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PROCESS FEASIBILITY STUDY IN SUPPORT OF
SILICON MATERIAL TASK I

Quarterly Technical Progress Report (XIII) for September—December 1978

By
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December 1978

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U.S. Department of Energy

Solar Energy

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QUARTERLY TECHNICAL PROGRESS REPORT (XIII)

Issue Date: December 1978

Reporting Period: September-December, 1978

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Approval Signature

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ABSTRACT

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

Analyses of process system properties was continued for materials involved in the alternate processes under consideration for solar cell grade silicon. The following property data are reported for trichlorosilane: critical constants, vapor pressure, heat of vaporization, gas heat capacity, liquid heat capacity, density, surface tension, viscosity, thermal conductivity, heat of formation and Gibb's free energy of formation.

Work has continued on the measurement of gas viscosity values of silicon source materials with emphasis on the modification of the apparatus to allow measurements up to higher temperatures and to develop the capability of handling pyrophoric materials such as silane. In additional activities, gas phase viscosity values for silicon tetrafluoride between 40°C and 200°C were experimentally determined. These values are shown to be in close agreement with a set of previously reported values in the same temperature range.

Major efforts were expended on completion of the preliminary economic analysis of the UCC Silane Process (Union Carbide Corporation Silane Process). Cost, sensitivity and profitability analysis results are presented based on a preliminary process design of a plant to produce 1,000 metric tons/year of silicon by the revised process. Fixed capital investment estimate for the plant is \$9.19 million (1975 dollars). Product cost without profit is 6.90 \$/kg of silicon (1975 dollars). The profitability results indicate a sales price of 9.88 \$/kg of silicon (1975 dollars) at a 20% DCF return on investment after taxes.

Major chemical engineering activities were started on the modified BCL process which includes additional data and engineering modification. The preliminary process design was initiated including specific base case conditions, reaction chemistry, process flow diagram and material balance for a plant to produce 1,000 MT/yr of solar cell grade silicon.

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I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

A. TRICHLOROSILANE PROPERTIES

During this reporting period, analysis of process system properties was continued for silicon source materials under consideration for solar cell grade silicon production. Primary activities focused on property data for trichlorosilane. Trichlorosilane is the feed source material in the conventional process for silicon and is involved in the processing operations of the UCC Silane Process for solar cell grade silicon.

Critical Properties (Table IA-1)

Experimental data for the critical temperature and critical volume are available (G33, G28) from a Russian investigation of orthobaric densities and critical parameters. The critical pressure for trichlorosilane was estimated by Lydersen method (G62, G67):

$$P_c = \frac{M}{(\sum \Delta p + 0.34)^2} \quad (\text{IA-1})$$

where P_c is critical pressure (atm), M is molecular weight (gr/gr-mol), and Δp is critical property increments for atoms making up the molecule. This method produced only 1.6% error when compared with the experimentally determined critical pressure of silicon tetrachloride.

The critical compressibility factor, Z_c , was calculated from its definition:

$$Z_c = \frac{P_c V_c}{R T_c} \quad (\text{IA-2})$$

Vapor Pressure (Figure IA-1)

Observed vapor pressure data from several sources (G15, G29, G44, G61) for trichlorosilane are in general agreement from -85°C to just above the boiling point. The experimental data were extended to cover the entire liquid phase using the YSSP correlation relation (G63):

$$\log P_v = A + \frac{B}{T} + C \log T + DT \quad (\text{IA-3})$$

where

P_v = vapor pressure of saturated liquid, mm of Hg

A, B, C, D = correlation constants for chemical compound

T = temperature, °K

The deviation of experimental and correlation results was small at 0.8% error for the 36 available data points.

Heat of Vaporization (Figure IA-2)

Heat of vaporization data for trichlorosilane are available only at the boiling point (G25, G18, G38, G46, G27). Using the known value at the boiling point, Watson's correlation (G62) was used to extend the heat of vaporization over the entire liquid phase:

$$\Delta H_v = \Delta H_{v1} \left[\frac{T_c - T}{T_c - T_1} \right]^n \quad (\text{IA-4})$$

where $n = .38$ and ΔH_{v1} applies at the boiling point (T_1).

Heat Capacity (Figures IA-3 and IA-4)

Heat capacity of the ideal gas at low pressure has been calculated by various Russian (G23, G25, G45, G11), American (G53, G56) and other (G6, G30) workers. The values, taken from various structural and spectral data, are in close agreement. The JANAF values (G53) were selected.

The liquid heat capacity of trichlorosilane is reported to be .23 between 25 and 60°C (G19, G46). The values are extended over all liquid temperatures by the relationship:

$$\text{Heat Capacity} \times \text{Density} = \text{Constant} \quad (\text{IA-5})$$

The constant, C., was estimated to be 0.298.

Testing of this relationship with available data for silicon tetrachloride produced an average deviation of 4%.

Liquid Density (Figure IA-5)

Liquid density data for trichlorosilane are available from -10°C to the critical point (G33, G32, G61, G12, G26). The experimental data was extrapolated to the melting point by use of the Yaws-Shah relationship (G63) for saturated liquid:

$$\rho_L = AB^{-(1-T_r)^{2/7}} \quad (\text{IA-6})$$

where $A = .4856$ and $B = .2618$. Correlation values and experimental results were in close agreement. The deviation was less than 1% for the 31 published data points from several independent sources.

Surface Tension (Figure IA-6)

Data for the surface tension of trichlorosilane are available from 0°C to 40°C (G32, G26). These data were extended using the Othmer relations (G62):

$$\sigma = \sigma_1 \left[\frac{T_c - T}{T_c - T_1} \right]^n \quad (\text{IA-7})$$

where σ_1 = surface tension at T_1 , dynes/cm, and n = the correlation parameter, 1.2. The other parameters have their usual meaning. Deviations between data and correlation values were 3% or less, largely due to the deviations between reported experimental values.

