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PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL TASK I

Quarterly Technical Progress Report (XI)

By
C. S. Fang
Keith C. Hansen
Joseph W. Miller, Jr.
Carl L. Yaws

June 1978

Work Performed Under Contract No. NAS-7-100-954343

Lamar University
Chemical Engineering Department
Beaumont, Texas

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Approval Signature Carl L. Yaws

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ABSTRACT

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

Analysis of process system properties was continued for silicon source materials. Primary efforts centered on data collection, analysis, estimation and correlation. Property data for silicon tetrachloride are reported for critical constants (temperature, pressure, volume, compressibility factor); vapor pressure; heat of vaporization; gas heat capacity and liquid heat capacity. Silicon tetrachloride is the source material in several processes under consideration for solar cell grade silicon production.

Final experimental values for gas phase thermal conductivity of the silicon source materials silane, dichlorosilane, trichlorosilane, tetrachlorosilane, and tetrafluorosilane are reported in the temperature range 25°C to 350°C. These final values reflect a refinement of previously reported preliminary values after complete calibration of the temperature measuring apparatus.

Chemical engineering analysis of the Union Carbide silane process (Case C-Revised Process) was continued with primary efforts being devoted to the preliminary process design. Status and progress are reported for base case conditions, process flow diagram, reaction chemistry and equipement design. Current engineering design is in progress for the several distillation columns which separate the liquid chlorosilanes and provide purified silane product.

TABLE OF CONTENTS

I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

A. SILICON TETRACHLORIDE PROPERTIES

Analysis of process system properties was continued during this reporting period for silicon source materials under consideration for solar cell grade silicon production. Primary activities focused on property data for silicon tetrachloride which is the source material for silicon in several processes.

Critical Properties (Table IA-1)

Experimental results for the critical temperature, pressure and volume of silicon tetrachloride are available (B5, B8, B9, B11, B32, B33, B35, B36, B44, B50, B56, B59, B82, B83). The results among the several investigators are in general agreement. Deviations from the selected values are 1.71%, 0.5%, and 10.8% respectively for critical temperature, pressure and volume.

The critical compressibility factor, Z_c, was calculated using the following equation:

$$Z_c = P_c V_c / R T_c \qquad \text{(IA-1)}$$

Also given in the table are values for the acentric factor, ω which is defined by:

$$\omega = -\log P_r - 1.000 \text{ (at } T_r = 0.70) \qquad \text{(IA-2)}$$

The acentric factor is an important paramenter in generalized thermodynamic correlations involving virial coefficients, compressibility factor, enthalpy and fugacity.

Vapor Pressure (Figure IA-1)

Experimental vapor pressure data for silicon tetrachloride are available (B7, B22, B24, B27, B30, B32, B43, B53, B78, B103) from slightly above the melting point (mp) to boiling point (bp) and at the critical point (cp). Available data were extrapolated using the YSSP vapor pressure correlation (B102):

$$\log P_v = A + \frac{B}{T} + C \log T + DT + ET^2 \qquad \text{(IA-3)}$$

where

P_v = vapor pressure of saturated liquid, mm of Hg

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

T = temperature, °K

The correlation constants (A, B, C, D and E) were determined using a generalized least squares computer program for minimizing deviation of calculated and experimental data values screened from the literature. Average absolute deviation was about 0.7% for the fifty-eight data points.

Heat of Vaporization (Figure IA-2)

Heat of vaporization data for silicon tetrachloride are available only at the boiling point (B5, B11, B22, B30, B36, B65, B82, B86). Watson's correlation was used to extend the heat of vaporization over the entire liquid phase:

$$\Delta H_v = \Delta H_{v_1} \left[\frac{T_c - T}{T_c - T_1}\right]^n \qquad \text{(IA-2)}$$

where ΔH_{v_1} is the heat of vaporization at the boiling point (T_1) and n = 0.38.

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

Heat capacity data for silicon tetrachloride as ideal gas at low pressure are available (B3, B10, B17, B20, B28, B32, B34, B43, B45, B52, B67, B73, B76, B82, B84, B86, B91). The values, which are primarily based on structural and spectral measurements, are in close agreement.

The heat capacity data for the gas phase were correlated by a series expansion in temperature

$$C_p = A + BT + CT^2 + DT^3 \qquad \text{(IA-3)}$$

where C_p - heat capacity of ideal gas at low pressure, cal/(g-mol)(°K); A, B, C and D = characteristic constants for the chemical compouns; and T = temperature, °K. Average absolute deviation is about 0.6%.

Liquid heat capacity data are available (B5, B22, B28, B30, B26, B43, B52, B60, B65, B76, B77, B82, B104) in the mp-bp temperature interval. The data were extended to cover the entire liquid phase with the relation:

$$\text{liquid heat capacity x density = constant} \qquad \text{(IA-4)}$$

The constant value was 0.3054. Testing of the relationship with the available data produced average deviation of 4%.

