption	C	28	.0036		
11.	Silane Purification (adsorption)	A	36	.0047		
12.	Silane compression	B	23	.003		
13.	Silane Condensation	B	23	.003		
14.	Materials Handling	A			48	.0063
15.	Waste Treatment	B	60	.0078		
16.	Silicon Fines Separation	A	15	.002		
	TOTAL		595	.0776	48	.0063

NOTES:

1. A Batch Process of Multiple Small Units
   B Average Process
   c Automated Process

2. Man hours/day Unit from Figure 4-6, Peters and Timmerhaus (7).

2. CASE B - Minimum Process Storage

A summation of the important features of CASE B is presented in the following table:

CASE B

Process..	Silane (Union Carbide)
Plant Size.....................................	1270 MT/year of Silane
Process Flow Sheet.............................	Original Received from Union Carbide
Process Chemistry & Equilibrium................	From Union Carbide
Intermediate Product Storage Considerations....	Minimum
Major Process Equipment........................	58 pieces of Process Equipment

The status of preliminary process design activities involving CASE B, including progress since the last reporting period, is given below for key items:

	Prior	Current
. Process Flow Diagram	0%	100%
. Material Balance	100%	100%
. Energy Balance	0%	100%
. Property Data	85%	100%
. Equipment Design	0%	100%
. Production Labor	0%	100%

The detailed status sheet is shown in Table IIA-1.0B, and is representative of all the activities that compose the preliminary process design. The flowsheet received from Union Carbide, upon which the design is based, is shown in Figure IIA-1.0B.

The results from the preliminary process design (CASE B) are summarized in a tabular format parallel to those representing CASE A. These tables are represented by the following guide to enable the reader to quickly locate items of interest.

- Base Case Conditions............................Table IIA-1.1B
- Reaction Chemistry..............................Table IIA-1.2B
- Redistribution Equilibrium......................Table IIA-1.3B
- Raw Material Requirement........................Table IIA-1.4B
- Utility Requirements............................Table IIA-1.4B
- Major Process Equipment.........................Table IIA-1.5B
- Production Labor Requirements...................Table IIA-1.6B

CASE B

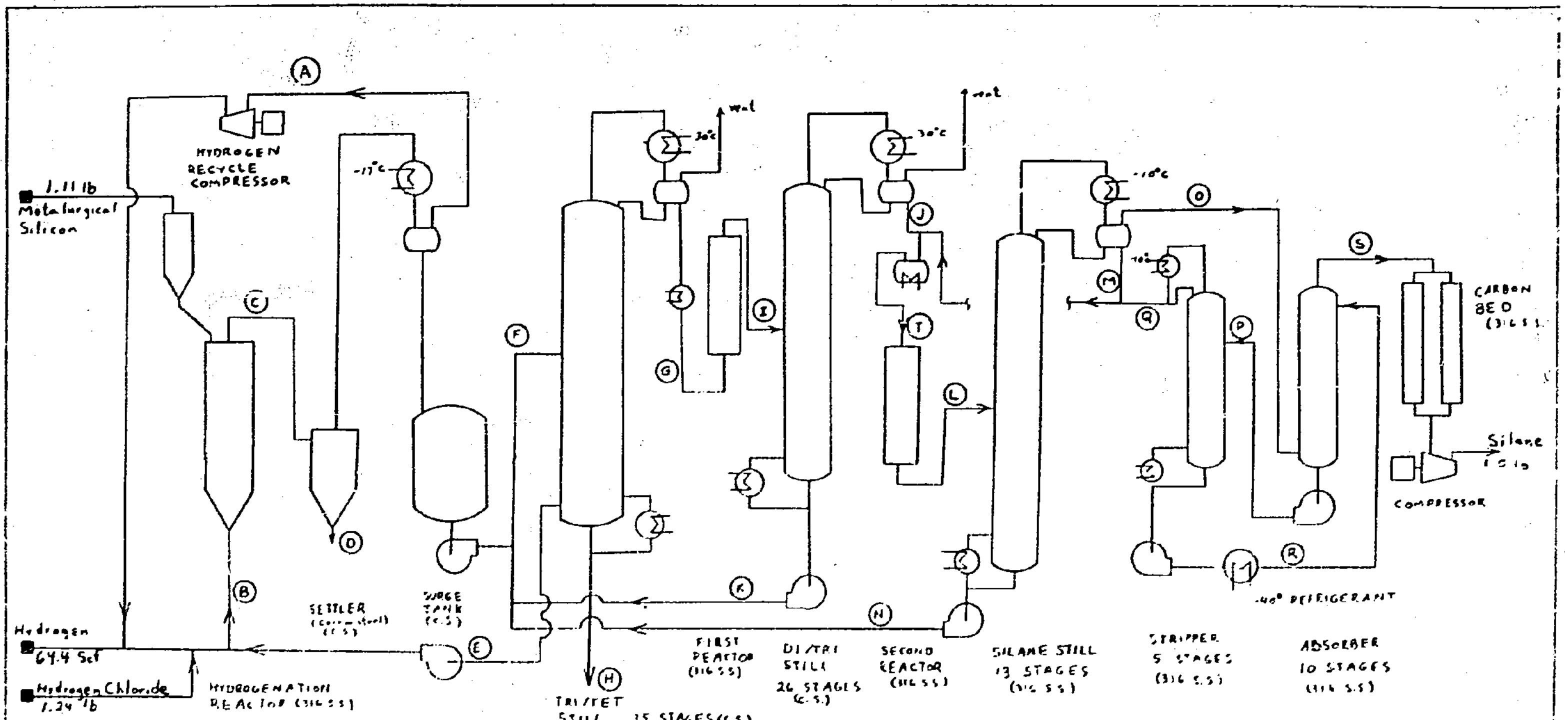
TABLE IIA-1.0B CHEMICAL ENGINEERING ANALYSES: PRELIMINARY PROCESS DESIGN ACTIVITIES FOR SILANE PROCESS-CASE B (UNION CARBIDE)

Prel. Process Design Activity	Status
1. Specify Base Case Conditions	●
1. Plant Size	●
2. Product Specifics	●
3. Additional Conditions	●
2. Define Reaction Chemistry	●
1. Reactants, Products	●
2. Equilibrium	●
3. Process Flow Diagram	●
1. Flow Sequence, Unit Operations	●
2. Process Conditions (T, P, etc.)	●
3. Environmental	●
4. Company Interaction (Technology Exchange)	●
4. Material Balance Calculations	●
1. Raw Materials	●
2. Products	●
3. By-Products	●
5. Energy Balance Calculations	●
1. Heating	●
2. Cooling	●
3. Additional	●
6. Property Data	●
1. Physical	●
2. Thermodynamic	●
3. Additional	●

Prel. Process Design Activity	Status
7. Equipment Design Calculations	●
1. Storage Vessels	●
2. Unit Operations Equipment	●
3. Process Data (P, T, rate, etc.)	●
4. Additional	●
8. List of Major Process Equipment	●
1. Size	●
2. Type	●
3. Materials of Construction	●
8a. Major Technical Factors (Potential Problem Areas)	●
	●
1. Materials Compatibility	●
2. Process Conditions Limitations	●
3. Additional	●
9. Production Labor Requirements	●
1. Process Technology	●
2. Production Volume	●
10. Forward for Economic Analysis	●

O Plan
◐ In Progress
● Complete

CASE B



Ref Point	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V
Temp., °C	-17	550	550	110	124		80	124	80	25	7.5	80	-5	85	-5	-5	-5	-40	-40	80		
Press. psig	50	50	45	45	50	50	50	50	50	50	50	45	45	50	45	35	35	45	45	45		
Flow, lb/hr	6.6	139	140	.16	131	263	136	1.3	136	10.2	125	207	9.3	9.0	2.3	2.25	1.3	1.6	1.03	20.7		
H_2, mole %	94.7	49.4	45.9	-	-	-	-	-	-									-	-			
$SiCl_4$, mole %	3.6	47.2	43.3	80	99.0	42.8	-	99.5	11.5		12.8					35.4	-	97.5	.2			
$HSiCl_3$, mole %	1.7	.3	10.8	20	1.0	57.2	99.0	.5	78	1	87.2	29	4.4	91.2		-	-		-	2.5		
H_2SiCl_2, mole %	-	-	-		-	-	1.0		10	99		35	67.8	8.3	15.1	27.8	44.3		-	80.2		
H_3SiCl, mole %	-	-	-		-	-			0.5			16	18.4		16.8	31	47.6	2.5	.7	12.2		
SiH_4, mole %	-	-	-		-	-			-			20	9.4		67.6	5.1	8.1		99.1	5.1		
HCl, mole %		1.1	-		-	-																

CHEMICALS AND PLASTICS
UNION CARBIDE CORPORATION
SISTERSVILLE, WEST VIRGINIA

Silicon to Silane Process Flowsheet

C-4334-2

Figure IIA-1.OB Process Flow Sheet for Silane Process -CASE B (Revised, Provided by Union Carbide)

CASE B

TABLE IIA-1.1B

BASE CASE CONDITIONS FOR SILANE PROCESS-CASE B (Union Carbide)