Viscosity (Figures IA-7 and IA-8)

Data for the gas viscosity of trichlorosilane were available only at 0°C and at boiling point (G25). The values at higher temperatures were estimated using the modified and revised corresponding-state method of Thodos and Yoon (G67, G68):

$$\eta_G \xi = 4.610 T_r^{0.618} - 2.04 e^{-0.449 T_r} + 1.94 e^{-4.058 T_r} + 0.1 \quad (\text{IA-8})$$

where η_G = viscosity, $\xi = T_c^{1/6} M^{-1/2} P_c^{-2/3}$, and T_r is the reduced temperature. The percentage error was less than .4%. Testing with silicon tetrachloride gave good agreement of correlation and experimental results (16 data points produced a 2% deviation).

Liquid viscosity data for trichlorosilane are available from -7°C to 60°C (G32, G26, G19, G25, G46). At low temperatures (from the boiling to the melting point), values were estimated using the $\log \eta_L$ vs $1/T$ linear relationship. At high temperatures (up to the critical point), the Stiel and Thodos correlation was used with $\mu_L \xi = f(Z_c, T_r)$ where $f(Z_c, T_r)$ is given as a generalized liquid viscosity correlation (G62). The percentage error with the available experimental data was about 2%.

Thermal Conductivity (Figures IA-9 and IA-10)

The gaseous thermal conductivity of trichlorosilane has recently been reported from 46°C to 350°C (G66). The experimental values were extended using a modified form of the Mistic and Thodos correlation (G63, G67):

$$\lambda_G = C_p / \gamma (10^{-6}) (14.52 T_r - 5.14)^n \quad (\text{IA-9})$$

where $n = .71$. The average absolute percentage error was 1.5%.

Liquid thermal conductivity data for trichlorosilane are not available. Using the estimation method of Sheffy and Johnson (G62):

$$\lambda_L = \{ (4.66) (10^{-3}) [1 - .00126 (T - T_m)] / T_m^{.216} M^{.300} \} \quad (\text{IA-10})$$

$\lambda_L = 2.783 \times 10^{-4}$ cal/cm x sec x °K was derived for the value at 60°C.

Using the Pachaiyappan-Vaidyanathan method of estimation (G64):

$$\lambda_L = 8.84 \times 10^{-4} C_p \rho_L \quad (\text{IA-11})$$

the value of 2.64×10^{-4} ca./cm x sec x °K was derived for 60°C.

These estimation methods produced errors of 16% and 17.5%, respectively, on the one published value for SiCl_4 ; and hence, should be taken to represent only an order of magnitude estimate. The estimate was extended over the entire liquid range using a modification of the Stiel and Thodos method (G62, G63):

$$\lambda_L = \frac{f(\rho_r)}{\gamma_c^5} + \lambda_G \quad (\text{IA-12})$$

Heat and Free Energy of Formation (Figures IA-11 and IA-12)

Values of the heat (ΔH_f) and Gibb's free energy of formation (ΔG_f) for the ideal gas are available from various Russian (G11, G45), American (G53) and other (G6, G30) sources and are in close agreement. The American values were selected.

TABLE IA-1

Critical Constants and Physical Properties of Trichlorosilane

<u>Identification</u>	<u>Trichlorosilane</u>
Formula	SiHCl_3
State (std. cond.)	liquid
Molecular Weight, M	135.453
Boiling Point, T_b , °C	31.8
Melting Point, T_m , °C	-126.6
Critical Temp., T_c , °C	206
Critical Pressure, P_c , atm	40.01*
Critical Volume, V_c , $\text{cm}^3/\text{gr mol}$	268
Critical Compressibility Factor, Z_c	.273*
Critical Density, ρ_c , gr/cm^3	.505
Acentric Factor (Ω)	.188*

*Estimated

Figure IA-1. Vapor Pressure vs Temperature for Trichlorosilane

Figure IA-2. Heat of Vaporization vs Temperature for Trichlorosilane

Figure IA-3. Gas Heat Capacity vs Temperature for Trichlorosilane

Figure IA-4. Liquid Heat Capacity vs Temperature for Trichlorosilane

Figure IA-5. Liquid Density vs Temperature for Trichlorosilane

Figure IA-6--Surface Tension vs Temperature for Trichlorosilane

Figure IA-7--Gas Viscosity vs Temperature for Trichlorosilane

Figure IA-8--Liquid Viscosity vs Temperature for Trichlorosilane

Figure IA-9--Gas Thermal Conductivity vs Temperature for Trichlorosilane

Figure IA-10--Liquid Thermal Conductivity vs Temperature for Trichlorosilane

Figure IA-11-Heat of Formation vs Temperature for Trichlorosilane

Figure IA-12--Free Energy of Formation vs Temperature for Trichlorosilane

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B. VISCOSITY INVESTIGATION

Work has been continued on the experimental determination of gas viscosity of silicon source materials. During this reporting period work has been directed toward increasing the temperature range of the measurements up to approximately 350°C. This has involved modifying the apparatus used at the lower temperatures (below 200°C) and installing and calibrating additional equipment which will allow accurate temperature control in the higher temperature range. Also some modification of the apparatus was necessary in order to be able to safely handle pyrophoric materials such as silane.

The viscosity of tetrafluorosilane (SiF_4) has been determined between 40°C and 200°C (Table IB-11 and Figure IB-17). There have been two previous reports of experimentally determined viscosity values for tetrafluorosilane. Ellis and Raw (references 45) reported values between 23°C and 134°C and McCoubrey and Singh (reference 43) reported values between 18°C and 190°C. The values of McCoubrey and Singh were in close agreement to the values reported in this study with less than 3% deviation through the whole temperature range. The values of Ellis and Raw were lower than the values reported in this study by as much as 7%.