TABLE IA-1 CRITICAL CONSTANTS AND PHYSICAL PROPERTIES OF SILICON TETRACHLORIDE

Identification	Silicon Tetrachloride
Formula	$SiCl_4$
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, cm^3/grmol	326.3
Critical Compressibility Factor, Z_c	0.290
Critical Density, ρ_c, gr/cm^3	0.5207
Acentric Factor, ω	0.2556

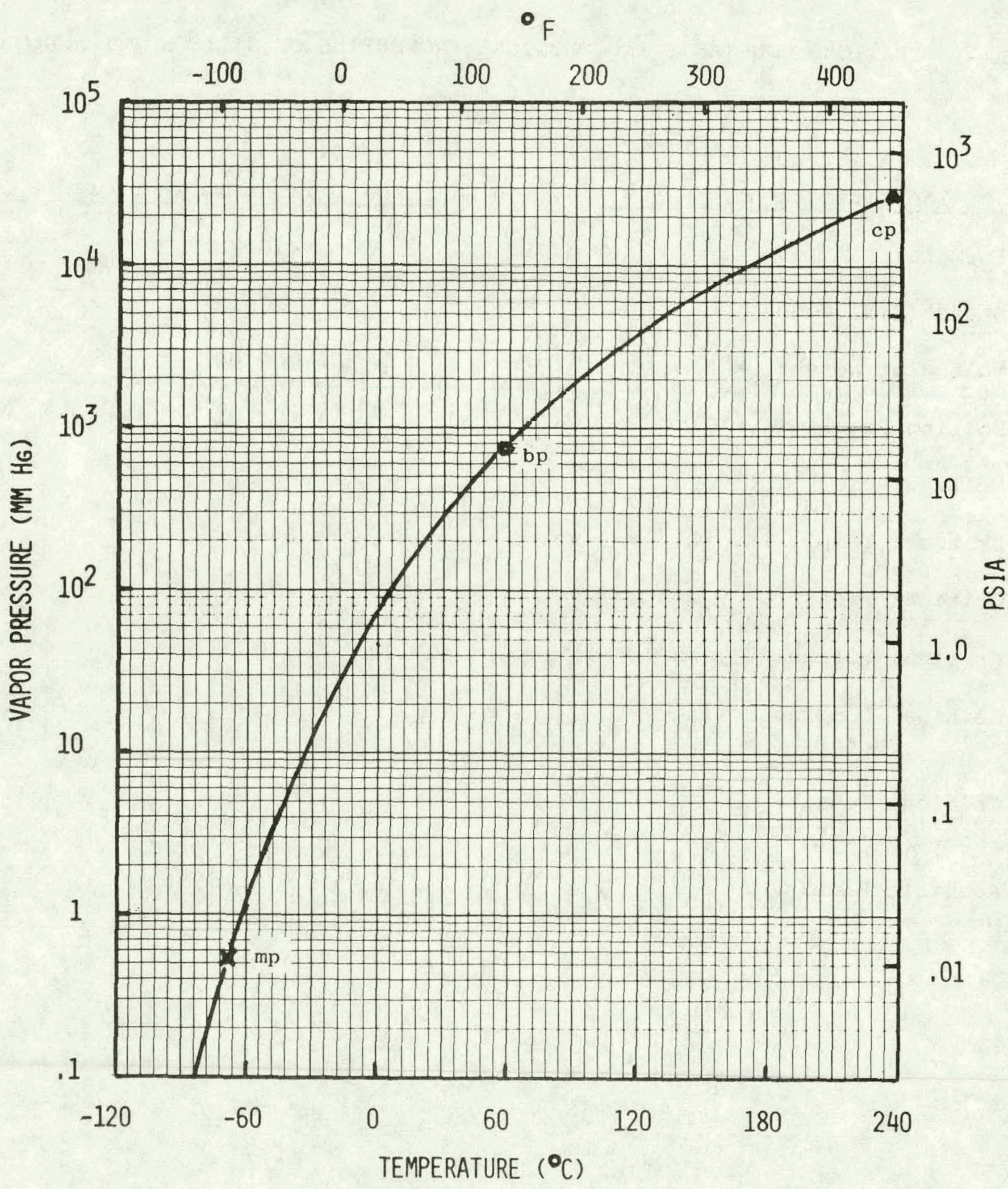

Figure IA-1 Vapor Pressure vs Temperature for Silicon Tetrachloride

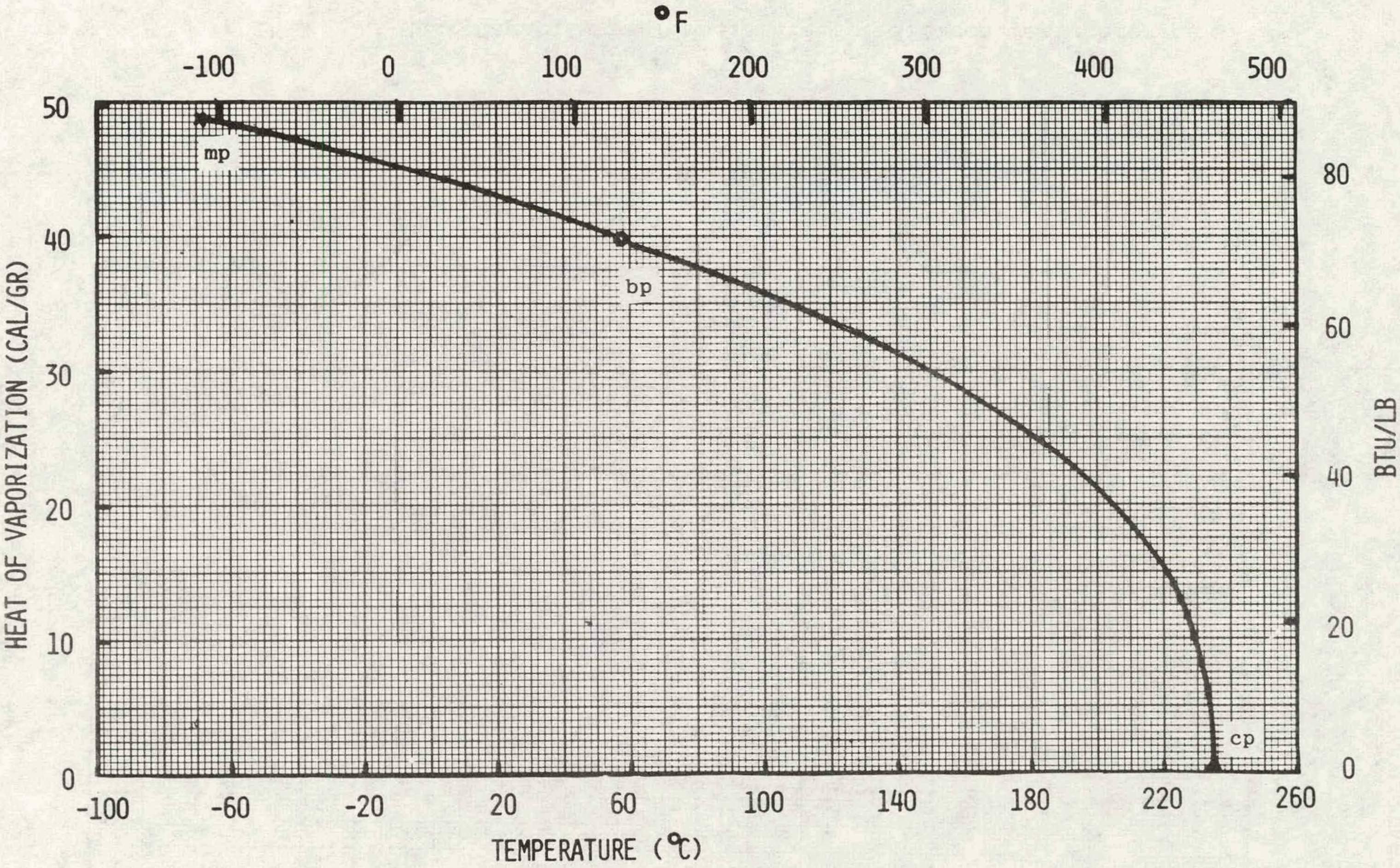

Figure IA-2 Heat of Vaporization vs Temperature for Silicon Tetrachloride

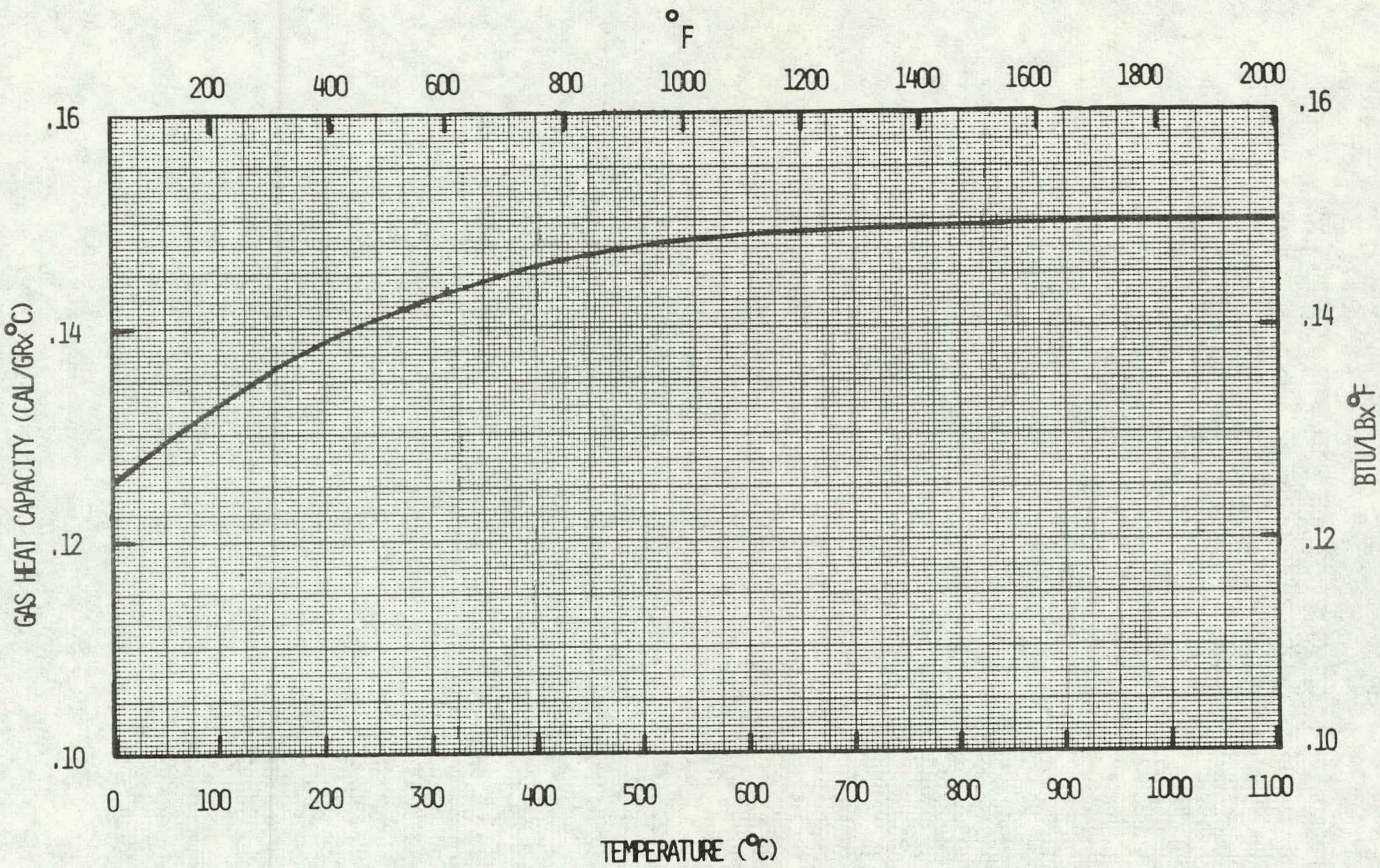

Figure IA-3 Gas Heat Capacity vs Temperature for Silicon Tetrachloride

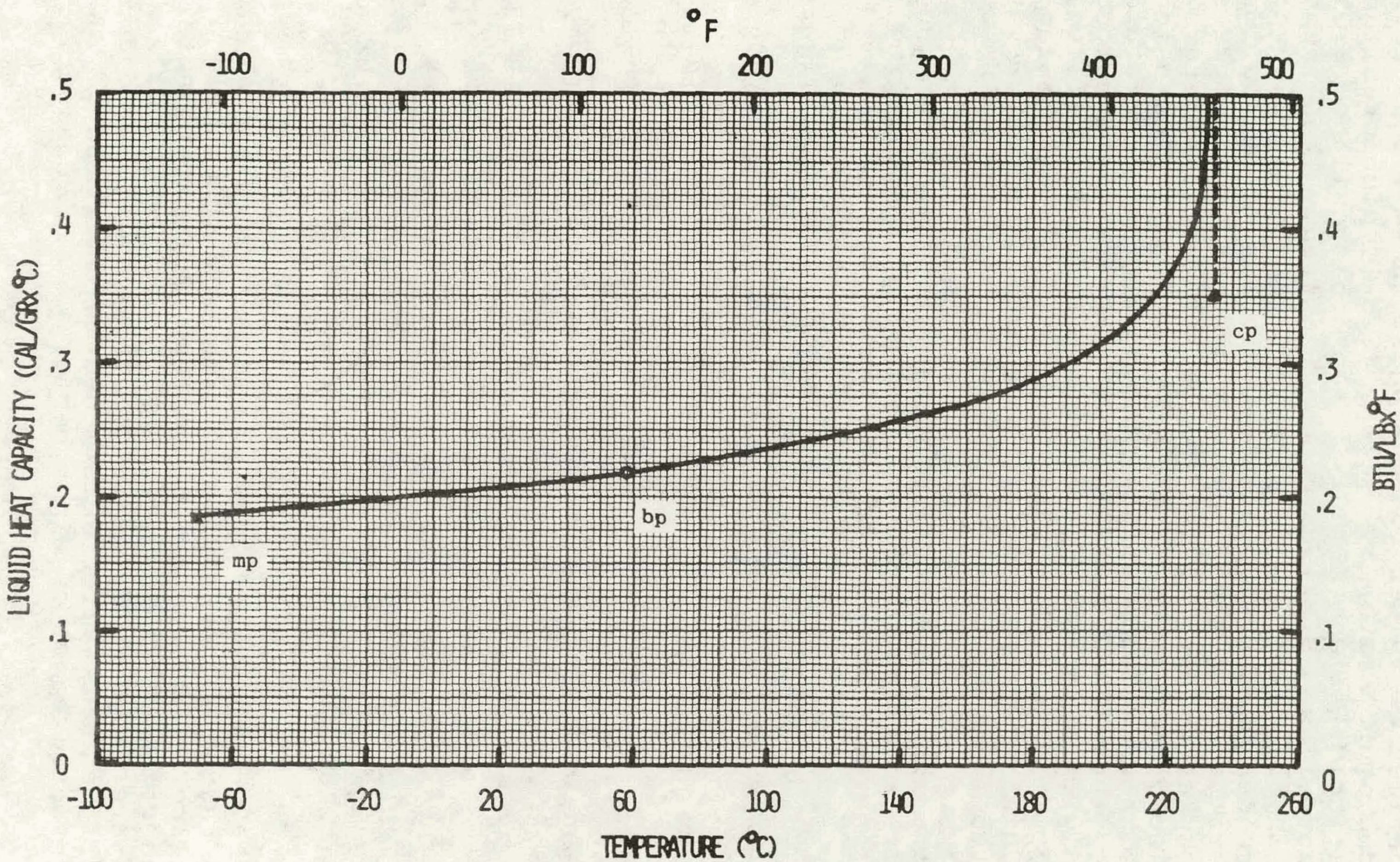

Figure IA-4 Liquid Heat Capacity vs Temperature for Silicon Tetrachloride

B. THERMAL CONDUCTIVITY INVESTIGATION

