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**PROCESS FEASIBILITY STUDY IN SUPPORT OF
SILICON MATERIAL TASK I**

Quarterly Technical Progress Report (III)

June 1976

Work Performed Under JPL Contract No. 954343

**Department of Chemical Engineering
Lamar University
Beaumont, Texas**

MASTER

**ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy**

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June, 1976

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ABSTRACT

Major efforts were devoted to process system properties, chemical engineering and economic analyses during this reporting period.

For Task 1, primary data analysis activities were expended on silicon source materials--silicon tetrachloride (SiCl_4) and silicon tetraiodide (SiI_4)--associated with two of the processes under consideration for solar cell grade silicon. Critical property data are available for silicon tetrachloride with general agreement of results among the several investigators. Selected values are presented. Property data for silicon tetraiodide are lacking with no data available for the critical constants--critical temperature, pressure, volume, compressibility factor and density.

Data source compilation for the additional source material--silicon tetrafluoride (SiF_4)--is nearing completion.

In Task 2, chemical engineering analysis activities were continued. Preliminary process design considerations were initiated for application to the alternate processes. In addition, major key guidelines were formulated for use in accomplishing the engineering design.

Preliminary process design for the Zn/SiCl_4 process (Battelle) was initiated. Process flow diagram, material balance, energy balance and major equipment design are in progress for the preliminary design.

For Task 3, review of methods for estimation of total product cost was initiated including adaption for application to solar cell grade silicon processes. Major expense items--direct manufacturing cost, indirect manufacturing cost, plant overhead, by-product credit (or debit) and general expenses--are covered. Provisions are also included for the raw materials, labor and utilities requirements associated with a particular process.

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I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

Major activities were continued during this reporting period on process system properties analyses. Primary efforts were devoted to preliminary data collection and analysis for the following silicon source materials--silicon tetrachloride (SiCl_4), silicon tetraiodide (SiI_4), and silicon tetrafluoride (SiF_4).

In the data analysis activity, experimental data were identified for the critical properties (B5, B8, B9, B11, B32, B33, B35, B36, B44, B50, B56, B59, B82, B83, B97) of silicon tetrachloride. The fairly recent results of Russian investigators (B50) were selected for critical temperature and volume. Critical pressure was selected as the average of the three experimental determinations (B44, B83, B97). The results among the several foreign investigators are in general agreement. Deviations from the selected values are 4.6%, 0.5%, and 10.8% respectively for critical temperature, pressure and volume. The selected values are presented in Table I.

Data analysis activities for silicon tetraiodide indicated that property data were very minimal and often lacking for this silicon source material. Preliminary data analysis results are tabulated in Table II. As disclosed in the table, no data exists for the critical constants--critical temperature, pressure, volume, compressibility factor and density.

Preliminary data collection activities were continued for silicon tetrafluoride. This included data source identification, procurement and compilation. Procurement requests were issued for both domestic and foreign sources.

Data source compilation for silicon tetrafluoride is nearing completion. Results will be published during the next reporting period.

TABLE I PRELIMINARY DATA ANALYSIS - CRITICAL CONSTANTS AND PHYSICAL
 PROPERTIES OF SILICON TETRACHLORIDE, SiCl_4

Identification	Silicon Tetrachloride SiCl_4
State (std. cond.)	Liquid
Molecular Weight, M	169.90
Boiling Point, T_b , °C	57.3
Melting Point, T_m , °C	-69.4
Critical Temp, T_c , °C	234.0
Critical Pressure, P_c , atm.	37.0
Critical Volume, V_c , $\text{cm}^3/\text{gr mol}$	326.3
Critical Compressibility Factor, Z_c	0.290
Critical Density, ρ_c , gr/cm^3	0.5207

TABLE II PRELIMINARY DATA ANALYSIS - CRITICAL CONSTANTS AND PHYSICAL
PROPERTIES OF SILICON TETRAIODIDE, SiI_4

<u>Identification</u>	<u>Silicon Tetriodide SiI_4</u>
State (std. cond.)	Solid (colorless, cubic)
Molecular Weight, M	535.7
Boiling Point, T_b , $^{\circ}\text{C}$	301 ± 1
Melting Point, T_m , $^{\circ}\text{C}$	122 ± 2
Critical Temp, T_c , $^{\circ}\text{C}$	- - -
Critical Pressure, P_c , atm.	- - -
Critical Volume, V_c , $\text{cm}^3/\text{gr mol}$	- - -
Critical Compressibility Factor, Z_c	- - -
Critical Density, ρ_c , gr/cm^3	- - -

II. CHEMICAL ENGINEERING ANALYSES (TASK 2)

Chemical engineering analyses were continued in accordance with the program schedule. Primary efforts were devoted to initiation of preliminary process design considerations, technical information exchange at the second task integration meeting and screening technical progress reports from several contractors.

