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A Process For High Volume Low Cost Production of Silane
Silicon Material Task of The
Low Cost Silicon Solar Array Project

Quarterly Report No. 1

By

W. C. Breneman

Period Covered

October 6, 1975 - December 18, 1975

Published

January 20, 1976

JPL Contract No. 954334

Union Carbide Corporation
Sistersville Technical Center
Sistersville, West Virginia 26175

This work was performed for the Jet Propulsion
Laboratory, California Institute of Technology
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Administration under Contract NAS 7-100.

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ABSTRACT

The objective of this research program is to demonstrate the feasibility for the high volume low cost production of silane (SiH_4), as an intermediate for solar or semi-conductor grade silicon. The goal of the program is to demonstrate the feasibility for achieving a net production cost of less than \$5.00 per kilogram of silane through the operation of a 10 pound/day scale pilot plant. The process to be investigated is based on the catalytic disproportionation of chlorosilanes using a tertiary amine ion exchange resin followed by fractional distillation to purify the silane product. The overall reaction sequence is represented by:

A portion of the program will be devoted to the synthesis of dichlorosilane from hydrogen chloride and silicon:

so that the quantity of SiCl_4 produced as a co-product might be reduced:

The initial effort has concentrated on the laboratory investigation of the redistribution chemistry. Results to date indicate good agreement with the proposed redistribution equilibrium composition and that the reaction rate at 60°C is within an order of magnitude of the anticipated value. A small laboratory glass reactor system has been operated to produce 0.27 pound of SiH_4 /hour/pound resin while consuming 4.2 pounds of H_2SiCl_2 /hour and producing 3.93 pounds/hour of a co-product stream of HSiCl_3 , SiCl_4 and unreacted H_2SiCl_2 . The latter could be recycled in a more elaborated apparatus. The preliminary design of a mini-plant based on these results is in progress. Work during the next quarter will include preliminary studies on the HCl/Si reaction, more laboratory work on the redistribution process and completing the preliminary design of the 10 pound/day silane plant.

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FIGURE 4	Program Cost Plan
FIGURE 5	Program Labor Plan

SUMMARY

A laboratory glassware apparatus has been designed and assembled to study the redistribution reaction of chlorosilanes. Other laboratory reactors have been designed and assembled to study the reaction of silicon metal with H_2 and HCl in the presence of copper catalyst and at temperatures from 250° to $650^\circ C$. The redistribution of dichlorosilane has been studied using as a catalyst, a tertiary amine macroreticular ion exchange resin, Rohm and Haas Amberlyst A-21. The results to date indicate that roughly 13 mole percent SiH_4 is produced in a single pass at $60^\circ C$ with 6 seconds contact time in the vapor phase. Using a small distillation unit in conjunction with the resin bed and recycling the by-product monochlorosilane and dichlorosilane a production rate of 0.27 pound of SiH_4 per hour per pound of resin has been achieved while consuming 4.2 pounds of dichlorosilane and producing 3.93 pounds per hour of a co-product trichlorosilane. The latter would be recycled also in a more elaborate apparatus. Based on these data, the preliminary design has been initiated for a "mini-plant" capable of producing 10 pounds/day of SiH_4 .

INTRODUCTION

This research program commenced October 6, 1975. Its purpose is to determine the feasibility for the high volume, low cost, production of silane (SiH_4) as a raw material for solar or semi-conductor grade silicon. The basic process to be investigated is the tertiary amine ion exchange resin catalyzed redistribution of chlorosilanes in combination with distillation to yield ultimately SiH_4 and co-product silicon tetrachloride (SiCl_4). The production cost goal is under \$5 per kilogram of SiH_4 after crediting SiCl_4 . A concurrent investigation is also programmed to study the direct synthesis of dichlorosilane from metallurgical silicon as a means of reducing the quantity of co-product SiCl_4 .

Prior to this program Union Carbide Corporation has shown experimentally that pure chlorosilanes can be redistributed into an equilibrium mixture of all other hydrochlorosilanes by contact with a tertiary amine ion exchange resin, Rohm and Haas Amberlyst A-21. Patent rights for this process and improvements there on have been filed.

Dichlorosilane (H_2SiCl_2) is produced in commercial quantities by the A-21 resin catalyzed redistribution of trichlorosilane (HSiCl_3) to H_2SiCl_2 with SiCl_4 a co-product.

This program will extend this technology toward the following major tasks.

1. Design, construction, operation and evaluation of an experimental mini-plant capable of producing up to 10 pounds of SiH_4 per day by the A-21 catalyzed redistribution of H_2SiCl_2 .