1. Plant Size
   - Allow for 10% losses of silane in production of silicon
   - 1270 metric tons/year of silane
   - Solar cell grade silicon

2. Hydrogenation Reaction
   - Metallurgical grade silicon, hydrogen, to produce trichlorosilane (TCS) make-up hydrogen chloride used and recycle silicon tetrachloride (TET)
   - Copper catalyzed
   - Fluidized bed
   - 550°C, 50 PSIG
   - 15.8% conversion of $SiCl_4$ (Union Carbide flowsheet)
3. TCS Redistribution Reaction
   - TCS from hydrogenation produces dichlorosilane (DCS)
   - Catalytic redistribution of TCS with tertiary amine ion exchange resin.
   - Liquid phase 50 PSIG, 80°C.
   - Conversion a function of inlet concentration per Figure IIA-2 (Union Carbide equilibrium)
   - Conversion from pure TCS feed is about 10% to DCS (example)
4. DCS Redistribution Reaction
   - DCS produces SiH_4 (silane)
   - Catalytic redistribution of DCS with tertiary amine ion exchange resin.
   - Gas phase 60-80°C
   - Conversion a function of inlet concentration per Figure IIA-1.1 (Union Carbide equilibrium)
   - Conversion from pure DCS feed is about 14% to Silane (example)
5. Recycles
   - Unreacted chlorosilanes separated by distillation and recycled

6. Silane Purification
   - Chlorosilanes removed by absorption in -40°C $SiCl_4$ (Tet)
   - Trace contaminants removed by carbon adsorption

7. Operating Ratio
   - Approximately 90% utilization
   - Approximately 7880 hour/year production

8. Storage Considerations
   - Feed materials (two week supply)
   - Product (two week supply)
   - Process (several days)

CASE B

TABLE IIA-1.2B

REACTION CHEMISTRY FOR SILANE PROCESS - CASE B (UNION CARBIDE)

1. Hydrogenation Reaction

$$3\ SiCl_4 + Si + 2H_2 \rightleftarrows 4SiHCl_3$$

2. Trichlorosilane Redistribution Reaction

$$2\ SiHCl_3 \rightleftarrows SiH_2Cl_2 + SiCl_4$$

3. Dichlorosilane Redistribution Reaction

$$3\ SiH_2Cl_2 \xrightarrow{\text{Distillation}} 2SiHCl_3 + SiH_4$$

Note

1. Reaction 1 Product contains H_2, $SiCl_4$, $SiHCl_3$, SiH_2Cl_2 (trace), other trace chlorides

2. Reaction 2 Product contains $SiHCl_3$, $SiCl_4$, SiH_2Cl_2, SiH_3Cl

3. Reaction 3 Product contains SiH_2Cl_2, $SiHCl_3$, $SiCl_4$, SiH_3Cl, SiH_4

CASE B

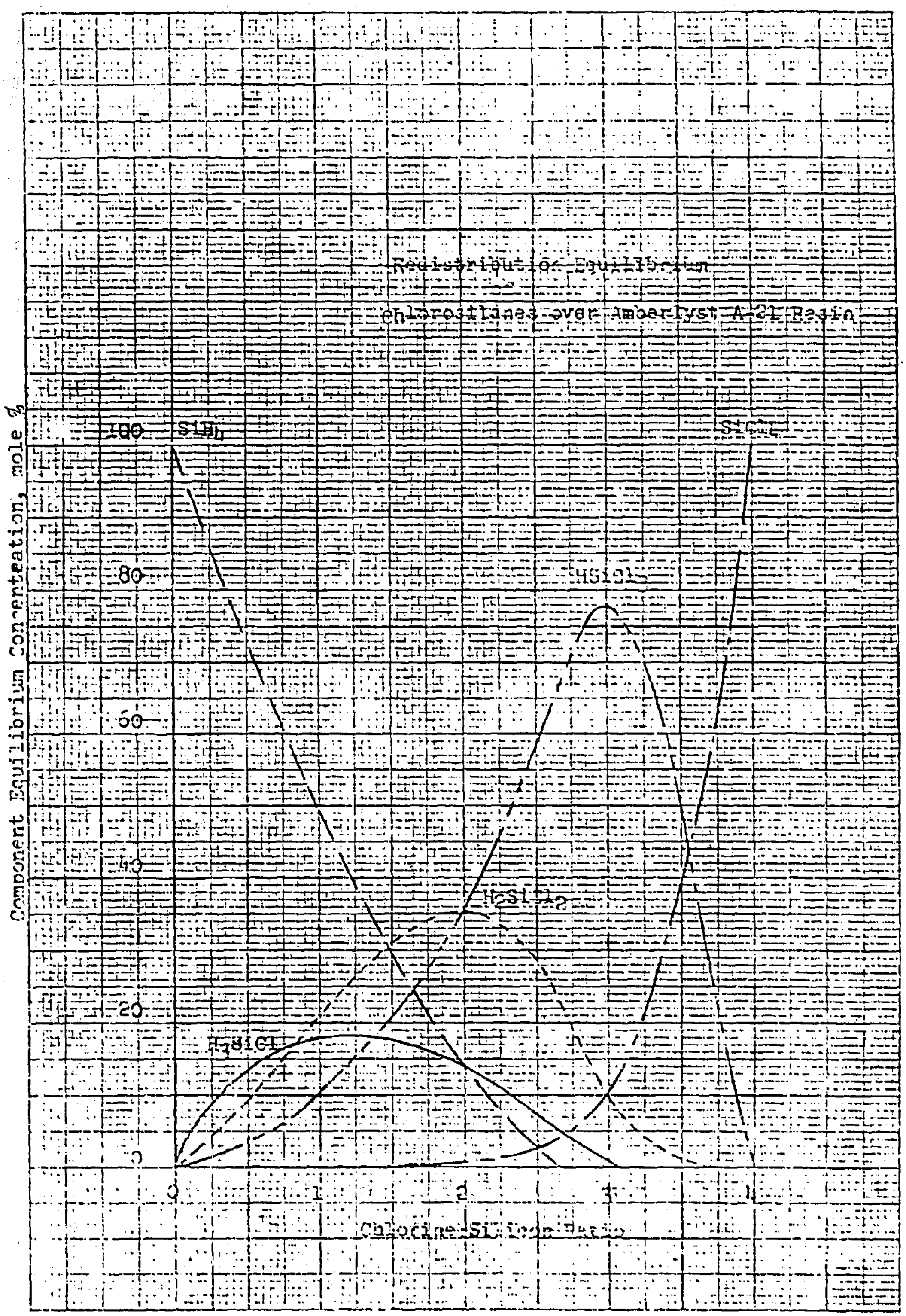


Figure IIA-1.1 Redistribution Equilibrium For Silane Process-CASE B
(Provided by Union Carbide)

CASE B

TABLE IIA-1.3B

RAW MATERIAL REQUIREMENTS FOR SILANE PROCESS-CASE B (Union Carbide)

Raw Material	Requirement lb/lb of Silane
1. Anhydrous HCl	1.239
2. Hydrogen	.362
3. Caustic (50%)	2.448
4. M.G. Silicon	1.11

CASE B

TABLE IIA - 1.4B

UTILITY REQUIREMENTS FOR SILANE PROCESS - CASE B (UNION CARBIDE)

Utility/Function	Requirements/lb of Silane Product	
1. Electricity		.212 KW-HR
1. All pump and Compressor Motors (16)	.212)	
2. Steam 250 Psia		186.72 lbs
1. #1 Distillation Column Preheater	(6.96)	
2. #1 Distillation Column Reboiler	(81.18)	
3. #2 Distillation Column Reboiler	(91.77)	
4. #2 Redistribution Reacotr Preheater	(3.0)	
5. #3 Distillation Column Reboiler	(3.29)	
6. #4 Distillation Column Reboiler	(0.41)	
7. Waste Treatment	(0.11)	
3. Cooling Water (10-120°F)		168.12 gallons
1. #1 Distillation Column Condenser	(146.12)	
2. #2 Distillation Column Condenser	(22.09)	
4. Process Water (90°F)		8.22 gallons
1. Waste Treatment	(8.22)	
5. Refrigerant (-20°F)		2303.2 BTU
1. #3 Distillation Column Condenser	(2058.0)	
2. #4 Distillation Column Condenser	(245.2)	
6. Refrigerant (-30°F)		30788.0 BTU
1. TCS Reactor Recycle Gas Condenser	(30788.0)	
7. Refrigerant (-40°F)		25.26 BTU
1. Silane Product Storage	(25.26)	
8. Refrigerant (-50°F)		517.2 BTU
1. Product Silane Condenser	(137.9)	
2. Absorbent Cooler	(379.3)	
9. High Temperature Heat Exchange Fluid		3.324×10^4 BTU
1. TCS Reactor Recycle Gas Heater	(6.591×10^3)	
2. HCl Vaporizer	(4.46×10^2)	
3. Tet Vaporizer	(2.464×10^4)	
4. Heat Nitrogen to Regenerate Char. Adsorbers	(70.95)	
5. TCS Reactor	(1.491×10^3)	