Figure IB-17. Viscosity of Gaseous Tetrafluorosilane

TABLE IB-11

Gaseous Viscosity of Tetrafluorosilane

Temperature °C	Viscosity		
	micropoise	N s m^{-2}	$\frac{\text{lb}_m \text{ s}^{-1}}{\text{ft}^{-1}}$
40	169.5	16.96×10^{-6}	11.40×10^{-6}
60	180.7	18.07×10^{-6}	12.14×10^{-6}
100	191.7	19.17×10^{-6}	12.88×10^{-6}
150	208.3	20.83×10^{-6}	14.00×10^{-6}
200	231.2	23.12×10^{-6}	15.54×10^{-6}

II. CHEMICAL ENGINEERING ANALYSES (TASK 2)

A. BCL PROCESS (BATTELLE COLUMBUS LABORATORIES)

Initial chemical engineering - economic analysis results for the BCL process were reported in 1976. These results were based on preliminary laboratory data and process flowsheet. Additional laboratory data and process modifications have been identified for the BCL process since reporting the initial results.

The chemical engineering analysis of the BCL process was initiated during this reporting period. These analyses involves the utilization of the additional data and engineering modifications for the process.

The status and progress for the preliminary process design including the additional data are shown below for key items:

	<u>Prior</u>	<u>Current</u>
.Base Case Conditions	0%	40%
.Reaction Chemistry	0%	40%
.Process Flow Diagram	0%	30%
.Material Balance	0%	20%

The detailed status sheet is shown in Table IIA-1.0, and is representative of the various subitems that make up the preliminary design activity.

TABLE IIA-1.0 CHEMICAL ENGINEERING ANALYSES:
PRELIMINARY PROCESS DESIGN ACTIVITIES FOR BCL PROCESS (BATTELLE COLUMBUS LABORATORIES)

<u>Prel. Process Design Activity</u>	<u>Status</u>	<u>Prel. Process Design Activity</u>	<u>Status</u>
1. Specify Base Case Conditions	0	7. Equipment Design Calculations	0
1. Plant Size	0	1. Storage Vessels	0
2. Product Specifics	0	2. Unit Operations Equipment	0
3. Additional Conditions	0	3. Process Data (P, T, rate, etc.)	0
2. Define Reaction Chemistry	0	4. Additional	0
1. Reactants, Products	0	8. List of Major Process Equipment	0
2. Equilibrium	0	1. Size	0
3. Process Flow Diagram	0	2. Type	0
1. Flow Sequence, Unit Operations	0	3. Materials of Construction	0
2. Process Conditions (T, P, etc.)	0	8a. Major Technical Factors	0
3. Environmental	0	(Potential Problem Areas)	0
4. Company Interaction	0	1. Materials Compatibility	0
(Technology Exchange)		2. Process Conditions Limitations	0
4. Material Balance Calculations	0	3. Additional	0
1. Raw Materials	0	9. Production Labor Requirements	0
2. Products	0	1. Process Technology	0
3. By-Products	0	2. Production Volume	0
5. Energy Balance Calculations	0	10. Forward for Economic Analysis	0
1. Heating	0		
2. Cooling	0		
3. Additional	0		
6. Property Data	0		
1. Physical	0	0 Plan	
2. Thermodynamic	0	0 In Progress	
3. Additional	0	0 Complete	

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. Motorola Process ($\text{SiF}_4/\text{SiF}_2$)
2. Westinghouse Process (Na/SiCl_4)
3. Dow Process (C/SiO_2)
4. SRI Process (Na/SiF_4)
5. Texas Instruments (SiHCl_3)
6. J. C. Schumacher Co. (SiBr_4)
7. Aerochem ($\text{Na}/\text{SiCl}_4/\text{SiF}_4$)

III. ECONOMIC ANALYSES (TASK 3)

A. UCC Silane Process (Union Carbide Corporation)

Major efforts during this reporting period focused on completion of the preliminary economic analysis of the UCC Silane Process (Union Carbide Corporation Silane Process). For CASE C, the revised process involves a more optimum arrangement of major process equipment, raw materials and operating conditions.

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

CASE C - Revised Process

1. Process.....	UCC Silane Process
2. Plant Size.....	1,000 Metric Tons/Year
3. Plant Product.....	Solar Cell Grade Silicon
4. Product Form.....	Liquid Phase Silicon
5. Cost Basis.....	1975 Dollars
6. Plant Investment (Fixed Capital).....	\$9,190,000
7. Product Cost (No Profit).....	\$6.90/KG of Silicon

The detailed results from the completed preliminary economic analysis are presented in a tabular format for CASE C. The guide for the tabular format is given below:

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

CASE C

Table IIIA-1.0C
ECONOMIC ANALYSES:
PRELIMINARY ECONOMIC ANALYSIS ACTIVITIES FOR SILANE PROCESS
CASE C (UNION CARBIDE)

<u>Prel. Process Economic Activity</u>	<u>Status</u>	<u>Prel. Process Economic Activity</u>	<u>Status</u>
1. Process Design Inputs	●	6. Production Labor Costs	●
1. Raw Material Requirements	●	1. Base Cost Per Man Hour	●
2. Utility Requirements	●	2. Cost/Kg Silicon Per Area	●
3. Equipment List	●	3. Total Cost/Kg Silicon	●
4. Labor Requirements	●		
		7. Estimation of Plant Investment	●
2. Specify Base Case Conditions	●	1. Battery Limits Direct Costs	●
1. Base Year for Costs	●	2. Other Direct Costs	●
2. Appropriate Indices for Costs	●	3. Indirect Costs	●
3. Additional	●	4. Contingency	●
		5. Total Plant Investment	●
3. Raw Material Costs	●	(Fixed Capital)	
1. Base Cost/Lb. of Material	●		
2. Material Cost/Kg of Silicon	●	8. Estimation of Total Product Cost	●
3. Total Cost/Kg of Silicon	●	1. Direct Manufacturing Cost	●
		2. Indirect Manufacturing Cost	●
4. Utility Costs	●	3. Plant Overhead	●
1. Base Cost for Each Utility	●	4. By-Product Credit	●
2. Utility Cost/Kg of Silicon	●	5. General Expenses	●
3. Total Cost/Kg of Silicon	●	6. Total Cost of Product	●
5. Major Process Equipment Costs	●		
1. Individual Equipment Cost	●		
2. Cost Index Adjustment	●		
		○ Plan	
		● In Progress	
		● Complete	

