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PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL
TASK I

Quarterly Technical Progress Report (IX), December 1977

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Chemical Engineering Department
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Solar Energy

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PROCESS FEASIBILITY STUDY IN SUPPORT OF
SILICON MATERIAL TASK I

QUARTERLY TECHNICAL PROGRESS REPORT (IX)

December, 1977

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ABSTRACT

During this reporting period, major activities were devoted to process system properties, chemical engineering and economic analyses.

Analyses of process system properties were continued for silicon source materials under consideration for solar cell grade silicon production. Major activities focused on properties of silicon tetrachloride which is the source material for several alternate processes. Status and progress are reported for vapor pressure, heat of vaporization, heat capacity, density, surface tension, viscosity, thermal conductivity, heat of formation and Gibb's free energy of formation.

Experimental thermal conductivity values for hydrogen, determined for instrument accuracy evaluation, were in very good agreement with recommended values from the literature. The deviations were only $\pm 2\%$ up to 300°C . Preliminary results for gas phase thermal conductivity are reported for silane (SiH_4) and dichlorosilane (SiH_2Cl_2) in the respective temperature ranges, 25 to 300°C and 25 to 400°C . There have been no previously reported experimental data in the literature for these silicon source materials.

Major efforts were continued on the preliminary process design of the silane process (Union Carbide). Progress and status are reported for key items. The process flow diagram, material balance and energy balance are 100% complete for the revised flowsheet (received Union Carbide). Major process equipment design is 85% complete. Estimate of production labor requirements is 75% complete.

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I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

A. SILICON TETRACHLORIDE PROPERTIES

Analyses were continued during this reporting period for process system properties of silicon source materials under consideration for solar cell grade silicon production.

Major activities focused on properties of silicon tetrachloride in areas of data collection, analysis, estimation and correlation. The status and progress since the last reporting period are summarized below for those properties required in the performance of the chemical engineering analyses of the alternate processes under consideration for solar cell grade silicon:

	<u>Prior</u>	<u>Current</u>
1. Vapor Pressure, P_v	50%	80%
2. Heat of Vaporization, ΔH_v	40%	60%
3. Gas Heat Capacity, C_p^g	60%	80%
4. Liquid Heat Capacity, C_p	25%	45%
5. Density, ρ_L	25%	45%
6. Surface Tension, σ_L	25%	45%
7. Gas Viscosity, η_G	25%	45%
8. Liquid Viscosity, η_L	25%	45%
9. Gas Thermal Conductivity, λ_G	25%	45%
10. Liquid Thermal Conductivity, λ_L	25%	45%
11. Heat of Formation, ΔH_f°	25%	45%
12. Gibb's Free Energy of Formation, ΔG_f°	25%	45%

B. THERMAL CONDUCTIVITY INVESTIGATION

During this reporting period, calibration studies on the thermal conductivity apparatus were completed and the experimental determination of the thermal conductivity of silicon source materials was initiated.

The thermal conductivity of hydrogen was determined in the temperature range 25°C to 400°C. (For this particular determination nitrogen was used as the reference gas rather than helium.) These values were compared to recommended values for thermal conductivity of hydrogen (reference 36) in order to evaluate the accuracy of data obtained on this apparatus (Figure IB-3) for gases of high thermal conductivity. The recommended values used for comparison were those presented in "Thermophysical Properties of Matter", Vol. 3 on Thermal Conductivity (TPRC), and were determined by a critical evaluation of published data. The thermal conductivity values obtained in this study agree with the recommended values to within $\pm 2\%$ up to 300°C and $\pm 5\%$ from 300°C to 400°C.

The thermal conductivity of silane (SiH_4) has been determined between 25°C and 300°C (Table IB-1 and Figure IB-4). Values above 300°C were not determined because above that temperature silane is thermally unstable and begins to deposit silicon. There have been no previously reported experimental data for gaseous thermal conductivity of silane. Estimated values have been determined using a modified Eucken Correlation (ref. 37) and these estimated values agree fairly well with the now available experimental values.