CASE B

TABLE IIA-1.4B (Continued)

Utility/Function		Requirements/lb of Silane Product
10. Nitrogen		5.54 SCF
1. Regenerate Charcoal Adsorbers	(5.54)	

CASE B

TABLE IIA -1.5B

LIST OF MAJOR PROCESS
EQUIPMENT FOR SILANE PROCESS - CASE B (UNION CARBIDE)

		Type	Function	Duty	Size	Materials of Construction
1.	(T1)	M.G. Silicon Storage Hopper	Raw Material Storage	2 weeks storage	1.363×10^4 gallons	CS
2.	(T2)	Hydrogen Storage Tank	Raw Material Storage	8 hours backup for pipeline failure	9.161×10^4 gallons 250 PSIA (spherical)	CS
3.	(T3)	Liquid HCl Storage Tank	Raw Material Storage	2 weeks storage	1.612×10^4 gallons 250 PSIA, $-50^{\circ}F$ (spherical)	Nickel Steel
4.	(T4)	Recycle TET Storage	For TCS REactor Feed	1 day storage	9.923×10^4 gallons 65 PSIA	CS
5.	(T5)	TCS Reactor Off-Gas Flash Tank	Phase Separation		1 ft. diameter by 4 ft. long, 65 PSIA, $0^{\circ}F$ 65 PSIA	CS
6.	(T6)	TCS/TET Storage	Feed Distillation Column #1	1 day hold-up	1.966×10^5 65 PSIA	CS
7.	(T7)	#1 Distillation Column Condensate Accumulator	Reflux feed; column Control	20 minutes hold-up	4.88×10^3 gallons 65 PSIA	CS
8.	(T8)	#2 Distillation Column Condensate Accumulator	Reflux feed; column Control	20 minutes hold-up	746 gallons 65 PSIA	SS
9.	(T9)	#3 Distillation Column Condensate Accumulator	Reflux feed; phase Separation; column control	20 minutes hold-up	194 gallons $5^{\circ}F$. 60 PSIA	SS

CASE B

TABLE IIA-1.5B (Continued)

10.	(T10)	#4 Distillation Column Condensate Tank	Reflux feed; column control	20 minutes hold-up	18 gallons 50 PSIA, $-7^{\circ}F$	SS
11.	(T11)	Waste Tank	Collect waste for Treatment and disposal	2 week storage	1.378×10^{4} gallons 65 PSIA	CS
12.	(T12)	Silane Storage	Final Product Storage	2 days storage 8.97×10^{3} BTU/Hr	4.349×10^{3} gallons $-40^{\circ}F$, 250 PSIA	SS
13.	(T13)	Caustic Storage	Raw Material Storage	2 weeks storage	2.304×10^{4} gallons	SS
14.	(H1)	TCS Reactor Recycle Gas Heater	Heat Recycle gas and Hydrogen to $550^{\circ}C$	2.342×10^{6} BTU/hr	752 ft^2 65 PSIA	CS
15.	(H2)	HCl Vaporizer	Heat Reactant to $550^{\circ}C$	1.587×10^{5} BTU/hr	34 Ft^2 65 PSIA	CS
16.	(H3)	TET Vaporizer	Heat Reactant to $550^{\circ}C$	8.755×10^{6} BTU/hr	2381 ft^2 65 PSIA	CS
17.	(H4)	TCS Teactor Recycle Condenser	Phase separation; Recycle hydrogen	1.094×10^{7} BTU/hr	1882 ft^2 65 PSIA	CS/SS
18.	(H5)	#1 Distillation Column Preheater	Preheat distillation feed to bubble point	2.044×10^{6} BTU/hr	164 ft^2 250 PSIA	CS
19.	(H6)	#1 Distilllation Column Condenser	Provide Reflux to Column	1.296×10^{7} BTU/hr	3189 ft^2 65 PSIA	CS
20.	(H7)	#1 Distillation Column Reboiler	Provide vapor to Column	2.382×10^{7} BTU/hr	2818 ft^2 250 PSIA	CS

CASE B

TABLE IIA-1.5B (Continued)

21.	(H8)	#2 Distillation Column Condenser	Provide Reflux to column	1.96×10^6 BTU/hr	956 ft^2 65 PSIA	CS/SS
22.	(H9)	#2 Distillation Column Reboiler	Provide Vapor to column	2.693×10^7 BTU/hr	2514 ft^2 250 PSIA	CS
23.	(H10)	#2 Redistribution Reactor Feed Vaporizer	Vaporize Reactants for Reactor	8.81×10^5 BTU/hr	78 ft^2 250 PSIA	CS/SS
24.	(H11)	#3 Distillation Column Condenser	Provide Column Reflux (Partial Condenser)	7.312×10^5 BTU/hr	593 ft^2 60 PSIA	CS/SS
25.	(H12)	#3 Distillation Column Reboiler	Provide Vapor to Column	9.64×10^5 BTU/hr	84 ft^2 250 PSIA	CS/SS
26.	(H13)	Silane Condenser	Condense Final Product for storage	4.9×10^4 BTU/hr	53 ft^2 250 PSIA	CS/SS
27.	(H14)	#4 Distillation Column Condenser	Provide Reflux	8.71×10^4 BTU/hr	84 ft^2 50 PSIA	CS/SS
28.	(H15)	#4 Distillation Column Reboiler	Provide Vapor to Column	1.2×10^5 BTU/hr	13 ft^2 250 PSIA	CS/SS
29.	(H16)	Absorber Precooler	Cool TET for absorption column	1.35×10^5 BTU/hr	35 ft^2 60 PSIA	CS/SS
30.	(H17)	Nitrogen Heater	Heat Nitrogen to regenerate Charcoal Adsorbers	2.52×10^4 BTU/hr	14.1 ft^2	CS
31.	(P1)	TCS Reactor Off Gas Recycle Compressor	Circulate Recycle Gas to Reactor	1.36×10^3 SCFM	26.5 Horsepower 75 PSIA Discharge	CS*

CASE B

TABLE IIA-1.5B(Continued)

32.	(P2)	#1 Distillation Column Feed Pump	Feed Column	136.5 gpm	106 PSI; 14.5 BHP	CS*
33.	(P3)	#1 Distillation Column Overheads Pump	Provide Reflux and remove overhead product	244 gpm	92.3 PSI; 22.5 BHP	CS*
34.	(P4)	#1 Distillation Column Bottoms Pump	Remove Bottoms Product to TET storage tank	69 gpm	106 PSI, 7.3 BHP	CS*
35.	(P5)	Process Water Feed Pump	Feed Process Water to Waste Treatment	48.6 gpm	82.5 PSI; 4 BHP	CS*
36.	(P6)	Caustic Feed	Feed Raw Material	1 gpm	118 PSI; 1/4 BHP	SS
37.	(P7)	#2 Distillation Column Overheads Pump	Provide Reflux and Remove Overhead Product	37.3 gpm	92.3 PSI; 3.4 BPH	SS
38.	(P8)	#2 Distillation Column Bottoms Pump	Remove Bottoms Product to TCS/TET storage tank	66.7 gpm	106.3 PSI; 7.1 BPH	SS
39.	(P9)	#3 Distillation Column Overhead Pump	Provide Reflux; Remove Overhead Product	9.7 gpm	87.3 PSI; 1 BHP	SS
40.	(P10)	#3 Distillation Column Bottoms Pump	Remove Bottoms Product to TCS/TET Tank	5.2 gpm	106.3 PSI; 1/2 BHP	SS

* Includes incremental higher cost for special purity requirements.

CASE B

TABLE IIA-1.5B (Continued)

41.	(P11)	#4 Distillation Column Overhead Pump	Provide Reflux, Remove Overhead Product	1 gpm	77.3 PSI; 1/4 BHP	SS
42.	(P12)	#4 Distillation Column Bottoms Pump	Remove Bottoms Product to Absorber Feed Tank	1 gpm	91.3 PSI; 1/4 BHP	SS
43.	(P13)	Silane Product Compressor	Liquefy Silane for Storage	66 SCFM	250 PSIA Discharge 6.5 HP	SS
44.	(P14)	Waste Feed Pump	Distillation Wastes to Waste Treatment	1 gpm	76.3 PSI; 1/4 BHP	CS
45.	(P15)	TCS Reactor Reed Pump	Feed TET to Reactor	69 gpm	92.3 PSI; 6.4 BHP	CS*
46.	(P16)	Waste Collection Pump	Distillation Wastes to Waste Tank	1 gpm	87.3 PSI; 1/4 BHP	CS
47.	(C1)	#1 Distillation Column	Separate TET from TCS	94,220 lb/hr of feed	7.56 ft. diameter 100 ft. tall, 50 trays	CS
48.	(C2)	#2 Distillation Column	Separate TCS from DCS	48,321 lb/hr of feed	10.6 ft. Diameter 136 ft. tall, 68 trays	CS
49.	(C3)	#3 Distillation Column	Separate Silane from other Chlorosilanes	7344 lb/hr of feed	2.01 ft. Diameter 29 ft. tall, 29 trays	SS
50.	(C4)	#4 Distillation Column	Strip TET for use in absorber	1007.7 lb/hr of feed	1.04 ft. Diameter 28.5 ft. tall, 38 trays	SS
51.	(C5)	Silane Absorber	Absorb Chlorosilane from Silane	819.3 lb/hr of vapor feed	0.823 ft. Diameter 12 ft. tall, 16 trays	SS

* Includes incremental higher cost for special purity requirements.