In progress reports submitted since October, 1977; preliminary experimental values for gas phase thermal conductivity of the silicon source materials silane (SiH_4), dichlorosilane (SiH_2Cl_2), trichlorosilane ($SiHCl_3$), tetrachlorosilane ($SiCl_4$), and tetrafluorosilane (SiF_4) have been reported. These reported values were in the temperature range 25°C to 400°C. The values reported were designated as preliminary because final calibration of the temperature measuring apparatus had not been completed. The thermocouples (type K) used to monitor the temperature of the thermal conductivity cell have now been calibrated using materials of known melting points throughout the temperature range of the study (25°C to 400°C). The EMF of the thermocouples was measured with a Leeds and Northrup, Model 8686, millivolt potentiometer which was calibrated and certified at the factory. The temperatures now reported for the thermal conductivity values are considered to be accurate to ±1°C.

Final experimental thermal conductivity values for the silicon source materials are now reported in the following tables and figures.

Compound	Table	Figure
Silane (SiH_4)	IB-6	IB-9
Dichlorosilane (SiH_2Cl_2)	IB-7	IB-10
Trichlorosilane ($SiHCl_3$)	IB-8	IB-11
Tetrachlorosilane ($SiCl_4$)	IB-9	IB-12
Tetrafluorosilane (SiF_4)	IB-10	IB-13

The final data reported is not significantly different from the preliminary values previously reported. The principle difference is more accurate reporting of the temperature for each data point. The final data for all compounds is summarized in Figure IB-14.

There have been no previously reported experimental values for gaseous thermal conductivity of silane, dichlorosilane, or trichlorosilane. There has been one report of experimental thermal conductivity values (ref. 42) for tetrachlorosilane in the temperature range 70°C to 300°C. Those values were approximately 10% lower than the values determined in this study. There has been one previous report of experimental thermal conductivity values for tetrafluorosilane (ref. 40) in the temperature range 60°C to 430°C. The experimental values determined in this study for tetrafluorosilane agree with the literature values to within 2% throughout the temperature range.

Table IB-6 Gaseous Thermal Conductivity Values of Silane

Temperature	Gaseous Thermal Conductivity		
^{o}C	$mW\ cm^{-1}\ {}^{o}K^{-1}$	$Cal\ cm^{-1} sec^{-1}\ {}^{o}C^{-1}$	$BTU\ hr^{-1} ft^{-1}\ {}^{o}F^{-1}$
28.0	0.234	56.02×10^{-6}	13.54×10^{-3}
45.7	0.249	59.44×10^{-6}	14.37×10^{-3}
94.7	0.297	70.96×10^{-6}	17.15×10^{-3}
139.4	0.345	82.34×10^{-6}	19.90×10^{-3}
184.1	0.400	95.67×10^{-6}	23.13×10^{-3}
227.4	0.449	107.24×10^{-6}	25.93×10^{-3}
269.5	0.497	118.86×10^{-6}	28.73×10^{-3}