2. Design, construction, operation and evaluation of an experimental facility to produce, from metallurgical silicon metal, a crude chlorosilane mixture enriched with H_2SiCl_2 .
3. Design and construction, operation and evaluation of maxi-plant which can produce 10 pounds of SiH_4 per day from trichlorosilane and/or dichlorosilane-trichlorosilane mixtures.
4. Process studies relating to the above plants including economics and energy use evaluations.

To date the major emphasis has been on the laboratory verification of the chemistry of the redistribution reaction which is fundamental to the overall project.

TECHNICAL DISCUSSION

To study the chemistry and confirm the process data required for the design of a plant for producing silane, a laboratory glassware apparatus was designed and constructed. The apparatus constructed to date included:

- Glass reactors for the redistribution of trichlorosilane and dichlorosilane to SiH_4 with A-21 resin catalyst. (Figure 1)
- Glass fluidized bed reactor for the reaction of a Cu/Si contact mass with HCl and H_2 gas for producing an H_2SiCl_2 enriched chlorosilane mixture.
- Vycor glass (quartz) fluidized bed reactor for the reaction of Si metal with H_2 and SiCl_4 to generate HSiCl_3 at high temperature.
- An apparatus to safely dispose of spontaneously flammable SiH_4 .

Supporting apparatus such as distillation columns, cold traps, metering valves, flowmeters and a on-line chromatograph were also installed.

1. LABORATORY INVESTIGATIONS

A. Equipment and Safety Review

Laboratory equipment and apparatus were standardized and calibrated, e.g., reactor temperature, flowmeters, gas burette, etc. All apparatus operated safely with no difficulties. The spontaneously flammable silane and monochlorosilane products were safely disposed of by burning in the hood atmosphere with no problems. All equipment functioned as designed.

B. Disproportionation of H_2SiCl_2

The disproportionation of H_2SiCl_2 to SiH_4 was carried out in apparatus shown in Figure 1 attached. Two sets of experiments were carried out.

1. Preliminary experiments were carried out to test the apparatus and to check out previous data obtained by C. J. Bakay of Union Carbide Corporation, 1970.¹ In this case, the dry-ice condenser and U-tube recycle assembly in Figure 1 were eliminated. Dichlorosilane was fed through a calibrated flowmeter into an Amberlyst A-21¹ resin bed, 21 mm diameter by 317 mm high, containing 40 grams of resin. At a H_2SiCl_2 flow of 2.8 grams per minute, 60°C, atmospheric pressure, the gas generated amounted to 92 cc per minute as measured by oil displacement. On a mole percent basis, 13.5 mole percent SiH_4 was produced from H_2SiCl_2 in one pass through the bed. These results compare favorably with the previous data of C. J. Bakay, i.e., 12 to 15 mole percent SiH_4 .

2. In order to simulate the proposed operation of a silane mini-plant, the unreacted H_2SiCl_2 and H_3SiCl by-product were recycled through the A-21 resin bed.

As shown in Figure 1, the product mixture from the resin bed was fed, first, into a packed distillation column to remove high-boiling by-products HSiCl_3 and SiCl_4 . Then the unreacted H_2SiCl_2 and by-product H_3SiCl were condensed with a dry-ice condenser

¹ See Background Data Section for additional details.

into a U-tube immersed in a dry-ice bath. One side of the U-tube was heated with a vacuum-jacketed insulated electric heater. The vaporized H_2SiCl_2 and H_3SiCl were moved by a slight hydrostatic pressure generated inside the U-tube and recycled through the A-21 resin bed. The SiH_4 was further purified by passing through another dry-ice trap to remove H_2SiCl_2 and H_3SiCl which might escape the first condenser. In this experiment, H_2SiCl_2 was fed at a constant flow rate of 2.8 grams per minute as in (1) into the same resin bed at 60°C . SiH_4 was produced at 137 cc per minute. The HSiCl_3 and SiCl_4 collected at the bottom of the distillation column were analyzed to give 17.8% H_2SiCl_2 , 80% HSiCl_3 , 2.1% SiCl_4 and less than 0.3% heavies. Thus with H_2SiCl_2 and H_3SiCl recycled, the stoichiometry of the overall disproportionation could be written approximately as:

or, theoretically, three moles of H_2SiCl_2 for every mole of SiH_4 . The calculated SiH_4 production rate based on 2.8 grams per minute of H_2SiCl_2 feed would be 227 cc per minute. Hence the measured SiH_4 production rate, 137 cc per minute, was only 60.5% of the calculated value. The observed, lower SiH_4 production rate was due to the loss of some H_2SiCl_2 in the $\text{HSiCl}_3/\text{SiCl}_4$ by-product stream, and retention of some $\text{H}_2\text{SiCl}_2/\text{H}_3\text{SiCl}$ in the second dry-ice trap. Future experiments will attempt to improve component separations in order to obtain a better material balance. A calculation based on these crude data showed that the SiH_4 production rate was 0.27 pound SiH_4 per hour per pound of A-21 resin; with a corresponding feed of 4.2 pounds H_2SiCl_2 per hour per pound A-21.