CASE B

TABLE IIA-1.5B (Continued)

52.	(C6)	Charcoal Adsorber	Activated Carbon Adsorbtion of Silane to remove Trace Chlorosilane	366 lb/hr of vapor feed	1 ft. Diameter 7 ft. tall (2), 623 lbs of carbon	SS
53.	(R1)	TCS Fluidized Bed Reactor	Produces TCS from TET, M.G. Silicon, and H_2		6.26 ft. in Diameter 26.5 ft. tall, 481 tubes 1", 16' long	SS
54.	(R2)	#1 Redistribution Reactor (2)	Redistribute TCS to DCS		2' Diameter by 15 ft. tall 1042 lbs catalyst	SS
55.	(R3)	#2 Redistribution Teactor (2)	Redistribute DCS to Silane		2.34' Diameter by 35 ft. tall 1667.2 lbs catalyst	SS
56.	(A1)	Fines Separator	Remove Silicon Fines carried over with TCS Reactor Off-gas		Standard design 30" Diameter	SS
57.	(A2)	Waste Treatment	Discharge innocuous effluent		1 column for adsorption + 1 heat exchanger to vaporize feed	SS
58.	(A3)	Hydrogen Flare	Dispose of Hydrogen from Waste Treatment		30 ft. stack 6" Diameter	CS

CASE B

TABLE IIA-1.6B

PRODUCTION LABOR REQUIREMENTS FOR
SILANE PROCESS -CASE B (Union Carbide)

	Unit Operation	Type	Skilled Labor, Man Hours Per Day	Skilled Labor, Man Hours Per lb. Silane	Semiskilled Labor Per Day	Semiskilled Labor Per lb. Silane
1.	TCS Production	B	65	.0085		
2.	Hydrogen Recycle	C	18	.0023		
3.	Raw Material Vaporization	C	50	.0065		
4.	TCS Condensation	C	50	.0065		
5.	TCS/TET Separation	C	62	.0081		
6.	#1 Redistribution Reactor	C	49	.0064		
7.	DCS/TCS Separation	C	52	.0068		
8.	#2 Redistibuiton Reactor	C	32	.0042		
9.	Silane Distillation	C	32	.0042		
10.	Silane Absorption	C	28	.0036		
11.	Silane Purification (adsorption)	A	36	.0047		
12.	Silane compression	B	23	.003		
13.	Silane Condensation	B	23	.003		
14.	Materials Handling	A			48	.0063
15.	Waste Treatment	B	60	.0078		
16.	Silicon Fines Separation	A	15	.002		
	TOTAL		595	.0776	48	.0063

NOTES:

1. A Batch Process of Multiple Small Units
   B Average Process
   c Automated Process

2. Man hours/day Unit from Figure 4-6, Peters and Timmerhaus (7).

3. CASE C - Revised Process

The status of preliminary process design activities involving Case C is shown in Table IIA-1.0C, which is representative of all the activities that compose the preliminary process design.

For the silane process, Union Carbide engineering, research and development personnel revised their flowsheet for a more optimum arrangement of major process equipment, raw material requirements and operating conditions. A joint meeing with Union Carbide and Lamar was conducted in late January for initial review of the revised flowsheet and potential lower plant capital investment and lower product cost for silane production.

In the revised silane process, the silicon tetrachloride is hydrogenated in a fluidized bed of silicon which is catalyzed by copper. The hydrogenation reaction is conducted at a higher pressure than originally proposed to increase the yeld of desireable trichlorosilane. The gas leaving the fluidized bed reactor is cooled and condensed to recover the liquid chlorosilanes. The hydrogen is recycled.

The condensed liquid chlorosilanes are separated by distillation. The inerts (dissolved gases) are removed in the initial distillation column. The remaining distillation columns separate the liquid chlorosilanes into primarily silicon tetrachloride, trichlorosilane, dichlorosilane and silane. The silicon tetrachloride is recycled back to the hydrogenation reactor. The trichlorosilane and dichlorosilane are sent to the redistribution reactors for rearranement of chlorine/hydrogen bonds to silicon. The final redistribution reactor product is sent to the silane distillation column. The silane is removed from this distillation and sent to product storage.

The initial review of the revised flowsheet for the silane process suggests favorable improvement over the original scheme.

The finalized flowsheet of the revised silane process (Case C) will issue in the next report.

CASE C

TABLE IIA-1.0C CHEMICAL ENGINEERING ANALYSES:
PRELIMINARY PROCESS DESIGN ACTIVITIES FOR SILANE PROCESS-CASE C (UNION CARBIDE)

Prel. Process Design Activity	Status	Prel. Process Design Activity	Status
1. Specify Base Case Conditions	◐	7. Equipment Design Calculations	O
1. Plant Size	◐	1. Storage Vessels	O
2. Product Specifics	◐	2. Unit Operations Equipment	O
3. Additional Conditions	●	3. Process Data (P, T, rate, etc.)	O
		4. Additional	O
2. Define Reaction Chemistry	◐		
1. Reactants, Products	◐	8. List of Major Process Equipment	O
2. Equilibrium	●	1. Size	O
		2. Type	O
3. Process Flow Diagram	◐	3. Materials of Construction	O
1. Flow Sequence, Unit Operations	◐		
2. Process Conditions (T, P, etc.)	◐	8a. Major Technical Factors	O
3. Environmental	◐	(Potential Problem Areas)	O
4. Company Interaction (Technology Exchange)	◐	1. Materials Compatibility	O
		2. Process Conditions Limitations	O
4. Material Balance Calculations	◐	3. Additional	O
1. Raw Materials	◐		
2. Products	◐	9. Production Labor Requirements	O
3. By-Products	●	1. Process Technology	O
		2. Production Volume	O
5. Energy Balance Calculations	◐		
1. Heating	◐	10. Forward for Economic Analysis	O
2. Cooling	◐		
3. Additional	◐		
6. Property Data	◐		
1. Physical	◐		
2. Thermodynamic	◐		
3. Additional	◐		

O Plan
◐ In Progress
● Complete

B. OTHER PROCESSES

For other processes under consideration for solar cell grade silicon production, the following technical progress reports are being received and screened:

1. Battelle Process ($Zn/SiCl_4$)
2. Union Carbide Process (SiH_4)
3. Motorola Process (SiF_4/SiF_2)
4. Westinghouse Process ($Na/SiCl_4$)
5. Dow Process (C/SiO_2)
6. SRI Process (Na/SiF_4)
7. AeroChem Process
8. J.C. Schumacher Co. ($SiBr_4$)

III. ECONOMIC ANALYSES (TASK 3)

A. Silane Process (Union Carbide)

Majoy efforts during this reporting period focused on completion of the preliminary economic analysis of the Union Carbide Silane Process as characterized by the original flowsheet received. Two cases are covered for the original flowsheet:

Case A - Regular Process Storage

Case B - Minimum Process Storage

In additional efforts, Union Carbide personnel have revised their original flowsheet for a more optimum arrangement of major process equipment, raw materials and operating conditions:

Case C - Revised Process

Several cost benefits (lower capital and operating costs) are suggested from initial review of the revised process.

Each case (A, B and C) is discussed separately in the following sections.

1. CASE A - Regular Process Storage

A summation of the key results for CASE A is presented in the following table:

CASE A - Regular Porcess Storage

Process	Silane (Union Carbide)
Plant Size	1270 MT/year Silane
Intermediate Product Storage Consideration	Regular
Cost Basis	1975 Dollars
Plant Investment	$19,094,000
Product Cost (No Profit)	$5.54/lb of Silane

The detailed results from the completed preliminary economic analysis are presented in a tabular format for Case A. Note that all dollar values are given per pound of Silane instead of per Kg of silicon. The guide for the tabular format is given below:

- Preliminary Economic Analysis Activities....Table IIIA-1.0A
- Process Design Inputs........................Table IIIA-1.1A
- Base Case Conditions.........................Table IIIA-1.2A
- Raw Material Cost............................Table IIIA-1.3A
- Utility Cost.................................Table IIIA-1.4A
- Major Porcess Equipment Cost.................Table IIIA-1.5A
- Production Labor Cost........................Table IIIA-1.6A
- Plant Investment.............................Table IIIA-1.7A
- Total Product Cost...........................Table IIIA-1.8A

CASE A

Table IIIA-1.0A

ECONOMIC ANALYSES:
PRELIMINARY ECONOMIC ANALYSIS ACTIVITIES FOR SILANE PROCESS
CASE A (UNION CARBIDE)