Preliminary process design considerations were initiated during this reporting period with respect to application to the alternate processes under consideration for production of solar cell grade silicon. The activities primarily focused on formulation of the following key guideline items for use in the preliminary process design:

1. Specify base case conditions (plant size, product, quality, etc.)
2. Define reaction chemistry (conversion, thermodynamics, etc.)
3. Process flow diagram (flow sequence, process operations, etc.)
4. Material balance calculations (raw material requirements, products, by-products, etc.)
5. Energy balance calculations (utilities, electrical, heating, etc.)
6. Property data (mp, bp, ρ , C_p , etc.)
7. Equipment design calculations (sizing equipment, type, etc.)
8. List of major process equipment (size, material of construction, etc.)
9. Production labor requirements
10. Forward for economic analysis (plant capital investment, manufacturing cost, etc.)

These key guideline items (1 to 10) will serve as the starting point for performance of the preliminary process design effort in the chemical engineering task.

Additional technical information exchange on several processes under consideration for solar cell grade silicon was accomplished at the second task integration meeting. This included an update on progress, status and plans on the following processes:

1. Zinc Reduction Process - Zn/SiCl_4 (Battelle)
2. Silicon Tetraiodide Process - H_2/SiI_4 or SiI_4 Dissoc. (Battelle)
3. Silane Process - SiH_4 (Union Carbide)

Technical progress reports were received from Battelle, Union Carbide and Texas Instruments. The reports were screened with respect to information relative to the chemical engineering analysis.

Preliminary process design on the Zn/SiCl₄ process (Battelle) for solar cell grade silicon was initiated in accordance with the major task guidelines. The status of the preliminary process design activities is detailed in Table III.

The preliminary process design for the Zn/SiCl₄ process is being conducted under the following base case conditions:

- Plant Size - 1000 metric tons/year
- Product - Solar Cell Grade Silicon
- January 1975 Cost Index

The reaction chemistry for the solar cell grade silicon production in the process is based on zinc reduction of silicon tetrachloride:

In the proposed process, the ZnCl₂ is separated and subjected to electrolysis for recovery of the Zn. The recovered Zn is then recycled for additional production of silicon.

The following activities are in progress for the preliminary process design:

- Process Flow Diagram
- Material Balance
- Energy Balance
- Property Data
- Equipment Design

In addition, several technical discussions with Battelle were accomplished in regards to technical information (T, P, flow sequence, etc.)--related to the above--which have been experimentally investigated under the contract effort.

One company was contacted regarding cost data and production technology for technical grade silicon tetrachloride which is the silicon source material in the Zn/SiCl₄ process.

TABLE III CHEMICAL ENGINEERING ANALYSES:
PRELIMINARY PROCESS DESIGN ACTIVITIES FOR Zn/SiCl_4 PROCESS

<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
		4. Additional	0
2. Define Reaction Chemistry	0	8. List of Major Process Equipment	0
1. Reactants, Products	0	1. Size	0
2. Equilibrium	0	2. Type	0
3. Process Flow Diagram	0	3. Materials of Construction	0
1. Flow Sequence, Unit Operations	0	8a. Major Technical Factors	0
2. Process Conditions (T, P, etc.)	0	(Potential Problem Areas)	0
3. Environmental	0	1. Materials Compatibility	0
4. Company Interaction	0	2. Process Conditions Limitations	0
(Technology Exchange)		3. Additional	0
4. Material Balance Calculations	0	9. Production Labor Requirements	0
1. Raw Materials	0	1. Process Technology	0
2. Products	0	2. Production Volume	0
3. By-Products	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	

III. ECONOMIC ANALYSES

Economic analysis activities were continued during this reporting period with respect to application to alternate processes under consideration for solar cell grade silicon.