TABLE IIIA-1.1C

PROCESS DESIGN INPUTS FOR
SILANE PROCESS - CASE C. (UNION CARBIDE)

1. Raw Material Requirements
 - M.G. Silicon, silicon tetrachloride, hydrogen, copper catalyst, lime
 - see table for "Raw Material Cost"
2. Utility
 - electrical, steam, cooling water, etc.
 - see table for "Utility Cost"
3. Equipment List
 - 93 pieces of major process equipment
 - process vessels, heat exchangers, reactor, etc.
4. Labor Requirements
 - production labor for purification, vaporization, product handling, etc.
 - see table for "Production Labor Cost"

CASE C

TABLE IIIA-1.2C

BASE CASE CONDITIONS FOR
SILANE RPROCESS - CASE C (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
- Raw Material Cost Index for Industrial Chemicals
- 1975 Cost Index Value = 206.9 (Wholesale Price Index, Producer Price Index)

4. Labor Cost

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

CASE C

TABLE IIIA-1.3C

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

<u>Raw Material</u>	<u>Requirement lb/KG of Si</u>	<u>\$/lb of Material</u>	<u>Cost \$/KG of Si</u>
1. M.G. Silicon (Si)	2.60	0.535	1.391
2. Silicon Tetrachloride (SiCl ₄ , make-up)	2.76	0.135	0.373
3. Liquid Hydrogen (H ₂ , make-up)	0.032	1.84	0.059
4. Copper Catalyst (Cu)	0.051	0.922	0.047
5. Hydrate Lime (Ca(OH) ₂)	2.43	0.015 (33.2\$/ton)	0.036
		TOTAL	1.906

Note:

1. Costs are 1975 Dollars

CASE C

TABLE IIIA-1.4C

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

<u>Utility</u>	<u>Requirement/KG of Silicon</u>	<u>Cost of Utility</u>	<u>Cost \$/KG of Silicon</u>
1. Electricity	3.050 KW	0.0324 \$/KW-HR	0.0988
2. Steam	172.200 lbs.	1.35 \$/Mlb	0.2325
3. Cooling Water	525.000 gallons	0.09 \$/Mgal	0.0473
4. Process Water	0.0709 gallons	0.405 \$/Mgal	0.0001
5. Refrigerant	968.000 Btu	10.50 \$/MM Btu	0.0102
6. Fuel	27,100.00 Btu	1.40 \$/MM Btu	<u>0.0379</u>
		TOTAL	0.4268

Note:

1. Costs are 1975 Dollars

CASE C

TABLE IIIA-1.5C

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

<u>Equipment</u>	<u>Purchased Cost, \$1000</u>
1. (D-01) Crude TCS/STC Stripping Column	5.5
2. (D-02) TCS/STC Distillation Column	32.6
3. (D-03) DCS/TCS Distillation Column	61.6
4. (D-04) Silane Distillation Column	50.3
5. (R-01) Hydrogenation Reactor	82.6
6. (R-02) DCS Redistribution Reactor	19.2
7. (R-03) TCS Redistribution Reactor	17.3
8-9. (R-04) Sludge Neutralization Reactor	10.3
10. (H-01) Liquid H ₂ Vaporizer (Provided by Vendor)	-----
11. (H-02) STC Cooler	26.4
12. (H-03) Quench Condenser	22.6
13. (H-04) Recycle STC Vaporizer	3.3
14. (H-05) Recycle STC Superheater	35.0
15. (H-06) Recycle H ₂ Heater	10.8
16. (H-07) Stripper Reboiler	11.5
17. (H-08) Stripper Condenser	1.4
18. (H-09) TCS/STC Reboiler	8.6
19. (H-10) TCS/STC Condenser	44.5
20. (H-11) DCS/TCS Reboiler	8.2
21. (H-12) DCS/TCS Condenser	16.2
22. (H-13) DCS Cooler	1.5
23. (H-14) TCS Cooler	3.0

CASE C

TABLE IIIA-1.5C (Continued)

24.	(H-15) Silane Reboiler	1.3
25.	(H-16) Silane Condenser	2.6
26.	(H-17) Silane Vaporizer/Superheater	2.4
27.	(H-18) Pyrolysis Hydrogen Cooler	4.1
28.	(H-19) First Stage H ₂ Intercooler	3.6
29.	(H-20) Second Stage H ₂ Intercooler	3.6
30.	(C-01) Pneumatic Conveying Fan	1.6
31.	(C-02) Recycle H ₂ Blower	4.7
32.	(C-03) First Stage H ₂ Compressor	9.7
33.	(C-04) Second Stage H ₂ Compressor	9.7
34.	(C-05) Third Stage H ₂ Compressor	9.7
35.	(P-01) Quench Contactor Pump	2.4
36.	(P-03) Recycle STC Pump	15.0
37.	(P-04) TCS Distillate Pump	19.8
38.	(P-05) DCS Distillate Pump	11.3
39.	(P-06) Lime Tank Pump	2.0
40.	(T-01) Crude TCS/STC Storage Tank	39.0
41.	(T-02) STC Storage Tank	17.0
42.	(T-03) Liquid H ₂ Storage (Provided By Vendor)	<hr/>
43.	(T-04) Waste Settler Tank	27.0
44.	(T-05) Waste Chloride Tank	1.8
45.	(T-06) Quench Condenser Receiver	8.8
46.	(T-07) Recycle H ₂ Receiver	7.2