C. Identification of SiH₄

Samples of SiH₄ produced in experiments (1) and (2) above were withdrawn into sample tubes. Identification of SiH₄ by comparison of its mass spec and IR spectra with those of an authentic SiH₄ sample was planned. A known sample of SiH₄ (Union Carbide Corporation, Linde Specialty Gas) and samples from the reaction product are being analyzed by mass spec. Equipment failure has delayed the IR analysis.

D. Miscellaneous Data Collected

1. The density of loosely packed, dry A-21 resin catalyst was determined to be 0.35 gram/cc (21.8 lb/cu ft) with about 65% void space. Thus the density of the solid resin was approximately 1 gram/cc. (Manufacturer's data indicates a density of 1.1 gram/cc.)

2. The linear gas velocity through the A-21 resin bed was 0.279 cm/sec based on the empty tube with 2.8 gram per minute H₂SiCl₂ feed in experiment (b.1.), and about 0.432 cm/sec in the case of recycled H₂SiCl₂/H₃SiCl in experiment (b.2.). The residence time based on the void space in the resin bed was approximately 6 seconds and 4 seconds respectively. As the linear upward velocity increased to about 0.686 cm/sec the A-21 resin bed became fluidized. Thus a downward gas flow direction would be preferred at higher gas velocities.

II. PILOT SCALE STUDIES

A. Physical Property File For Fractionation Study

A compilation of physical properties for the five principle compounds involved in this study, SiCl₄, HSiCl₃, H₂SiCl₂,

H_3SiCl and SiH_4 has been made. Basically, the data are those derived by Boggs, et al of Texas Instruments Company for the chlorosilanes and by the Matheson Gas Products Company for SiH_4 . The ultimate source was from the International Critical Tables and JANAP. Unavailable properties were estimated by Boggs using various well known and reasonably reliable correlations. The data were compiled into equation form by a least squares method to fit with an average deviation of less than 2% for the heat capacity and less than 1% for the vapor pressure (by an extended Antoine equation) over the range of interest. The physical property data is attached. (Table II)

B. Process Equipment

The two major items of equipment which will be required and which have a reasonably long delivery and/or operational time delay are the diaphragm compressor to be used to package the product silane and a chromatograph to analyze the various product streams.

1. Compressor. The compressor judged suitable for this application is a diaphragm type manufactured by Pressure Products Industries, Model 1084. This unit has a capacity of 0.3 cfm at a suction pressure of 0 psig and a discharge pressure of 300 psig. This capacity is, at 0 psig, approximately 30% higher than required for a 10 pound SiH_4 per 8 hour day rate. The capacity would increase significantly at higher suction pressures and is thus a very flexible unit for this particular application. The quoted price was \$6,500 for the standard model desired with a delivery time of twenty-four weeks.

2. Chromatograph. Due to the air and moisture sensitivity of the materials to be analyzed, an on-line vapor phase chromatograph is virtually mandatory for accurate analysis. Based on consultation with C. Thrash of the Union Carbide Sistersville Analytical Group, a recognized expert in the field, two units were considered most feasible; a Hewlett Packard Model 5712A unit with recorder and accessories for \$7,730, or a Hewlett Packard Model 5832A unit for \$11,500. The first is a manual unit with only a strip chart readout. The second is a complete analytical reporting system which will calculate compositions directly. Due to the large number of sample points and the relatively high frequency of samples, the second unit is recommended by Mr. Thrash. A decision will be required on this item by the end of January, 1976.

CONCLUSIONS

The good agreement between the equilibrium redistribution composition found in the initial experiment and the earlier work which formed the basis for this current program is encouraging. The design of the mini-plant will proceed forthwith.

NEW TECHNOLOGY

No new technology is presented in this report which has been developed under the scope of Contract 954334.