Prel. Process Economic Activity	Status
1. Process Design Inputs	●
1. Raw Material Requirements	●
2. Utility Requirements	●
3. Equipment List	●
4. Labor Requirements	●
2. Specify Base Case Conditions	●
1. Base Year for Costs	●
2. Appropriate Indices for Costs	●
3. Additional	●
3. Raw Material Costs	●
1. Base Cost/Lb. of Material	●
2. Material Cost/lb of Silane	●
3. Total Cost/lb of Silane	●
4. Utility Costs	●
1. Base Cost for Each Utility	●
2. Utility Cost/lb of Silane	●
3. Total Cost/lb of Silane	●
5. Major Process Equipment Costs	●
1. Individual Equipment Cost	●
2. Cost Index Adjustment	●

Prel. Process Economic Activity	Status
6. Production Labor Costs	●
1. Base Cost Per Man Hour	●
2. Cost/lb Silane Per Area	●
3. Total Cost/lb Silane	●
7. Estimation of Plant Investment	◐
1. Battery Limits Direct Costs	●
2. Other Direct Costs	●
3. Indirect Costs	●
4. Contingency	●
5. Total Plant Investment (Fixed Capital)	●
8. Estimation of Total Product Cost	●
1. Direct Manufacturing Cost	●
2. Indirect Manufacturing Cost	●
3. Plant Overhead	●
4. By-Product Credit	●
5. General Expenses	●
6. Total Cost of Product	●

O Plan
◐ In Progress
● Complete

TABLE IIIA-1.1A

PROCESS DESIGN INPUTS FOR
SILANE PROCESS - CASE A (UNION CARBIDE)

1. Raw Material Requirements
   - M.G. Silicon, anhydrous HCl, caustic, hydrogen.
   - see table for "Raw Material Cost"
2. Utility
   - electrical, steam, cooling water, etc.
   - see table for "Utility Cost"
3. Equipment List
   - 76 pieces of major process equipment
   - process vessels, heat exchangers, reactor, etc.
   - see table for "Major Process Equipment Cost"
4. Labor Requirements
   - production labor for purification, vaporization, product handling, etc.
   - see table for "Production Labor Cost"

CASE A

TABLE IIIA-1.2A

BASE CASE CONDITIONS FOR
SILANE PROCESS-CASE A (UNION CARBIDE)

1. Capital Equipment

   -January 1975 Cost Index for Capital Equipment Cost
   -January 1974 Cost Index Value = 430

2. Utilities

   -Electrical, Steam, Cooling Water, Nitrogen
   -January 1975 Cost Index (U.S. Dept. Labor)
   -Values determined by literature search and summarized in cost standardization work

3. Raw Material Cost

   -Chemical Marketing Reporter
   -January 1975 Value
   -Other Sources

4. Labor Cost

   -Average for Chemical Petroleum, Coal and Allied Industries (1975)
   -Skilled $6.90/hr
   -Semiskilled $4.90/hr

CASE A

TABLE IIIA-1.3A

RAW MATERIAL COST FOR SILANE PROCESS - CASE A
(UNION CARBIDE)

Raw Material	Requirement lb/lb of Silane	$/lb of Material	Cost $/lb of Silane
1. HCl	1.239	.10	.12
2. Hydrogen	.362	.96	.35
3. Caustic (50%)	2.448	.0382	.09
4. M.G. Silicon	1.11	.454	.50
			1.06/lb Silane

CASE A

TABLE IIIA-1.4 A

UTILITY COST FOR SILANE PROCESS -CASE A
(UNION CARBIDE)

Utility	Requirement/lb of Silane	Cost of Utility	Cost $/lb of Silane
1. Electricty	.253. Kw-Hr	$ .03/Kw-Hr	.0076
2. Steam	190.34 lbs	1.25/M lb	.2379
3. Cooling Water	168.12 gallons	.08/M gal	.0134
4. Process water	8.22 gallons	.35/M gal	.0029
5. Refrigerant (23°F)	27.1 BTU	4.75/MM BTU	.0001
6. Refrigerant (5°F)	79.1 BTU	6.40/MM BTU	.0005
7. Refrigerant (-7°F)	26.4 BTU	7.50/MM BTU	.0002
8. Refrigerant (-20°F)	2.3 M BTU	8.70/MM BTU	.0200
9. Refrigerant (-30°F)	30.8 M BTU	9.60/MM BTU	.2957
10. Refrigerant (-40°F)	369 BTU	10.50/MM BTU	.0039
11. Refrigerant (-50°F)	3.5 M BTU	11.42/MM BTU	.0400
12. High Temperature Heat Exchange Fluid	3.324×10^4 BTU	3.0/MM BTU	.099
13. Nitrogen	5.54 SCF	.50/M SCF	.0028
			.724 /lb Silane

CASE A

TABLE IIIA-1.5A

PURCHASED COST OF MAJOR PROCESS EQUIPMENT FOR SILANE PROCESS-CASE A (UNION CARBIDE)

	Equipment	Purchased Cost, $1000
1.	(T1) M.G. Silicon Storage Hopper	12.05
2.	(T2) Hydrogen Storage Tank	179.2
3.	(T3) Liquid HCl Storage Tank	95.27
4.	(T4) Recycle TET Storage	214.4
5.	(T5) TCS Reactor Off-Gas Flash Tank	0.71
6.	(T6) TCS/TET Storage	214.4
7.	(T7) #1 Distillation Column Condensate Accumlator	8.51
8.	(T8) #1 Redistribution Reactor Feed Tank	244.99
9.	(T9) #1 Redistribution Reactor Product Tank	245.0
10.	(T10) #2 Distillation Column Condensate Accumulator	7.37
11.	(T11) #2 Redistribution Reactor Feed Tank	76.03
12.	(T12) #2 Redistribution Reactor Product Tank	221.17
13.	(T13) #3 Distillation Column Condensate Accumulator	2.76
14.	(T14) #3 Distillation Column Condensate Tank	147.44
15.	(T15) #4 Distillation Column Feed Tank	53.45
16.	(T16) #4 Distillation Column Condensate Accumulator	2.76
17.	(T17) #4 Distillation Column Condensate Tank	34.1
18.	(T18) Waste Tank	17.01
19.	(T19) Absorber Feed Tank	16.59
20.	(T20) Silane Storage	255.9
21.	(T21) Caustic Storage	92.15
22.	(H1) TCS Reactor Recycle Gas Heater	8.12
23.	(H2) HCl Vaporizer	1.15
24.	(H3) TET Vaporizer	18.48
25.	(H4) TCS Reactor Recycle Condenser	38.98

CASE A

TABLE IIIA-1.5A(continued)

26.	(H5)	#1 Distillation Column Preheater	3.24
27.	(H6)	#1 Distillation Column Condenser	22.4
28.	(H7)	#1 Distillation Column Reboiler	23.7
29.	(H8)	#2 Distillation Column Condenser	21.08
30.	(H9)	#2 Distillation Column Reboiler	21.16
31.	(H10)	#2 Redistribution Reactor Feed Vaporizer	3.67
32.	(H11)	#2 Redistribution Reactor Product condenser	8.62
33.	(H12)	#3 Distillation Column Preheater	2.86
34.	(H13)	#3 Distillation Column Condenser	14.95
35.	(H14)	#3 Distillation Column Reboiler	3.88
36.	(H15)	Silane Condenser	2.29
37.	(H16)	#4 Distillation Column Condenser	3.48
38.	(H17)	#4 Distillation Column Reboiler	1.33
39.	(H18)	Absorber Pre-cooler	1.79
40.	(H19)	Nitrogen Heater	.92
41.	(P1)	TCS Reactor Off-gas Recycle Compressor	35.1
42.	(P2)	#1 Distillation Column Feed Pump	5.03
43.	(P3)	#1 Distillation Column Overheads Pump	6.04
44.	(P4)	#1 Distillation Colum Bottoms Pump	3.59
45.	(P5)	Process Water Feed Pump	2.87
46.	(P6)	Caustic Feed Pump	1.25
47.	(P7)	#1 Redistribution Reactor Feed Pump	4.02
48.	(P8)	#2 Distillation Column Feed Pump	3.59
49.	(P9)	#2 Distillation Column Overheads Pump	2.57
50.	(P10)	#2 Distillation Column Bottoms Pump	3.59
51.	(P11)	#2 Redistribution Reactor Feed Pump	2.09
52.	(P12)	#3 Distillation Column Feed Pump	1.77

TABLE IIIA-1.5A(continued)

53.	(P13)	#3 Distillation Column Overheads Pump	1.77
54.	(P14)	#3 Distillation Column Bottoms Pump	1.47
55.	(P15)	#4 Distillation Column Feed Pump	1.23
56.	(P16)	#4 Distillation Column Overheads Pump	1.23
57.	(P17)	#4 Distillation Column Bottoms Pump	1.23
58.	(P18)	#4 Distillation Condensate Recycle Pump	1.23
59.	(P19)	Silane Product Compressor	17.55
60.	(P20)	Waste Feed Pump	.62
61.	(P21)	TCS Reactor Feed Pump	3.31
62.	(P22)	#3 Distillation Condensate Recycle Pump	1.47
63.	(P23)	Waste Collection Pump	.62
64.	(P24)	Absorber Feed Pump	1.23
65.	(C1)	#1 Distillation Column	100.66
66.	(C2)	#2 Distillation Column	214.08
67.	(C3)	#3 Distillation Column	40.19
68.	(C4)	#4 Distillation Column	21.14
69.	(C5)	Silane Absorber	15.06
70.	(C6)	Charcoal Adsorber	18.0
71.	(R1)	TCS Fluidized Bed Reactor	155.06
72.	(R2)	#1 Redistribution Reactor	13.26
73.	(R3)	#2 Redistribution Reactor	33.14
74.	(A1)	Fines Separator	2.0
75.	(A2)	Waste Treatment	18.72
76.	(A3)	Hydrogen Flare	0.10
		TOTAL PURCHASED EQUIPMENT COST	$3079.31