CASE C

TABLE IIIA-1.5C (Continued)

47.	(T-08) Stripper Reflux pot	1.2
48.	(T-09) TCS/STC Reflux pot	6.1
49.	(T-10) DCS/TCS Reflux pot	11.2
50.	(T-11) A, B Silane Shift Tank (two)	20.6 ea.
51.	(T-13) Pyrolysis H ₂ Receiver	7.9
52.	(T-14) Lime Make-up Tank	5.7
53.	(T-15) Sludge Pump Tank	11.5
54.	(B-01) M. G. Silicon Storage Hopper	12.2
55-56.	(B-04) Pyrolysis Dust Bin	1.7
57.	(F-01) Crude TCS/STC Filter	0.7
58.	(F-02) Waste Hydroxide Filter	5.0
59.	(F-03) Pyrolysis H ₂ Filter	0.7
60.	(F-04) M. G. Silicon Unloading Filter	1.6
61.	(S-01) M. G. Silicon Unloading Cyclone	1.4
62.	(S-02) Double Shell Blender	13.0
63.	(S-03) M. G. Silicon Load Hopper	5.8
64.	(U-01) Quench Contactor Ejector	1.3
65.	(U-02) Lime Tank Agitator	1.3
66.	(U-03) Vent Gas Combustor	6.3
67.	(U-04) Vent Gas Ejector	1.3
68.	(R-05) Silane Pyrolysis Reactor (six)	46.8 ea.
69.	(X-01) Melters (six)	53.0 ea.
70.	(B-05) Powder Hoppers (two)	19.9 ea.
71.	(X-02) Hydrogen Cooler	4.1

CASE C

TABLE IIIA-1.5C (Continued)

72.	(X-03) Hydrogen Blower	2.5
73.	(X-04) Dust Filter	0.8
74.	(X-05) Star Valve (six)	1.2 ea.
75.	(X-06) Conveyor	8.3
76.	(X-07) Drum Loader	<u>16.6</u>
TOTAL		1481.9

CASE C

TABLE IIIA-1.6C

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

<u>Section/ Unit Operation</u>	<u>Skilled Labor Man-Hrs/KG of Si</u>	<u>Semiskilled Labor Man-Hrs/KG of Si</u>	<u>Cost \$/KG of Si</u>
1. Hydrogenation	0.00745	0.000745	0.0879
2. Silane	0.02230	-----	0.1539
3. Pyrolysis	0.02980	-----	0.2056
4. Waste Treatment	0.00745	-----	0.0514
5. Hydrogen Compression	0.00745	-----	0.0514
		TOTAL	0.5502

Note:

1. Costs are 1975 Dollars
2. Based on labor costs of \$6.90 skilled, \$4.90 semiskilled.

CASE C

TABLE IIIA-1.7C

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

	<u>Investment</u> <u>\$1000</u>
1. DIRECT PLANT INVESTMENT COSTS	
1. Major Process Equipment Cost	1,481.9
2. Installation of Major Process Equipment	637.2
3. Process Piping, Installed	1,096.6
4. Instrumentation, Installed	281.6
5. Electrical, Installed	148.2
6. Process Buildings, Installed	148.2
1a. SUBTOTAL FOR DIRECT PLANT INVESTMENT COSTS (PRIMARILY BATTERY LIMIT FACILITIES)	3,793.7
2. OTHER DIRECT PLANT INVESTMENT COSTS	
1. Utilities, Installed	711.3
2. General Services, Site Development, Fire Protection, etc.	177.8
3. General Buildings, Offices, Shops, etc.	207.5
4. Receiving, Shipping Facilities	311.2
2a. SUBTOTAL FOR OTHER DIRECT PLANT INVESTMENT COSTS (PRIMARILY OFFSITE FACILITIES OUTSIDE BATTERY LIMITS)	1,407.8
3. TOTAL DIRECT PLANT INVESTMENT COST, 1a + 2a	5,201.5
4. INDIRECT PLANT INVESTMENT COSTS	
1. Engineering, Overhead, etc.	815.0
2. Normal Cont. for Floods, Strikes, etc.	1,052.1
4a. TOTAL INDIRECT PLANT INVESTMENT COST	1,867.1
5. TOTAL DIRECT AND INDIRECT PLANT INVESTMENT COST, 3 + 4a	7,068.6
6. OVERALL CONTINGENCY, % of 5	2,120.6
7. FIXED CAPITAL INVESTMENT FOR PLANT, 5 + 6	9,189.2
8. WORKING CAPITAL INVESTMENT FOR PLANT	
9. TOTAL PLANT INVESTMENT, 7 + 8	

CASE C

TABLE IIIA-1.8C

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

	<u>\$/KG of Si</u>
1. Direct Manufacturing Cost (Direct Charges)	
1. Raw Materials	1.906
2. Direct Operating Labor	0.550
3. Utilities	0.427
4. Supervision and Clerical	0.083
5. Maintenance and Repairs	0.919
6. Operating Supplies	0.184
7. Laboratory Charge	0.083
2. Indirect Manufacturing Cost (Fixed Charges)	
1. Depreciation	0.919
2. Local Taxes	0.184
3. Insurance	0.092
3. Plant Overhead	0.656
4. By-Product Credit	-----
4a. Total Manufacturing Cost, 1 + 2 + 3 + 4	6.003
5. General Expenses	
1. Administration	0.360
2. Distribution and Sales	0.360
3. Research and Development	0.180
6. Total Cost of Product, 4a + 5	<u>6.903</u>

Note:

Costs are 1975 Dollars

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. Motorola Process ($\text{SiF}_4/\text{SiCl}_4$)
2. Westinghouse Process (Na/SiCl_4)
3. Dow Process (C/SiO_2)
4. SRI Process (Na/SiF_4)
5. AeroChem Process ($\text{Na}/\text{SiCl}_4/\text{SiF}_4$)
6. J. C. Schumacher Co. (SiBr_4)
7. Texas Instruments (SiHCl_3)

IV. SUMMARY - CONCLUSIONS

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

1. Task 1

Analyses of process system properties was continued for materials involved in the alternate processes under consideration for solar cell grade silicon. The following property data are reported for trichlorosilane: critical constants, vapor pressure, heat of vaporization, gas heat capacity, liquid heat capacity, density, surface tension, viscosity, thermal conductivity, heat of formation and Gibbs free energy of formation.