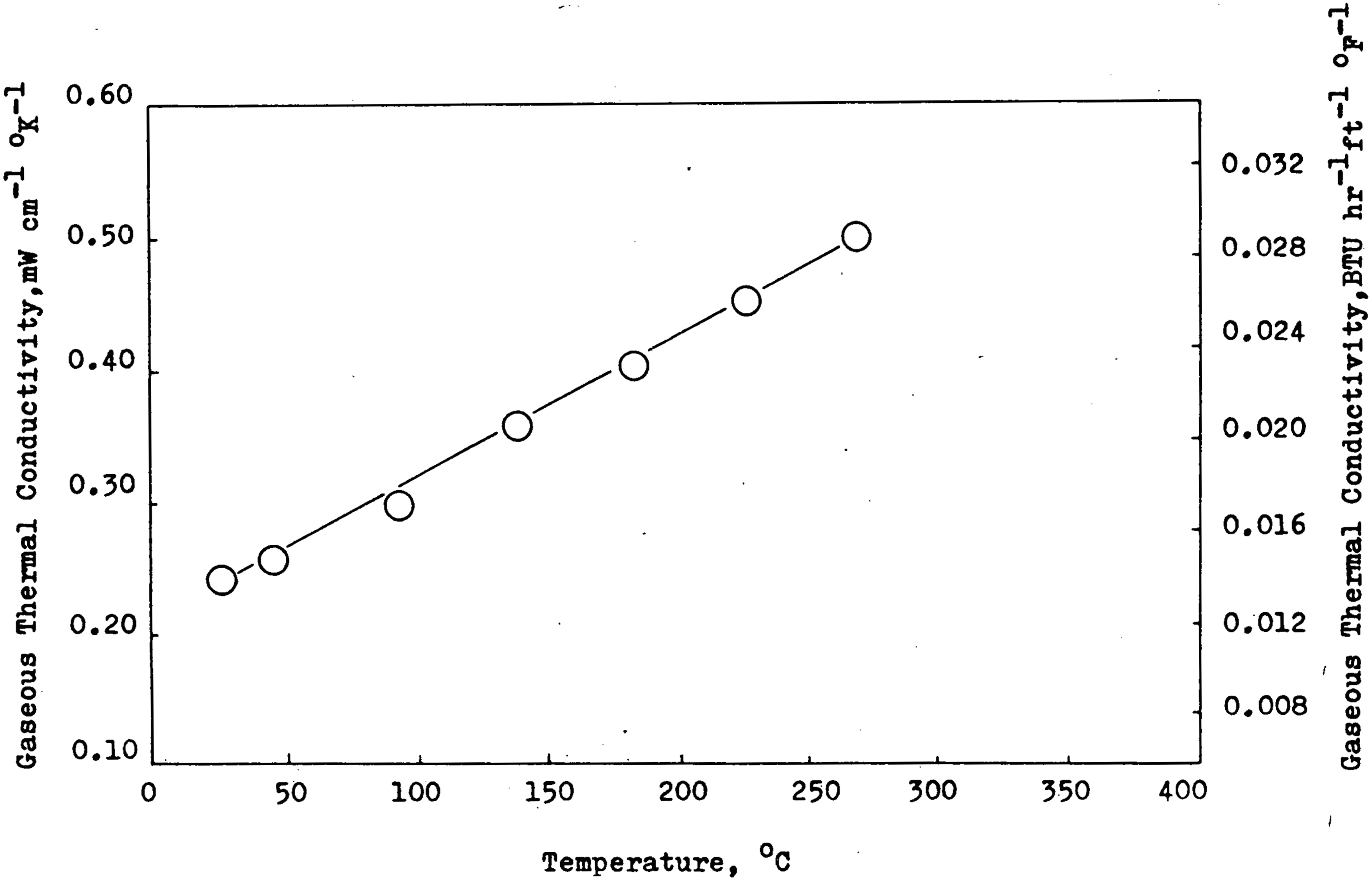

Figure IB-9 Gaseous Thermal Conductivity of Silane

Table IB-7 Gaseous Thermal Conductivity Values of Dichlorosilane

Temperature	Gaseous Thermal Conductivity		
^{o}C	mW cm^{-1} $^{o}K^{-1}$	Cal $cm^{-1} sec^{-1}$ $^{o}C^{-1}$	BTU $hr^{-1} ft^{-1}$ $^{o}F^{-1}$
28.0	0.102	24.43×10^{-6}	5.91×10^{-3}
45.7	0.108	25.72×10^{-6}	6.22×10^{-3}
94.7	0.129	30.86×10^{-6}	7.46×10^{-3}
139.4	0.148	35.42×10^{-6}	8.56×10^{-3}
184.1	0.169	40.37×10^{-6}	9.76×10^{-3}
227.4	0.194	46.46×10^{-6}	11.23×10^{-3}
269.5	0.217	51.79×10^{-6}	12.52×10^{-3}
311.3	0.243	58.15×10^{-6}	14.06×10^{-3}
350.6	0.267	63.70×10^{-6}	15.40×10^{-3}