CASE A

TABLE IIIA-1.6A

PRODUCTION LABOR COST FOR SILANE PROCESS - CASE A
(UNION CARBIDE)

Unit Operation	Skilled Labor Man-Hrs/lb Silane	Semiskilled Labor Man-Hrs/lb Silane	Cost $/lb of Silane
1. TCS Production	.0085		.05865
2. Hydrogen Recycle	.0023		.01587
3. Raw Material Vaporization	.0065		.04485
4. TCS Condensation	.0065		.04485
5. TCS/TET Separation	.0081		.05589
6. #1 Redistribution Reactor	.0064		.04416
7. DCS/TCS Separation	.0068		.04692
8. #2 Redistribution Reactor	.0042		.02898
9. Silane Distillation	.0042		.02898
10. Silane Absorption	.0036		.02484
11. Silane Purification (Adsorption)	.0047		.03243
12. Silane Compression	.003		.0207
13. Silane Condensation	.003		.0207
14. Materials Handling	--	.0063	.03087
15. Waste Treatment	.0078		.05382
16. Silicon Fines Separation	.002		.0138
		TOTAL COST	$.5663/lb of Silane

NOTES

Based on labor costs of $6.90 skilled, $4.90 semiskilled.

TABLE IIIA -1.7A

ESTIMATION OF PLANT INVESTMENT FOR SILANE PROCESS - CASE A (UNION CARBIDE)

	Investment $1000
1. DIRECT PLANT INVESTMENT COSTS	
1. Major Process Equipment Cost	$3079.31
2. Installation of Major Process Equipment	1324.10
3. Process Piping, Installed	2278.69
4. Instrumentation, Installed	585.07
5. Electrical, Installed	307.93
6. Process Buildings, Installed	307.93
1a. SUBTOTAL FOR DIRECT PLANT INVESTMENT COSTS (PRIMARILY BATTERY LIMIT FACILITIES)	7883.03
2. OTHER DIRECT PLANT INVESTMENT COSTS	
1. Utilities, Installed	1478.07
2. General Services, Site Development, Fire Protection, etc.	369.52
3. General Buildings, Offices, Shops, etc.	431.10
4. Receiving, Shipping Facilities	646.66
2a. SUBTOTAL FOR OTHER DIRECT PLANT INVESTMENT COSTS (PRIMARILY OFFSITE FACILITIES OUTSIDE BATTERY LIMITS)	2925.35
3. TOTAL DIRECT PLANT INVESTMENT COST, 1a + 2a	10808.38
4. INDIRECT PLANT INVESTMENT COSTS	
1. Engineering, Overhead, etc.	1693.62
2. Normal Cont. for Floods, Strikes, etc.	2186.31
4a. TOTAL INDIRECT PLANT INVESTMENT COST	3879.93
5. TOTAL DIRECT AND INDIRECT PLANT INVESTMENT COST, 3 + 4a	14688.31
6. OVERALL CONTINGENCY	4406.49
7. FIXED CAPITAL INVESTMENT FOR PLANT, 5 + 6	19094.80
8. WORKING CAPITAL INVESTMENT FOR PLANT	
9. TOTAL PLANT INVESTMENT, 7 + 8	

CASE A

TABLE IIIA-1.8A

ESTIMATION OF TOTAL PRODUCT COST FOR SILANE PROCESS- CASE A
(UNION CARBIDE)

		$/lb of Silane
1.	Direct Manfacturing Cost (Direct Charges)	
	1. Raw Materials- from prel. design	1.06
	2. Direct Operating Labor- from prel. design	.5663
	3. Utilities-from prel. design	.724
	4. Supervision and Clerical, .	.085
	5. Maintenance and Repairs,	.682
	6. Operating Supplies,	.136
	7. Laboratory Charge,	.085
	8. Patents and Royalties, . costs	----
2.	Indirect Manufacturing Cost (Fixed Charges)	
	1. Depreciation	.682
	2. Local Taxes	.136
	3. Insurance	.068
	4. Interest	----
3.	Plant Overhead	.595
4.	By-Product Credit- from prel. design	--
4a.	Total Manufacturing Cost, 1 + 2 + 3 + 4	4.819
5.	General Expenses	
	1. Administration, .	.289
	2. Distribution and Sales, cost	.289
	3. Research and Development, . cost	.145
6.	Total Cost of Product, 4a + 5	5.54

2. CASE B - Minimum Process Storage

A summation of the key results for Case B is presented in the following table:

CASE B - Minimum Process Storage

Process	Silane (Union Carbide)
Plant Size	1270 MT/year Silane
Intermediate Product Storage Consideration	Minimum
Cost Basis	1975 Dollars
Plant Investment	$11,138,000
Product Cost (No Profit)	$4.58/lb of Silane

The detailed results from the completed preliminary economic analysis are presented in a tabular format for Case B. Note that all dollar values are given per pound of silane instead of per Kg of silicon. The guide for the tabular format is given below:

- Preliminary Economic Analysis Activities...Table IIIA-1.0B
- Process Design Inputs...Table IIIA-1.1B
- Base Case Conditions...Table IIIA-1.2B
- Raw Material Cost...Table IIIA-1.3B
- Utility Cost...Table IIIA-1.4B
- Major Process Equipment Cost...Table IIIA-1.5B
- Production Labor Cost...Table IIIA-1.6B
- Plant Investment...Table IIIA-1.7B
- Total Product Cost...Table IIIA-1.8B

CASE B

TABLE IIIA-1.0B

ECONOMIC ANALYSES:

PRELIMINARY ECONOMIC ANALYSIS ACTIVITES FOR SILANE PROCESS - CASE B (UNION CARBIDE)

	Prel. Process Economic Activity	Status
1.	Process Design Inputs	●
	1. Raw Material Requirements	●
	2. Utility Requirements	●
	3. Equipment List	●
	4. Labor Requirements	●
2.	Specify Base Case Conditions	●
	1. Base Year for Costs	●
	2. Appropriate Indices for Costs	●
	3. Additional	●
3.	Raw Material Costs	●
	1. Base Cost/Lb. of Material	●
	2. Material Cost/lb of Silane	●
	3. Total Cost/lb of Silane	●
4.	Utility Costs	●
	1. Base Cost for Each Utility	●
	2. Utility Cost/lb of Silane	●
	3. Total Cost/lb of Silane	●
5.	Major Process Equipment Costs	●
	1. Individual Equipment Cost	●
	2. Cost Index Adjustment	●

	Prel. Process Economic Activity	Status
6.	Production Labor Costs	●
	1. Base Cost Per Man Hour	●
	2. Cost/lb Silane Per Area	●
	3. Total Cost/lb Silane	●
7.	Estimation of Plant Investment	●
	1. Battery Limits Direct Costs	●
	2. Other Direct Costs	●
	3. Indirect Costs	●
	4. Contingency	●
	5. Total Plant Investment (Fixed Capital)	●
8.	Estimation of Total Product Cost	●
	1. Direct Manufacturing Cost	●
	2. Indirect Manufacturing Cost	●
	3. Plant Overhead	●
	4. By-Product Credit	●
	5. General Expenses	●
	6. Total Cost of Product	●

O Plan
◐ In Progress
● Complete

CASE B

TABLE IIIA-1.1B

PROCESS DESIGN INPUTS FOR
SILANE PROCESS - CASE B (UNION CARBIDE)

1. Raw Material Requirements
   - M.G. Silicon, anhydrous HCl, caustic, hydrogen.
   - see table for "Raw Material Cost"

2. Utility
   - electrical, steam, cooling water, etc.
   - see table for "Utility Cost"

3. Equipment List
   - 58 pieces of major process equipment
   - process vessels, heat exchangers, reactor, etc.
   - see table for "Major Process Equipment Cost"

4. Labor Requirements
   - production labor for purification, vaporization, product handling, etc.
   - see table for "Production Labor Cost"

CASE B

TABLE IIIA-1.2B

BASE CASE CONDITIONS FOR
SILANE PROCESS - CASE B (UNION CARBIDE)

1. Capital Equipment

   -January 1975 Cost Index for Capital Equipment Cost
   -January 1975 Cost Index Value = 430

2. Utilities

   -Electrical, Steam, Cooling Water, Nitrogen
   -January 1975 Cost Index (U.S. Dept. Labor)
   -Values determined by literature search and summarized in cost standardization work