Apparatus modification has been completed which will allow gas viscosity measurements to be made at temperatures up to 350°C. Experimental determination of gas phase viscosity values of silicon source materials was continued. Values for the viscosity of tetrafluorosilane between 40°C and 200°C are reported.

2. Task 2

Major efforts were started on the BCL process which has been modified and improved by Battelle Columbus Laboratory since the last evaluation. The preliminary process design was initiated including specific base case conditions, reaction chemistry, process flow diagram and material balance. The silicon production rate is 1,000 MT/yr.

3. Task 3

Major efforts were expended on completion of the preliminary economic analysis of the UCC Silane Process (Union Carbide Corporation Silane Process). Cost, sensitivity and profitability analysis results are presented based on a preliminary process design of a plant to produce 1,000 metric tons/year of silicon by the revised process. Fixed capital investment estimate for the plant is \$9.19 million (1975 dollars). Product cost without profit is 6.90 \$/kg of silicon (1975 dollars). The profitability results indicate a sales price of 9.88 \$/kg of silicon (1975 dollars) at a 20% DCF return on investment after taxes.

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.

Measurement of gas phase viscosity values for silicon source materials will be continued.

2. Task 2

Continue chemical engineering analysis of candidate processes under consideration for solar cell grade silicon.

3. Task 3

Perform cost analysis of processes as results issue from chemical engineering analysis.

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APPENDIX

COST ANALYSIS OF UCC

SILANE PROCESS

COST ANALYSIS OF UCC SILANE PROCESS

ABSTRACT

Cost, sensitivity and profitability analysis results are presented based on a preliminary process design of a plant to produce 1000 metric tons/year of silicon by the UCC Silane Process (Union Carbide Corporation Silane Process). The profitability results indicate a sales price of 9.88 \$/Kg of silicon (1975 dollars) at 20% DCF return on investment after taxes. These results indicate good potential for meeting the 1975 LSA cost goal of \$10 per Kg.

INTRODUCTION

The Low-Cost Solar Array (LSA) Project at Jet Propulsion Laboratory (JPL) in Pasadena, California is being funded by the Department of Energy (DOE) for effective cost reduction in the production of silicon for solar cells. An important overall objective of the project is to reduce the cost of electricity produced with solar cells from today's \$10-25 per watt (Peak) to \$0.50 per watt by 1986. Cost reductions in the manufacture of solar cells are allocated to major tasks encompassing everything from initial silicon production to final array assembly. The cost goal for the silicon material that goes into solar cells is about \$10 per kg of material (1975 dollars).

Semiconductor grade silicon which is currently produced via the conventional Siemens process by several major manufacturers (Dow-Corning, Monsanto, Motorola, Texas Instruments and Great Western) in the United States is too expensive to meet the silicon material cost goal. Lower cost silicon is needed for solar cells. New technologies that depart from the conventional process are being developed by several concerns to produce a less costly silicon material. This article presents results for the UCC Silane Process (Union Carbide Corporation Silane Process). Other processes now being developed will be reported in forthcoming articles.

PROCESS DESCRIPTION / PLANT DESIGN

The process flowsheet for the UCC Silane Process which consists of several major processing operations is shown in Figure 1.

Hydrogen, silicon tetrachloride, and metallurgical grade silicon are feed to the hydrogenation reactor (fluidized bed, 500°C, 515 psia, copper catalyst) to produce a mixture of chlorosilanes. The mixture of chlorosilanes from the hydrogenation reaction is condensed and subjected to a several state distillation to separate components and remove impurities.

Initially, the condensed liquid mixture is sent to D-01 stripper (90 psia) to remove inert gases and volatile impurities. The stripper bottoms go to D-02 distillation (55 psia) which separates TCS (trichlorosilane) and STC (silicon tetrachloride). The TCS redistribution reactor (liquid phase, 85 psia, 140°F, catalyst) is used to produce DCS (dichlorosilane). The separation of DCS and TCS is achieved in D-03 distillation (220 psia). The overhead goes to DCS redistribution reactor (liquid phase, 510 psia, 140°F, catalyst) to produce silane (SiH₄). The silane is purified by separation from trace impurities (such as B₂H₆) by D-04 distillation (355 psia).

The purified silane is used to produce silicon powder via the pyrolysis reaction:

The hydrogen from the reaction is compressed and recycled to the hydrogenation reactor. The silicon powder from the pyrolysis is consolidated to provide the molten silicon product.

Table I summarizes the reaction chemistry involved in these processing operations.

Figure 1. PROCESS FLOWSHEET FOR UCC SILANE PROCESS

TABLE I

REACTION CHEMISTRY FOR UCC SILANE PROCESS

1. Hydrogenation Reaction 2. Trichlorosilane Redistribution Reaction 3. Dichlorosilane Redistribution Reaction 4. Waste Treatment (representative) 5. Silane Pyrolysis Reaction Note:

1. Reaction 1 product contains H_2 , HCl , SiCl_4 , SiHCl_3 , SiH_2Cl_2 (trace), other trace chlorides
2. Reaction 2 product contains SiCl_3 , SiCl_4 , SiH_2Cl_2 , SiH_3Cl
3. Reaction 3 product contains SiH_2Cl_2 , SiHCl_3 , SiCl_4 , SiH_3Cl , SiH_4

A process design was performed to obtain data for a cost analysis of a plant to produce silicon by this new technology. The design was based on a plant to produce 1000 metric tons/year of silicon via the UCC Silane Process. Table II presents details for the plant design.