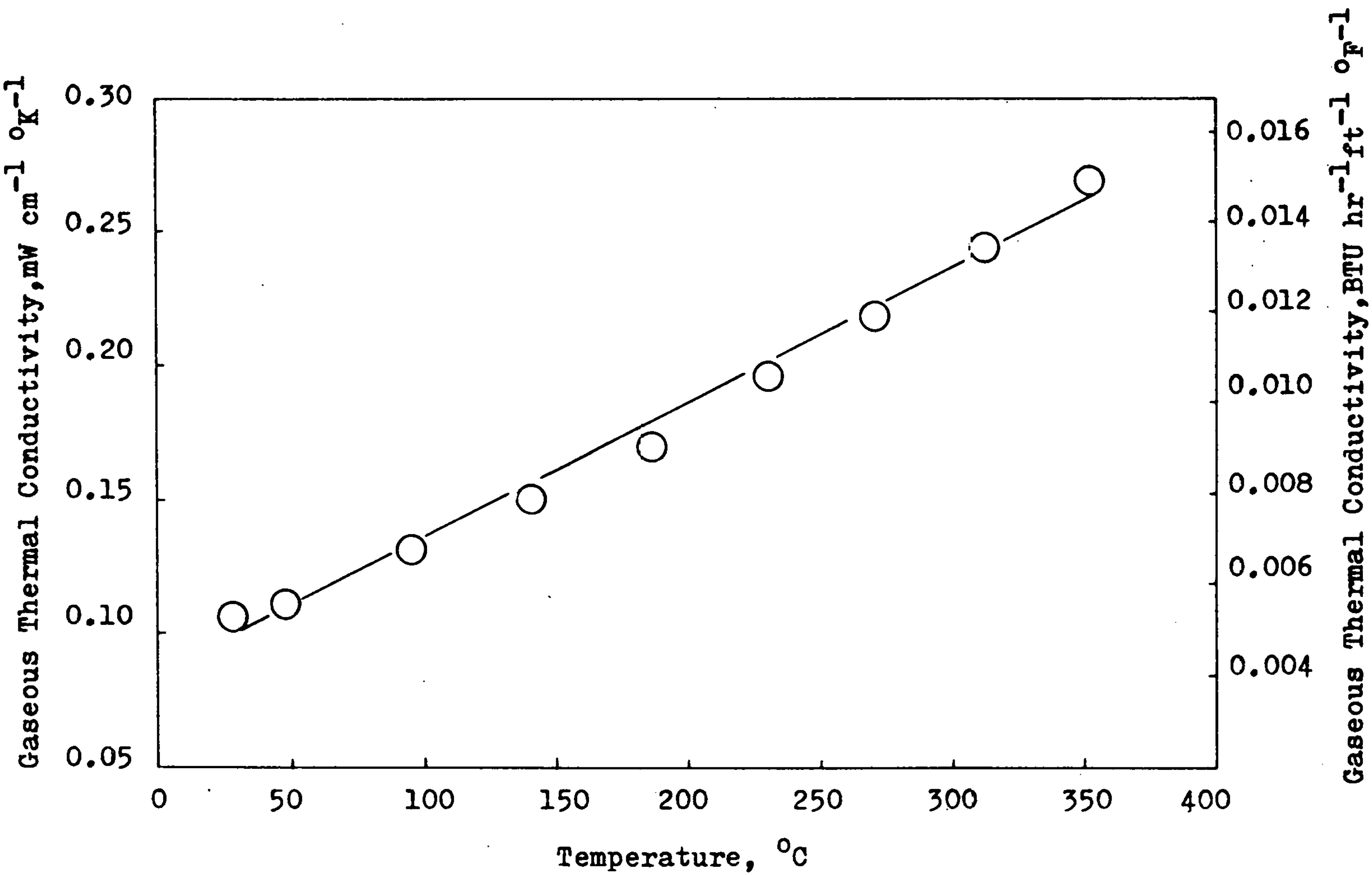

Figure IB-10 Gaseous Thermal Conductivity of Dichlorosilane

Table IB-8 Gaseous Thermal Conductivity Values of Trichlorosilane

Temperature	Gaseous Thermal Conductivity		
^{o}C	mW cm^{-1} $^{o}K^{-1}$	Cal $cm^{-1} sec^{-1}$ $^{o}C^{-1}$	BTU $hr^{-1} ft^{-1}$ $^{o}F^{-1}$
45.7	0.093	22.13×10^{-6}	5.35×10^{-3}
94.7	0.110	26.22×10^{-6}	6.34×10^{-3}
139.4	0.126	30.16×10^{-6}	7.29×10^{-3}
184.1	0.144	34.35×10^{-6}	8.30×10^{-3}
227.4	0.161	38.55×10^{-6}	9.32×10^{-3}
269.5	0.180	43.05×10^{-6}	10.41×10^{-3}
311.3	0.198	47.24×10^{-6}	11.42×10^{-3}
350.6	0.216	51.58×10^{-6}	12.47×10^{-3}