PROJECTED SECOND QUARTER ACTIVITIES

DICHLOROSILANE SYNTHESIS

During the next quarter the attention of the laboratory program will shift initially to a preliminary evaluation of the direct synthesis of dichlorosilane from metallurgical silicon and hydrogen chloride. This reaction, catalyzed by copper and run in the presence of hydrogen should yield a crude chlorosilane mixture containing 16 to 20% H_2SiCl_2 . This relatively large amount of H_2SiCl_2 could significantly reduce the quantity of by-product SiCl_4 in subsequent processing to SiH_4 as compared to the use of only HSiCl_3 which is the normal product resulting from hydrochlorination of silicon. Attention will be given to the nature and amount of copper catalyst required and to the effect of hydrogen gas in the reactant HCl stream. Past experience with copper/silicon contact masses has shown the two-metal interaction to be most significant in determining the composition of the product chlorosilane mixture. The conditions will be sought which are required to maximize the formation of H_2SiCl_2 . Other process parameters which should have a significant effect are the reaction temperature, contact time, H_2/HCl ratio and pressure. The latter cannot be studied in laboratory glassware and will be deferred until the steel reactor system is operating.

Since the mechanical design of the dichlorosilane synthesis plant is dependent on the residence time required in the reaction; the final reactor design will not be frozen until at least the preliminary glassware studies are well underway.

The preliminary design of the H_2SiCl_2 synthesis plant, with respect to auxiliary services - condensers, raw material delivery system, and product collection system will be proceeding concurrent with the laboratory investigation.

REDISTRIBUTION STUDIES

With the good results obtained to date, future work in this area will deal with determining reaction rates and equilibrium compositions as a function of temperature, identifying unknown by-products [other than H_xSiCl_y ($x + y = 4$)], if any, and precise determination of equilibrium compositions.

The design of the mini-plant will proceed concurrently using process information generated to date. Computer aided design of the fractionating column(s) will use an in-house multi-component fractionation design program.

GENERAL

Other items programmed for the next quarter include an evaluation of silane purification methods and selection of that one(s) applicable to the impurities most likely present by this process. A review of the problems of storage of silane gas will be made and recommendations made for short and longer term actions.

PROGRAM STATUS UPDATE

Update versions of the approved program plan and labor and cost summaries are shown in Figure 3, 4, 5.

BACKGROUND DATA

REDISTRIBUTION CHEMISTRY

Laboratory investigations by C. J. Bakay of Union Carbide in 1971-1972 on the synthesis of hydrochlorosilanes demonstrated that liquid or vapor trichlorosilane can be redistributed to dichlorosilane and silicon tetrachloride using a macroreticular tertiary amine ion exchange resin manufactured as Rohm and Haas Amberlyst A-21 (Table I). The equilibrium reaction would thus be written:

The similar reaction of pure dichlorosilane also gave a mixture of all chlorosilanes including a significant amount of silane, SiH_4 .

Working from five feed compositions, Mr. Bakay generated a description of the process which showed the equilibrium product composition to be only a function of the Cl:Si ratio of the feed material, Figure 2. The laboratory program of this contract will corroborate this data and determine the reaction rate.

TABLE I

Properties of Rohm and Haas Amberlyst A-21

Property	Amberlyst A-21
Appearance	Hard, Spherical, Light Tan Water-saturated Beads
Chemical Composition	Styrene-divinyl Benzene Copolymer Modified With Dimethylamine
Ionic Form	Free Base
Moisture Holding Capacity, %	45-53
Exchange Capacity Weight Capacity, meg/g Dry Resin.	4.7 - 5.0
Density, Lb/Ft ³	38 - 42
Effective Size, mm	0.40 to 0.55
Uniformity Coefficient	2.0
Fines (% Through No. 50 Sieve)	1.0 Maximum
Hydraulic Expansion, Free Base Form at 20 gpm/cu ft, 30°C, %	120 Maximum
Whole Bead Content	100
Porosity, %	35 to 45
Average Pore Diameter, Å	700 to 1200
Surface Area, m ² /g	20 to 30
Solid, %	47 to 55
Percent Swelling Form Dry State to Solvent Saturated State	
Hexane	20
Toluene	25
Diethylether	22
Acetone	22
Ethanol	30
Water	25

TABLE II

Physical Properties of ChlorosilanesVapor Pressure

Silane	$\text{LnP} = 6.55 - 1482.4/T + 2.1077 \text{ LnT} - 0.00874 T$
Monochlorosilane	$\text{LnP} = 441.3 - 12720/T - 75.58 \text{ LnT} + 0.135 T$
Dichlorosilane	$\text{LnP} = 117.6 - 5826.5/T - 17.23 \text{ LnT} + 0.024 T$
Trichlorosilane	$\text{LnP} = 4.909 - 3435/T + 5.02 \text{ LnT} - 0.0193 T$
Silicontetrachloride	$\text{LnP} = 562.237 - 17732/T - 95.577 \text{ LnT} + 0.1585 T$