The process design provided detailed data for raw materials, utilities, major process equipment and production labor requirements which are necessary for polysilicon production.

PRELIMINARY COST/SENSITIVITY ANALYSIS

The preliminary cost and sensitivity analysis was performed using the detailed data from the process plant design. Details for the cost analysis are given in Table III.

The cost analysis results for producing silicon by this new technology are presented in Table IV including costs for raw materials, labor, utilities and other items composing the product cost (total cost of producing silicon). The tabulation summarizes all of these items to give a total product cost without profit of 6.90 \$/Kg (1975 dollars) and 8.90 \$/Kg (1978 dollars) for 1975 and 1978 time periods. This product cost without profit includes direct manufacturing cost, indirect manufacturing cost, plant overhead and general expenses.

A preliminary cost sensitivity analysis was performed to determine the influence of cost parameters on the economics of producing silicon by this new technology. The cost sensitivity results for the 1975 time period are given in Figure 2 in which product cost (\$/Kg Si) is plotted versus variation (-100% to 0% to +100%) of the primary cost parameters (plant investment, raw materials, labor and utilities). The 0% variation represents the base case. The -100% variation corresponds to the case of no costs for the parameter; and the +100% represents the case for a doubling of cost for each parameter. The plot illustrates that product cost is influenced most by plant investment and least by utilities.

The cost sensitivity results for 1978 time period are presented in Figure 3 for the primary cost parameters. The results indicate that the cost parameter influence on product cost is: plant investment (most), raw materials (intermediate), labor (intermediate) and utilities (least).

The product cost represents all cost associated with producing one kilogram of silicon. On top of these costs a producing company will include some profit. The sales price of the product silicon will actually be the sum of the product cost and a profit for the company. The profit is usually measured in terms of rate of return on the capital investment that the company spent in going into the polysilicon business. Two profitability methods which are commonly used are the return on original investment (% ROI) and discounted cash flow rate of return (% DCF).

The cost and profitability analysis summary for this process are presented in Table V for both 1975 and 1978 time periods. The sales price of polysilicon at various rates of return for both profitability methods (% ROI and % DCF) is shown in the lower half of the table. The results indicate a sales price of 9.88 \$/Kg of silicon (1975 dollars) at 20% DCF return on investment.

TABLE II

PROCESS PLANT DESIGN FOR UCC SILANE PROCESS

1. Plant Size

- Silicon produced from silane
- 1000 metric tons/year of silicon
- Solar cell grade silicon
- Molten silicon product

2. Major Processing Unit Operations (1-9)

- Hydrogenation reaction to produce chlorosilanes
- D-01 stripper to remove inert gases and volatile impurities
- D-02 distillation to separate TCS (trichlorosilane) and STC (silicon tetrachloride)
- TCS redistribution reaction to produce DCS (dichlorosilane)
- D-03 distillation to separate DCS and TCS
- DCS redistribution reaction to produce silane (SiH_4)
- D-04 distillation to provide purified silane by removal of trace impurities (B_2H_6 , example)
- Pyrolysis of silane to produce silicon powder
- Consolidation of silicon powder to provide molten silicon product
- Waste treatment provisions
- Storage consideration for feed, in-process and product materials

3. Raw Material Requirements (1-17)

- Metallurgical grade silicon for hydrogenation
- Silicon tetrachloride for hydrogenation
- Hydrogen for hydrogenation
- Copper catalyst for hydrogenation
- Hydrate lime for waste treatment

4. Utility Requirements (1-17)

- Electricity for pumps
- Steam for reboilers
- Cooling water for condensers
- Process water for waste treatment
- Refrigeration for low temperature
- Fuel for heating

5. Major Process Equipment Requirements (1-27)

- Distillation columns to separate components
- Reactors for hydrogenation, redistribution, waste treatment
- Heat exchangers for column reboilers, condensers, vaporizers, coolers, etc.
- Pumps and compressors for flow and compression
- Tanks and bins for raw materials, in-process materials, product and surge storage
- Pyrolysis reactors to convert silane to silicon powder
- Melters for consolidation of powder to silicon product
- Miscellaneous equipment such as ejectors, agitators, filters, combustors, etc.

6. Production Labor Requirements (1-9, 18-27)

- Hydrogenation section including fluid bed reactor and condensation
- Silane section with distillation and redistribution reactors
- Pyrolysis section with silicon powder and consolidation
- Waste treatment
- Hydrogen compression

TABLE III

COST ANALYSIS FOR UCC SILANE PROCESS

1. Raw Material Cost (1-17, 28-30)
 - Metallurgical Grade Silicon, Silicon Tetrachloride, Liquid Hydrogen, Copper Catalyst and Lime
 - Chemical Marketing Reporter
 - Industrial Consultation
 - Material Cost Index (Industrial Chemicals, Wholesale Price Index)
 - Other Personal Communication
2. Utility Cost (1-17, 29-32)
 - Steam, Electricity, Cooling Water, Process water, Refrigeration and Fuel
 - Intermediate Industrial Costs
 - Chemical Week (Plant Sites)
 - Peters and Timmerhaus
 - Utility Cost Index (Wholesale Price Index)
 - Industrial Communication
3. Labor Cost (1-9, 18-27, 29, 33)
 - Average for Petroleum, Coal, Chemical and Allied Industries
 - Average of 5.90 \$/hr (1975 dollars)
 - Skilled Labor: Add 1 \$/hr
 - Semiskilled: Subtract 1 \$/hr
 - Labor Cost Index (Chemicals and Allied Products)
4. Major Process Equipment Cost (1-27, 34-44)
 - Purchased Equipment Cost
 - Vendor Quotations
 - Richardson Process Plant Construction Estimating Standards
 - Guthrie, Popper, Peters and Timmerhaus
 - M & S Equipment Cost Index
 - Other Personal Sources
5. Capital Investment Cost (1-27, 34-49)
 - Major Process Equipment
 - Installation, Piping, Instrumentation, Electrical, Process Buildings
 - Offsites, Utilities, Site Development, General Services, Offices, Receiving, Shipping
 - Engineering, Contingency
 - Fixed Capital Investment for Plant
 - CE Plant Cost Index