A review of methods for estimation of total product cost was initiated. Results of the review are shown in Table IV for estimation of total product cost.

In Table IV, direct manufacturing costs are covered in item 1. The first several subitems (1.1, 1.2, and 1.3)--raw materials, direct operating labor and utilities--depend on the process technology and evolve from the preliminary process design. For example, one process may only require one or two feed raw materials for production of the solar cell grade silicon. Another process may require three or more feed raw materials for the silicon production. The raw material requirements, both specific materials and quantities, will be different for the alternate processes. The preliminary process design provides the raw material requirements for the particular process. The operating labor and utility requirements also depend on the process. The preliminary process design provides the direct operating labor and utility requirements which are a function of the processing steps associated with the particular process under consideration for solar cell grade silicon.

The additional subitems (1.4 to 1.8) in direct manufacturing cost cover supervision (1.4) of personnel operating the production facilities; repair and maintenance (1.5) for the upkeep of the facilities; operating supplies (1.6); laboratory work (1.7) for process and quality control; and patent and royalty provisions (1.8) for the process. These subitems depend on the particular process and may be determined from the labor and capital investment requirements of the particular process.

Item 2 covers the indirect manufacturing costs which are a result of the production operation (but not a direct function of). The indirect manufacturing costs reflect fixed charges which, more or less, remain constant regardless of the production level. Indirect manufacturing costs includes provisions for depreciation (2.1) for the fixed capital investment, local taxes (2.2) on the production plant; insurance coverage (2.3) of the plant and interest (2.5) on the capital borrowed for the plant.

Plant overhead is included in item 3. This includes costs for holidays, vacations, disability pay, pensions, medical services, safety, maintenance on roads, sewer service, plant protection, general plant upkeep, etc. In general, plant overhead is related to direct labor; supervision and clerical and maintenance labor.

Table IV ECONOMIC ANALYSIS-
ESTIMATION OF TOTAL PRODUCT COST

	<u>Ref. 7</u>	<u>Ref. 8</u>	<u>Ref. 9</u>	<u>Ref. 10</u>	<u>Ref. 11</u>	<u>Range</u>
1. Direct Manufacturing Cost (Direct Charges)						
1. Raw Materials - from prel. design	--	--	--	--	--	--
2. Direct Operating Labor - from prel. design	--	--	--	--	--	--
3. Utilities - from prel. design	--	--	--	--	--	--
4. Supervision And Clerical, % of 1.2	10-25%	10-25%	15%	--	10-25%	10-25%
5. Maintenance And Repairs, % of fixed capital (50% labor, 50% materials)	2-10%	4-8%	4%	--	3-6%	2-10%
6. Operating Supplies, % of 1.5	10-20%	12.5-25%	15%	--	5-10%	5-25%
7. Laboratory Charge, % of 1.2	10-20%	20%	20%	--	3-20%	3-20%
8. Patents And Royalties, % of prod. cost	0-6%	--	2%	--	1.2-6%	0-6%
2. Indirect Manufacturing Cost (Fixed Charges)						
1. Depreciation, % of fixed capital	10%	10%	10%	--	5-10%	5-10%
2. Local Taxes, % of fixed capital	1-4%	1-2%	2-3%	.5-1.5%	2%	0.5-4%
3. Insurance, % of fixed capital	0.4-1%	1-2%	1%	.5%	1%	0.4-2%
4. Interest, % of fixed capital	0-8%	--	2-6%	--	6-8%	0-8%
3. Plant Overhead, % of Labor in 1.2 + 1.4 + 1.5	50-70%	40-60%	72%	--	40-60%	40-72%
4. By-Product Credit - from prel. design	--	--	--	--	--	--
4a. Total Manufacturing Cost, 1 + 2 + 3 + 4						
5. General Expenses						
1. Administration, % of manuf. cost	2.3-5.8%	--	3-6%	2.4-12%	--	2.3-12%
2. Distribution And Sales, % of manuf. cost	2.3-23%	--	5-22%	2.4-24%	--	2.3-24%
3. Research And Development, % of manuf. cost	5.8%	--	3.5-8%	2.4-6%	--	2.4-6%
6. Total Cost of Product, 4a + 5						

Item 4 covers credit for by-products. Some processes do not produce a by-product. Other processes may produce one or several by-products. If the by-product is saleable, a credit is obtained. If the by-product is not saleable and expenses are incurred in its disposal, then a debit will result for that by-product. The economics here depend on the nature of the by-product associated with the particular process under consideration.