The thermal conductivity of dichlorosilane (SiH_2Cl_2) has been determined between 25°C and 400°C (Table IB-2 and Figure IB-5). There have been no previously reported experimental data for gaseous thermal conductivity of dichlorosilane. Estimated values have been reported (reference 39) in the temperature range 0°C to 70°C which were determined by a Eucken approximation. These estimated values are considerably lower than the experimental values now reported.

Figure IB-3. Apparatus Calibration with Hydrogen

TABLE IB-1

Preliminary Results for Gaseous Thermal Conductivity of Silane

<u>Temperature</u> °C	<u>Gaseous Thermal Conductivity</u>		
	<u>mWcm⁻¹ K⁻¹</u>	<u>cal sec⁻¹cm⁻¹C⁻¹</u>	<u>Btu hr⁻¹ft⁻¹F⁻¹</u>
28	0.234	56.02 X 10 ⁻⁶	13.54X 10 ⁻³
48	0.250	59.78 X 10 ⁻⁶	14.45 X 10 ⁻³
100	0.300	71.73 X 10 ⁻⁶	17.34 X 10 ⁻³
150	0.351	83.96 X 10 ⁻⁶	20.30 X 10 ⁻³
200	0.411	98.28 X 10 ⁻⁶	23.76 X 10 ⁻³
250	0.464	110.83 X 10 ⁻⁶	26.79 X 10 ⁻³
300	0.518	123.91 X 10 ⁻⁶	29.95 X 10 ⁻³

Figure IB-4. Thermal Conductivity of Silane

TABLE IB-2

Preliminary Results for Gaseous Thermal Conductivity of Dichlorosilane

<u>Temperature</u> <u>C°</u>	<u>Gaseous Thermal Conductivity</u>		
	<u>mWcm⁻¹ K⁻¹</u>	<u>Cal sec⁻¹ cm⁻¹ C⁻¹</u>	<u>Btu hr⁻¹ ft⁻¹ F⁻¹</u>
28°	0.102	24.43 X 10 ⁻⁶	5.91 X 10 ⁻³
48°	0.108	25.88 X 10 ⁻⁶	6.26 X 10 ⁻³
100°	0.131	31.19 X 10 ⁻⁶	7.54 X 10 ⁻³
150°	0.151	36.14 X 10 ⁻⁶	8.74 X 10 ⁻³
200°	0.174	41.47 X 10 ⁻⁶	10.02 X 10 ⁻³
250°	0.201	48.02 X 10 ⁻⁶	11.61 X 10 ⁻³
300°	0.226	53.94 X 10 ⁻⁶	13.04 X 10 ⁻³
350°	0.255	60.83 X 10 ⁻⁶	14.70 X 10 ⁻³
394°	0.280	66.82 X 10 ⁻⁶	16.16 X 10 ⁻³

Figure IB-5. Thermal Conductivity of Dichlorosilane

II. CHEMICAL ENGINEERING ANALYSES (TASK 2)

A. SILANE PROCESS (UNION CARBIDE)

Major efforts were continued during this reporting period on the preliminary process design for the silane process (Union Carbide). The status, including progress since the last reporting period for the process design is given below for key guideline items:

	<u>Prior</u>	<u>Current</u>
. Process Flow Diagram	95%	100%
. Material Balance	95%	100%
. Energy Balance	50%	100%
. Property Data	65%	85%
. Equipment Data	60%	85%
. Production Labor	50%	75%

The detailed status sheet is shown in Table IIA-1.0 in order to present the items that make up the preliminary process design. The revised process flowsheet received from Union Carbide is given in Figure IIA-1.0.

The summarized results for the preliminary process design are presented in a tabular format to make it easier to locate items of specific interest. The guide for these tables is given below:

.Base Case Conditions.....	Table IIA-1.1
.Reaction Chemistry.....	Table IIA-1.2
.Redistribution Equilibrium.....	Figure IIA-1.1
.Raw Materials Requirements.....	Table IIA-1.3
.Utility Requirements.....	Table IIA-1.4

The list of major process equipment required in the plant to produce silane is being compiled in current activities. Upon completion of the equipment compilation list, the production labor requirements will be determined.