TABLE IV

ESTIMATION OF PRODUCT COST FOR UCC SILANE PROCESS

	Cost \$/Kg of Silicon (1975 dollars)	Cost \$/Kg of Silicon (1978 dollars)
1. Direct Manufacturing Cost (Direct Costs).....	4.15	5.23
Raw Materials		
Direct Operating Labor		
Utilities		
Supervision and Clerical		
Maintenance and Repairs		
Operating Supplies		
Laboratory Charge		
2. Indirect Manufacturing Cost (Fixed Cost).....	1.19	1.50
Depreciation		
Local Taxes		
Insurance		
3. Plant Overhead.....	0.66	0.83
4. General Expenses.....	0.90	1.14
Administration		
Distribution and Sales		
Research and Development		
5. Product Cost Without Profit.....	6.90	8.70

FIGURE 2: COST SENSITIVITY: PRODUCT COST VS. VARIATION (PLANT INVESTMENT, RAW MATERIALS, LABOR, UTILITIES) FOR 1975 TIME PERIOD.

FIGURE 3: COST SENSITIVITY: PRODUCT COST VS. VARIATION (PLANT INVESTMENT, RAW MATERIALS, LABOR, UTILITIES) FOR 1978 TIME PERIOD

TABLE V

COST AND PROFITABILITY ANALYSIS SUMMARY FOR UCC SILANE PROCESS

1. Process.....	UCC Silane Process
2. Plant Size.....	1,000 Metric Tons/year
3. Plant Product.....	Solar Cell Grade Silicon
4. Product Form.....	Liquid Phase Silicon
5. Plant Investment.....	\$10,570,000 / \$13,280,000
	(1975 dollars) (1978 dollars)

Fixed Capital	\$9.19 Mega	\$11.55 Mega
Working Capital	\$1.38 Mega	\$ 1.73 Mega
(15%) Total	\$10.57 Mega	\$13.28 Mega
	(1975 dollars)	(1978 dollars)

6. Return on Original Investment, after taxes (%ROI)

	Sales Price \$/Kg of Silicon (1975 dollars)	Sales Price \$/Kg of Silicon (1978 dollars)
0% ROI.....	6.90	8.70
5% ROI.....	7.92	9.98
10% ROI.....	8.93	11.25
15% ROI.....	9.95	12.53
20% ROI.....	10.96	13.81
25% ROI.....	11.98	15.09
30% ROI.....	13.00	16.36
40% ROI.....	15.03	18.92

7. Discounted Cash Flow Rate of Return, after taxes (% DCF)

	Sales Price \$/Kg of Silicon (1975 dollars)	Sales Price \$/Kg of Silicon (1978 dollars)
0% DCF.....	6.90	8.70
5% DCF.....	7.55	9.52
10% DCF.....	8.27	10.43
15% DCF.....	9.05	11.41
20% DCF.....	9.88	12.45
25% DCF.....	10.74	13.54
30% DCF.....	11.84	14.66
40% DCF.....	13.51	17.01

Based on 10 year project life and 10 year straight line depreciation.

The variation of sales price with return on investment is given in Figure 4 by plotting sales price versus time at various profit levels for 1975, 1976, 1977 and 1978 time periods.

The effect of inflation (higher costs for items such as raw materials, utilities, labor, etc.) is shown in Figure 5. In the figure, the sales price is plotted against time at various inflation levels (0%, 5%, 7% and 10% inflation) for the 1975 to 1990 time period.

STATUS OF TECHNOLOGY

Most of the major processing unit operations have been experimentally determined in small bench-scale equipment (laboratory size) with short run times (several hours to several days). These bench-scale experimental findings have yielded favorable results for silane generation and silicon powder formation from silane. Based on these favorable findings, primary activities are being focused on scale-up to larger size equipment (pilot plant size) with longer run times. Specific current plans involve engineering design of a 100 MT/yr size facility (experimental process development unit).

SUMMARY

A preliminary process design was performed to provide detailed data for cost analysis. The design was based on a plant size of 1000 metric/tons year production of solar cell grade silicon.

Cost and sensitivity analysis results are presented for producing silicon by this new technology including costs for raw materials, labor, utilities, and other items composing product cost (total cost of producing silicon). The results indicate a total product cost without profit of 6.90 \$/Kg (1975 dollars) and 8.70 \$/Kg (1978 dollars) for 1975 and 1978 time periods. For sensitivity analysis, the order of cost parameter influence on product cost is given by plant investment (most), raw materials (intermediate), labor (intermediate) and utilities (least).

A cost and profitability analysis summary is also presented including sales price of polysilicon at various rates of return on investment. The profitability results indicate a sales price of 9.88 \$/Kg of silicon (1975 dollars) at a 20% DCF return on investment.

This new technology for producing polysilicon shows good promise for meeting the cost goal of \$10 per Kg of silicon material (1975 dollars) for solar cells.

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FIGURE 2: COST SENSITIVITY: PRODUCT COST VS. VARIATION (PLANT INVESTMENT, RAW MATERIALS, LABOR, UTILITIES) FOR 1975 TIME PERIOD

FIGURE 5: SALES PRICE VS. YEAR AT VARIOUS INFLATION LEVELS

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MILESTONE CHART

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PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL TASK I

JPL Contract No. 954343

UNIVERSAL PLANNING FORM 3/16" TYPE T1-9470-A