Item 4a provides the total manufacturing cost. It is equal to the sum of direct manufacturing cost (1), indirect manufacturing costs (2), plant overhead (3) and by-product credit (4).

The general expenses associated with production of the product are covered in item 5. The general expenses includes provisions for administration expenses (5.1) for management salaries, legal fees, communications, etc.; distribution and sales expenses (5.2) and research and development costs (5.3).

Item 6 provides the total cost of the product for the particular process under consideration. The total product cost is the sum of the direct manufacturing cost (1), indirect manufacturing cost (2), plant overhead (3), by-product credit (4) and general expenses (5).

This method of estimation of total product cost is used widely in the chemical industry, particularly in evaluation of the process in the early research stage. Its early use aids in the decision making on whether to proceed with a process or to drop the process and investigate an alternate process. If the total product cost compares favorably with target cost goal (total product cost satisfies goal), then additional funding is merited for the process. However, if the total product cost does not compare favorably with target cost goal (total product cost exceeds goal), then additional funding and further investigation of the process is not merited.

IV. SUMMARY - CONCLUSIONS

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

1. Task 1

Primary data analysis efforts were devoted to silicon tetrachloride (SiCl_4) and silicon tetraiodide (SiI_4) which are silicon source materials associated with two of the processes under consideration for solar cell grade silicon.

Critical property data are available for silicon tetrachloride. Results among the various sources are in general agreement.

Property data for silicon tetraiodide are lacking. Preliminary data analysis indicate no data exists for the critical constants--critical temperature, pressure, volume, compressibility factor and density.

Data source compilation for silicon tetrafluoride (SiF_4) is nearing completion.

2. Task 2

Chemical engineering analyses were continued in accordance with the program schedule.

Preliminary process design considerations were initiated. Key guidelines were formulated for application of the design activity to the processes under consideration for solar cell grade silicon.

Preliminary process design for the Zn/SiCl_4 process (Battelle) was initiated. Material balance, energy balance and equipment design calculations are in progress for the preliminary design.

Technical progress reports were received and screened from several industrial contractors.

3. Task 3

A review of methods for estimation of total product cost was initiated. The review activity focused on adaption of the method for application to solar cell grade silicon processes.

The total product cost and its dependence on the particular process under consideration are discussed. The discussion covers major expense items--direct manufacturing cost, indirect manufacturing cost, by-product credit (or debit) and general expense--involved in the production of a product, such as solar cell grade silicon. Provisions for raw materials, labor and utilities requirements associated with a particular process are included.

V. PLANS

Plans for the next reporting period are summarized below:

1. Task 1

Continue analyses of process system properties including data analysis and estimation methods activities.

Complete and issue data source compilation for silicon tetrafluoride (SiF_4) which is involved in two processes under consideration.

2. Task 2

Complete preliminary process design for Zn/SiCl_4 process (Battelle) in accordance with major task guidelines.

Initiate chemical equilibrium activities for application to reactions for production of solar cell grade silicon.

Initiate preliminary process design for additional processes under consideration for solar cell grade silicon.

Screen technical progress reports for information relative to chemical engineering analysis.

3. Task 3

Continue economic analyses activities for application to processes under consideration for solar cell grade silicon. Select nominal values (cost factors) for use in estimation of total product cost.

Perform economic analysis for Zn/SiCl_4 process (Battelle) including estimation of total product cost and capital investment.

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MILESTONE CHART

TASK	1975			1976								
	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												
3. Estimation Methods												
4. Exp.-Corr. Activities												
5. Prel. Prop. Values												
2. Chemical Engineering												
<u>Analyses</u>												
1. Prel. Process Flow Diag.												
2. Reaction Chemistry												
3. Kinetic Rate Data												
4. Major Equip. Req.												
5. Chem. Equil.-Exp. Act.												
6. Process Comparison												
3. Economic Analyses												
1. Cap. Invest. Est.												
2. Raw Materials												
3. Utilities												
4. Direct Manuf. Costs												
5. Indirect Costs												
6. Total Cost												
7. Process Comparison												
Final Report												

PROCESS FEASIBILITY STUDY IN
SUPPORT OF SILICON MATERIAL TASK I