TABLE IIA-10 CHEMICAL ENGINEERING ANALYSES:
PRELIMINARY PROCESS DESIGN ACTIVITIES FOR SILANE PROCESS (UNION CARBIDE)

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

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

TABLE IIA-1.1

BASE CASE CONDITIONS FOR SILANE PROCESS (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)

TABLE IIA-1.2

REACTION CHEMISTRY FOR SILANE PROCESS (UNION CARBIDE)

1. Hydrogenation Reaction

2. Trichlorosilane Redistribution Reaction

3. Dichlorosilane Redistribution Reaction

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

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

TABLE IIA-1.3

RAW MATERIAL REQUIREMENTS FOR SILANE PROCESS (UNION CARBIDE)

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

TABLE IIA-1.4

UTILITY REQUIREMENTS FOR SILANE PROCESS (UNION CARBIDE)

<u>Utility/Function</u>	<u>Requirements/lb of Silane Product</u>
1. Electricity	.25 KW-HR
1. All pump and compressor motors (24)	(.25)
2. Stea. 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
1. #1 Distillation Column Condenser	(146.12)
2. #2 Distillation Column Condenser	(22.09)
4. Process Water (90°F)	8.22
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)

TABLE IIA-1.4 (Continued)

9.	Refrigerant (-30°F)		30788.0 BTU
1.	TCS Reactor Recycle Gas Condenser	(30788.0)	
10.	Refrigerant (-40°F)		369.0 BTU
1.	#2 Redistribution Reactor Condensate Receiver	(192.2)	
2.	Silane Product Storage	(176.8)	
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 Regenerate Char. Adsorbers	(70.95)	
5.	TCS Reactor	(1.491×10^3)	
13.	Nitrogen		5.54 SCF
1.	Regenerate Charcoal Adsorbers	(5.54)	

B. OTHER PROCESSES

Technical progress reports are being received and screened for the following processes:

1. Battelle Process ($2n/SiCl_4$)
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 ($SiCl_4/H$ atom)

These technical progress reports are being continuously monitored for technical information pertinent to chemical engineering analysis of the alternate processes under consideration for solar cell grade silicon.

III. SUMMARY - CONCLUSIONS

Based on major activities accomplished during this reporting period, the following summary-conclusions are made:

1. Task 1

Analyses of process system properties were continued for silicon source materials under consideration for solar cell grade silicon production.

Major activities focused on properties of silicon which is the source material for several alternate processes. Properties analysis for vapor pressure, heat of vaporization, gas heat capacity, liquid heat capacity and density is about 70% complete.

Preliminary calibration and evaluation of the instrument assembled to measure thermal conductivity of gases was completed. The experimentally determined values for the thermal conductivity of hydrogen are in excellent agreement with recommended values from the literature.

In addition, the experimental determination of thermal conductivity for silicon source materials was initiated. Preliminary gas thermal conductivity results for silane (SiH_4) and dichlorosilane (SiH_2Cl_2) are reported in the respective temperature ranges of 25 to 300° C and 25 to 400°C. There have been no previously reported experimental data in the literature for gas phase thermal conductivity of these silicon source materials.

2. Task 2

Major efforts were continued on the preliminary process design of the silane process (Union Carbide). The process flow diagram, material balance and energy balance are 100% complete for the revised flowsheet (received from Union Carbide). Major process equipment design is 85% complete. Estimate of production labor requirements is 75% complete.

IV. 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 collection for silicon source materials will be continued with emphasis on halogenated silanes.

2. Task 2

Complete preliminary process design of the silane process (Union Carbide).

3. Task 3

Initiate cost analysis of the silane process (Union Carbide).

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MILESTONE CHART

TASK	1975			1976												1977												1978											
	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S			
1. Analyses of Process																																							
<u>System Properties</u>																																							
1. Prel. Data Collection																																							
2. Data Analysis																																							
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