3. Raw Material Cost

   -Chemical Marketing Reporter
   -January 1975 Value
   -Other Sources

4. Labor Cost

   -Average for Chemical Petroleum, Coal and Allied Industries (1975)
   -Skilled \$6.90/hr
   -Semiskilled \$4.90/hr

CASE B

TABLE IIIA-1.3B

RAW MATERIAL COST FOR SILANE PROCESS-CASE B
(UNION CARBIDE)

Raw Material	Requirement lb/lb of Silane	$/lb of Material	Cost $/lb of Silane
1. HCl	1.239	.10	.12
2. Hydrogen	.362	.96	.35
3. Caustic (50%)	2.448	.0382	.09
4. M.G. Silicon	1.11	.454	.50
			1.06/lb Silane

CASE B

TABLE IIIA-1.4B

UTILITY COST FOR SILANE PROCESS -CASE B
(UNION CARBIDE)

Utility	Requirement/lb of Silane	Cost of Utility	Cost $/lb of Silane
1. Electricty	.212 KW-Hr	$ .03/Kw-Hr	.0064
2. Steam	186.72 lbs	1.25/M lb	.2334
3. Cooling Water	168.12 gallons	.08/M gal	.0134
4. Process Water	8.22 gallons	.35/M gal	.0029
5. Refrigerant (-20°F)	2.3 M BTU	8.70/MM BTU	.0200
6. Refrigerant (-30°F)	30.8 M BTU	9.60/MM BTU	.2957
7. Refrigerant (-40°F)	25.3 BTU	10.50/MM BTU	.0003
8. Refrigerant (-50°F)	517.2 BTU	11.42/MM BTU	.0059
9. High Temperature Heat Exchange Fluid	33.24 M BTU	3.0/MM BTU	.0997
10. Nitrogen	5.54 SCF	.50/M SCF	.0028
			.6805/lb Silane

CASE B

TABLE 111A-1.5B

PURCHASED COST OF MAJOR PROCESS EQUIPMENT FOR
[illegible] - CASE B (UNION CARBIDE)

Equipment			Purchased Cost, $1000
1.	(T1)	[illegible]	12.05
2.	(T2)	Hydro[illegible]	179.2
3.	(T3)	[illegible]	95.27
4.	(T4)	[illegible]	125.55
5.	(T5)	TCS Reactor Off-Gas Flash Tank	0.71
6.	(T6)	[illegible]	214.4
7.	(T7)	[illegible] Condensate Accumulator	8.51
8.	(T8)	#2 Distillation Column Condensate Accumulator	7.37
9.	(T9)	#3 Distillation Column Condensate Accumulator	2.76
10.	(T10)	#4 Distillation Column Condensate Accumulator	2.76
11.	(T11)	Waste [illegible]	17.01
12.	(T12)	Silane Stor[illegible]	82.09
13.	(T13)	Caustic [illegible]	92.15
14.	(H1)	TCS Reactor Recycle Gas Heater	8.12
15.	(H2)	HCl Vaporizer	1.15
16.	(H3)	TET Vaporizer	18.48
17.	(H4)	TCS Reactor Recycle Condenser	38.98
18.	(H5)	#1 Distillation Column Preheater	3.24
19.	(H6)	#1 Distillation Column Condenser	22.4
20.	(H7)	#1 Distillation Column Reboiler	23.7
21.	(H8)	#2 Distillation Column Condenser	21.08

CASE B

TABLE IIIA -1.5B (Continued)

22.	(H9)	#2 Distillation Column Reboiler	21.16
23.	(H10)	#2 Redistributor Reactor Feed Vaporizer	3.67
24.	(H11)	#3 Distillation Column Condenser	14.95
25.	(H12)	#3 Distillation Column Reboiler	3.88
26.	(H13)	Silane Condenser	2.29
27.	(H14)	#4 Distillation Column Condenser	3.48
28.	(H15)	#4 Distillation Column Reboiler	1.33
29.	(H16)	Absorber Pre-cooler	1.79
30.	(H17)	Nitrogen Heater	.92
31.	(P1)	TCS Reactor Off-gas Recycle Compressor	35.1
32.	(P2)	#1 Distillation Column Feed Pump	5.03
33.	(P3)	#1 Distillation Column Overheads Pump	6.04
34.	(P4)	#1 Distillation Column Bottoms Pump	3.59
35.	(P5)	Process Water Feed Pump	2.87
36.	(P6)	Caustic Feed Pump	1.25
37.	(P7)	#2 Distillation Column Overheads Pump	2.57
38.	(P8)	#2 Distillation Column Bottoms Pump	3.59
39.	(P9)	#3 Distillation column Overheads Pump	1.77
40.	(P10)	#3 Distillation Column Bottoms Pump	1.47
41.	(P11)	#4 Distillation Column Overheads Pump	1.23
42.	(P12)	#4 Distillation Column Bottoms Pump	1.23
43.	(P13)	Silane Product Compressor	17.55
44.	(P14)	Waste Feed Pump	.62

CASE B

TABLE IIIA-1.5b (Continued)

45.	(P15)	TCS Reactor Feed Pump	3.31
46.	(P16)	Waste Collection Pump	.62
47.	(C1)	#1 Distillation Column	106.66
48.	(C2)	#2 Distillation Column	214.08
49.	(C3)	#3 Distillation Column	40.19
50.	(C4)	#4 Distillation Column	21.14
51.	(C5)	Silane Absorber	15.06
52.	(C6)	Charcoal Adsorber	18.0
53.	(R1)	TCS Fluidized Bed Reactor	155.06
54.	(R2)	#1 Redistribuiton Reactor	26.52
55.	(R3)	#2 Redistribution Reactor	66.28
56.	(A1)	Fines Separator	2.0
57.	(A2)	Waste Treatment	18.72
58.	(A3)	Hydrogen Flare	0.10
		TOTAL PURCHASED EQUIPMENT COST	1796.17

CASE B

TABLE IIIA-1.6

PRODUCTION LABOR COST FOR SILANE PROCESS - CASE B
(UNION CARBIDE)

	Unit Operation	Skilled Labor Man-Hrs/lb Silane	Semiskilled Labor Man-Hrs/lb Silane	Cost $/lb of Silane
1.	TCS Production	.0085		.05865
2.	Hydrogen Recycle	.0023		.01587
3.	Raw Material Vaporization	.0065		.04485
4.	TCS Condensation	.0065		.04485
5.	TCS/TET Separation	.0081		.05589
6.	#1 Redistribution Reactor	.0064		.04416
7.	DCS/TCS Separation	.0068		.04692
8.	#2 Redistribution Reactor	.0042		.02898
9.	Silane Distillation	.0042		.02898
10.	Silane Absorption	.0036		.02484
11.	Silane Purification (Adsorption)	.0047		.03243
12.	Silane Compression	.003		.0207
13.	Silane Condensation	.003		.0207
14.	Materials Handling	--	.0063	.03087
15.	Waste Treatment	.0078		.05382
16.	Silicon Fines Separation	.002		.0138

TOTAL COST $.5663/lb of Silane

NOTES

Based on labor costs of $6.90 skilled, $4.90 semiskilled.

CASE B

TABLE IIIA-1.7B

ESTIMATION OF PLANT INVESTMENT FOR SILANE PROCESS CASE B
(UNION CARBIDE)

	Investment $1000
1. DIRECT PLANT INVESTMENT COSTS	
1. Major Process Equipment Cost	$ 1796.17
2. Installation of Major Process Equipment	772.35
3. Process Piping, Installed	1329.17
4. Instrumentation, Installed	341.27
5. Electrical, Installed	179.62
6. Process Buildings, Installed	179.62
1a. SUBTOTAL FOR DIRECT PLANT INVESTMENT COSTS (PRIMARILY BATTERY LIMIT FACILITIES)	4598.2
2. OTHER DIRECT PLANT INVESTMENT COSTS	
1. Utilities, Installed	862.16
2. General Services, Site Development, Fire Protection, etc.	215.54
3. General Buildings, Offices, Shops, etc.	251.46
4. Receiving, Shipping Facilities	377.20
2a. SUBTOTAL FOR OTHER DIRECT PLANT INVESTMENT COSTS (PRIMARILY OFFSITE FACILITIES OUTSIDE BATTERY LIMITS)	1706.36
3. TOTAL DIRECT PLANT INVESTMENT COST, 1a + 2a	6304.56
4. INDIRECT PLANT INVESTMENT COSTS	
1. Engineering, Overhead, etc.	987.89
2. Normal Cont. for Floods, Strikes, etc.	1275.28
4a. TOTAL INDIRECT PLANT INVESTMENT COST	2263.17
5. TOTAL DIRECT AND INDIRECT PLANT INVESTMENT COST, 3 + 4a	8567.73
6. OVERALL CONTINGENCY	2570.32
7. FIXED CAPITAL INVESTMENT FOR PLANT, 5 + 6	11138.05
8. WORKING CAPITAL INVESTMENT FOR PLANT	
9. TOTAL PLANT INVESTMENT, 7 + 8	

CASE B

TABLE IIIA-1.8B

ESTIMATION OF TOTAL PRODUCT COST FOR SILANE PROCESS -CASE B

ION CARBIDE)

	$/lb of Silane
1. Direct Manfacturing Cost (Direct Charges)	
1. Raw Materials- from prel. design	1.06
2. Direct Operating Labor- from prel. design	.5663
3. Utilities-from prel. design	.6805
4. Supervision and Clerical,	.0849
5. Maintenance and Repairs,	.3976
6. Operating Supplies,	.0795
7. Laboratory Charge,	.0849
8. Patents and Royalties, costs	----
2. Indirect Manufacturing Cost (Fixed Charges)	
1. Depreciation	.3976
2. Local Taxes	.0795
3. Insurance	.0398
4. Interest	-----
3. Plant Overhead	.51
4. By-Product Credit- from prel. design	--
4a. Total Manufacturing Cost, 1 + 2 + 3 + 4	3.9806
5. General Expenses	
1. Administration, cost	.2388
2. Distribution and Sales, cost	.2388
3. Research and Development, cost	.1194
Total Cost of Product, 4a + 5	4.58

3. CASE C -Revised Process

Initial review of the revised silane process (Case C) suggests favorable cost benefits over the original scheme.