Where P - mm of Hg

T - °K

Vapor Heat Capacity

SiH_4	$C_p = 0.2233 + 1.32 \times 10^{-4} T + 6.19 \times 10^{-7} T^2$
H_3SiCl	$C_p = 0.1134 + 3.057 \times 10^{-4} T - 9.83 \times 10^{-8} T^2$
H_2SiCl_2	$C_p = 0.1072 + 1.813 \times 10^{-4} T - 6.37 \times 10^{-8} T^2$
HSiCl_3	$C_p = 0.1147 + 1 \times 10^{-4} T - 3.66 \times 10^{-8} T^2$
SiCl_4	$C_p = 0.1161 + 5.790 \times 10^{-5} T - 2.429 \times 10^{-8} T^2$

Where C_p - Cal/gm. °K

T - °K

FIGURE I

APPARATUS FOR DISPROPORTIONATION OF H_2SiCl_2 TO SiH_4

Jeffrey P. Mui
December 18, 1975

FIGURE 3

YEAR
MONTH OF YEAR
MONTH OF CONTRACT

1975		1976												1977											
11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	

I. PRODUCTION OF SiH₄A. Mini-Plant

1. Preliminary Planning
2. Preliminary Design
3. Site Prep and Order Equipment
4. Final Design
5. Installation
6. Initial Operation
7. Integration With OP II

1	2																							
	2	3																						
		3	4																					
				5	6																			
						7	8																	
								9	10	11	12	13	14	15	16									
													14	15	16									

B. Maxi-Plant

1. Preliminary Planning
2. Initial Decision For Maxi
3. Order Long Delivery Items
4. Preliminary Design
5. Final Decision For Maxi
6. Site Preparation
7. Final Design
8. Installation
9. Initial Operation
10. Integration With OP II

			5																					
			▲																					
				6	7																			
										12	13													
										▲														
										13	14													
											14	15	16	17										
													17	18										
																19	20	21						
																			22	23	24			

C. Process Studies

1. Determine Reaction Parameters
2. Evaluate Purification Methods
3. Evaluate Storage Methods
4. Energy and Mass Balance
5. Kinetics Studies
6. Process Evaluation
7. Process Cost Estimation

1	2	3	4	5																				
		3	4	5																				
			4	5																				
								9	10	11	12	13	14				19	20	21	22	23	24		
								9	10	11	12	13	14				19	20	21	22	23	24		
										11	12	13	14						21	22	23			
											12	13	14							22	23	24		

II. DICHLOROSILANE SYNTHESISA. Direct Synthesis Plant

1. Preliminary Planning
2. Preliminary Design
3. Order Equipment
4. Site Preparation
5. Final Design
6. Installation
7. Initial Operation

1	2																							
	2	3																						
		▲	3																					
			4	5																				
					6	7																		
							8	9																
									10	11	12	13	14	15	16			19	20	21	22	23	24	

B. Integration With Mini-Plant

											13	14	15	16										
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C. Integration With Maxi-Plant

																				22	23	24		
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D. Process Studies

1. Determine Reaction Parameters
2. Spent Mass Disposal
3. Energy and Mass Balances
4. Kinetics Studies
5. Process Evaluation
6. Process Cost Estimation

1	2	3	4	5	6																			
			4	5	6	7																		
										11	12	13	14							22	23	24		
										11	12	13	14							22	23	24		
										11	12	13	14							22	23			
											12	13	14							22	23	24		

E. Report

1. Final Report
2. Recommendation For Long Range Development

																							25	
																						24	25	

FIGURE 5 UNION CARBIDE CORPORATION SISTERSVILLE PLANT

BY WCB
CHKD. BY

DATE 12/1/75
DATE

SUBJECT Total Cost Plan For
Low Cost Silane Project (Revised)

SHEET NO. 954334
JOB NO. OF

FIGURE 4
UNION CARBIDE CORPORATION
SISTERSVILLE PLANT

BY WCB
 CHKD. BY
 DATE 12/1/75

SUBJECT Direct Labor Hours Planned For
 High Volume, Low Cost Silane Project
 (Revised)

SHEET NO. 954334
 OF
 JOB NO.

FIGURE 2
Equilibrium Distribution of Hydrochlorosilanes
From
Amberlyst A-21 Catalyzed Redistribution