Operation of the silicon tetrachloride reaction at higher pressure for increased trichlorosilane yield should lower recycle requirements. Lower recycle requirements will lower capital equipment and labor costs. The distillation train as now proposed will operate at several hundred pounds pressure compared to original lower pressure. This higher pressure permits use of cooling water in the condensers and does not require expensive low temperature refrigeration as originally proposed. This will provide lower operating (utilities) cost in 3 of the 4 distillation columns. The higher pressure also permits use of smaller diameter columns (vapor loading, density proportional to pressure). The elimination of hydrogen chloride reduces starting material costs. Also, the use of hydrogen from the silane pyrolysis provides additional lower feed material costs.

The revised silane process (Case C) should provide the following cost benefits:

- -lower capital costs
- -lower raw material costs
- -lower operating labor costs

B. OTHER PROCESSES

The following technical progress reports are being received and screened for technical information for additional processes under consideration for solar cell grade silicon:

1. Battelle Process ($Zn/SiCl_4$)
2. Union Carbide Process (SiH_4)
3. Motorola Process ($SiF_4/SiCl_4$)
4. Westinghouse Porcess ($Na/SiCl_4$)
5. Dow Process (C/SiO_2)
6. SRI Process (Na/SiF_4)
7. AeroChem Process
8. J.C. Schumacher Co. ($SiBr_4$)

IV. SUMMARY - CONCLUSIONS

The following summary-conclusions are made as a result of major activities accomplished during this reporting period:

1. Task 1

In analyses of process system properties, major activities focused on properties of silicon tetrachloride which is the source material for several alternate processes under consideration for solar cell grade silicon production. The status and progress are reported for physical, thermodynamic and transport property data.

Experimental determination of gaseous thermal conductivity of silicon source materials was continued. Initial results for gas thermal conductivity of silicon tetrafluoride and trichlorosilane are reported in respective temperature ranges of 25 to 400°C and 50 to 400°C.

2. Task 2

The preliminary process design was completed for the Union Carbide Silane Process as characterized by the original flowsheet. Two cases were considered for the original flowsheet:

Case A - Regular Process Storage

Case B - Minimum Process Storage

Two cases were presented because of the large recycle requirements for this process, necessitating considerable tankage for material storage. The major resulting differnce between CASES A and B is 76 versus 58 pieces of major process equipment.

For the silane process, Union Carbide engineering-research personnel revised their flowsheet. The revised process (Case C) involves a more optimum arrangement of major process equipment, raw material requirements and operating conditions:

Case C - Revised Process

A joint meeing with Union Carbide and Lamar was conducted during this reporting period for review of the revised flowsheet. Initial review results suggest that the revised process is a favorable improvement over the original scheme.

3. Task 3

The preliminary process design results for Cases A and B were used for economic analyses. Because of the large differences in surge tankage between major unit operations, the fixed capital investment varied from $19,094,000 to $11,138,000 for Cases A and B, respectively. The product cost for Case A is $5.54/lb of silane versus $4.58/lb of silane for Case B.

The initial issue of the revised flowsheet (Case C) for the silane process indicates favorable cost benefits over the original scheme. This includes higher pressure silicon tetrachloride hydrogenation for increased trichlorosilane yield with lower recycle requirements, higher pressure distillation not requiring expensive low temperature refrigeration, and improved raw material feed requirements. The revised silane process (Case C) should provide the following cost benefits:

-lower capital cost
-lower raw material costs
-lower operating labor costs

V. PLANS

Plans for the next reporting period are summarized below:

1. Task 1

Continue analyses of process system properties for silicon source materials under consideration for solar grade silicon including additional correlation of experimental data.

Experimental thermal conductivity data measurements for silicon source materials will be continued with emphasis on chlorinated silanes.

2. Task 2

Continue interactions with Union Carbide on their revised silane process (Case C).

Initiate preliminary design of revised silane process using finalized flowsheet.

3. Task 3

Economic analyses will be initiated as preliminary process designs are forwarded.

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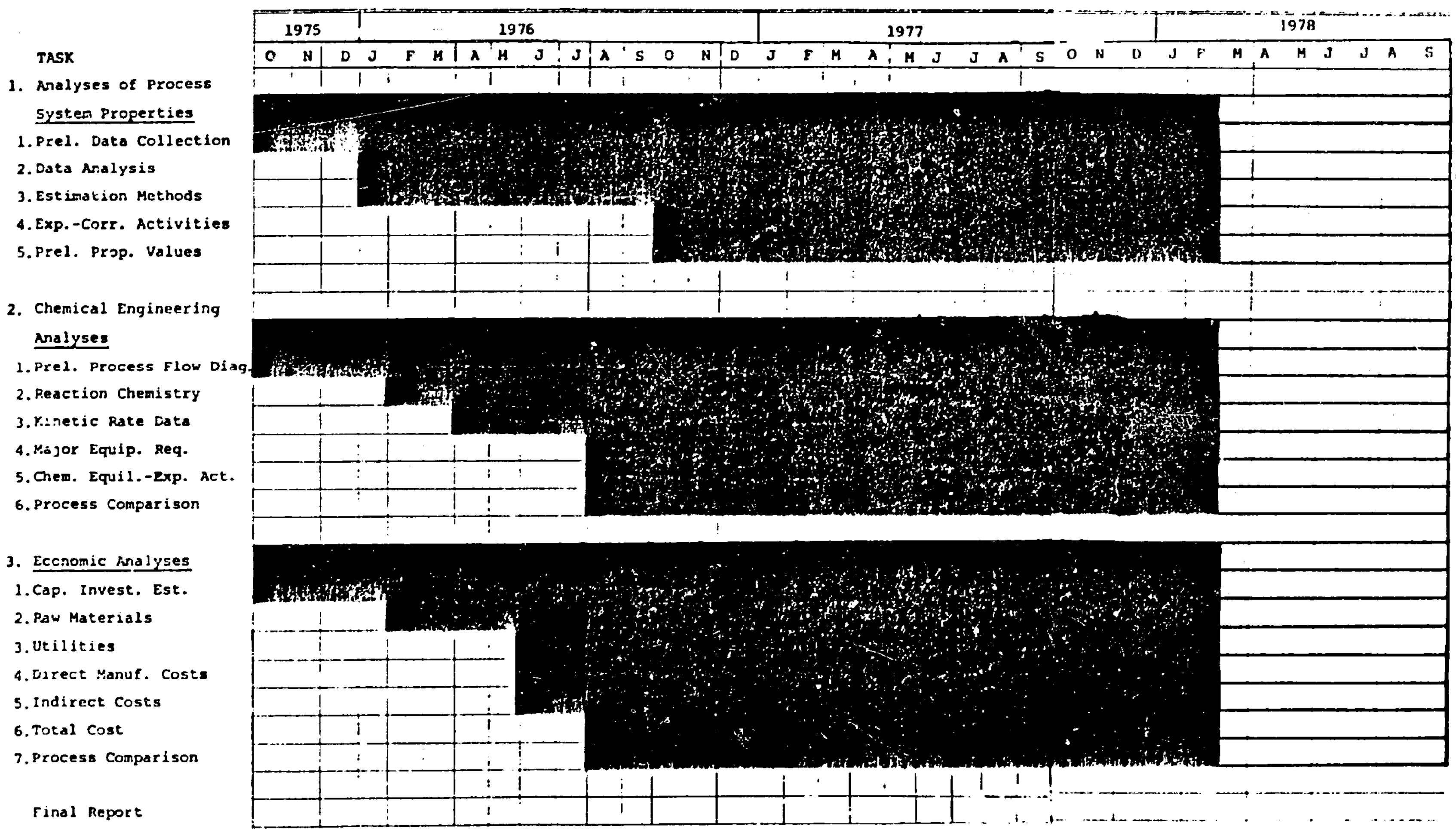
MILESTONE CHART



PROCESS FEASIBILITY STUDY IN
SUPPORT OF SILICON MATERIAL TASK I

JPL Contract No. 954343