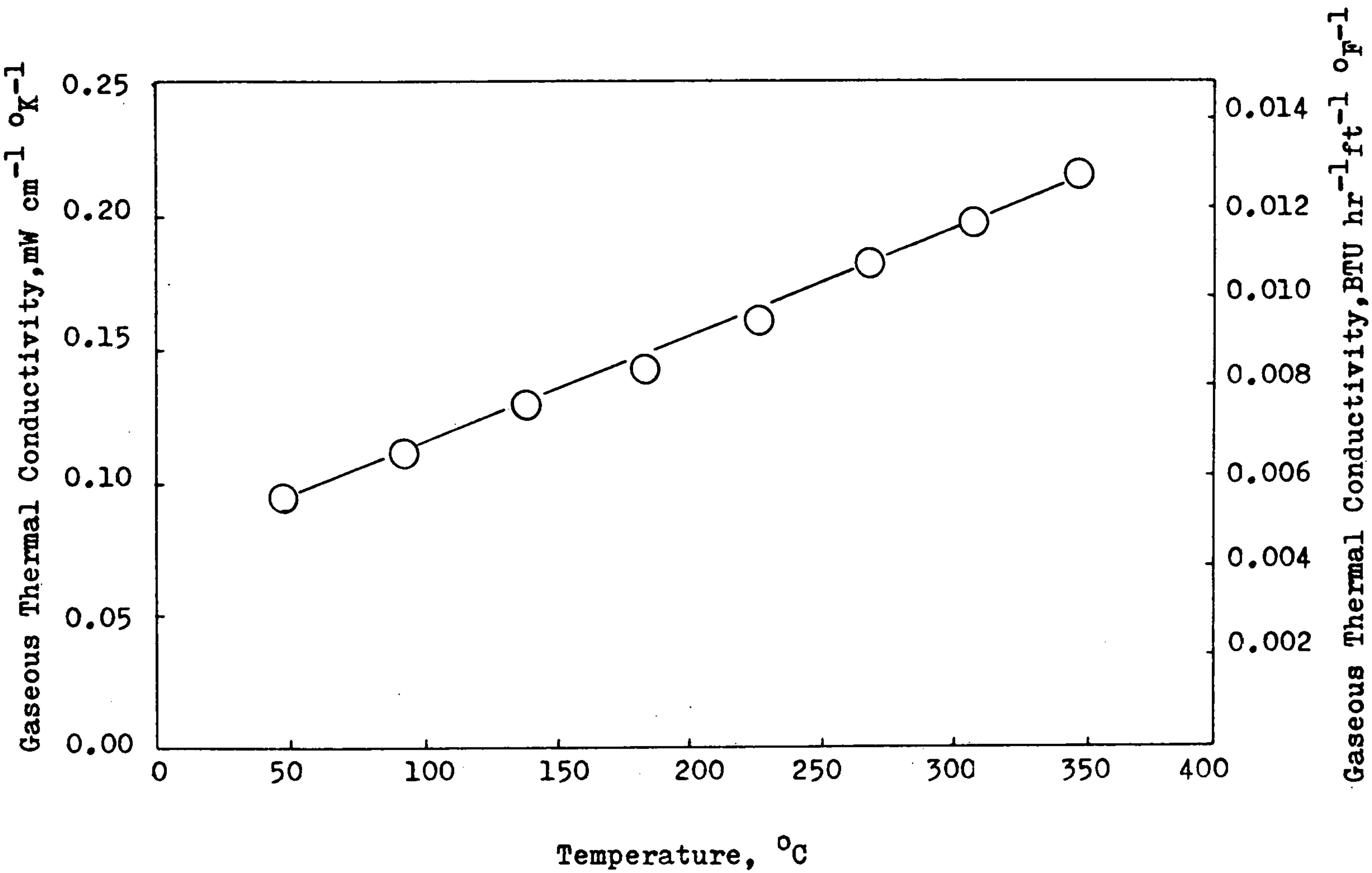

Figure IB-11 Gaseous Thermal Conductivity of Trichlorosilane

Table IB-9 Gaseous Thermal Conductivity Values of Tetrachlorosilane

Temperature	Gaseous Thermal Conductivity		
^oC	$mW\ cm^{-1}\ {}^oK^{-1}$	$Cal\ cm^{-1} sec^{-1}\ {}^oC^{-1}$	$BTU\ hr^{-1} ft^{-1}\ {}^oF^{-1}$
94.7	0.100	23.93×10^{-6}	5.78×10^{-3}
139.4	0.111	26.43×10^{-6}	6.39×10^{-3}
184.1	0.124	29.59×10^{-6}	7.15×10^{-3}
227.4	0.138	32.89×10^{-6}	7.95×10^{-3}
269.5	0.153	36.59×10^{-6}	8.85×10^{-3}
311.3	0.169	40.39×10^{-6}	9.76×10^{-3}
350.6	0.193	46.13×10^{-6}	11.15×10^{-3}

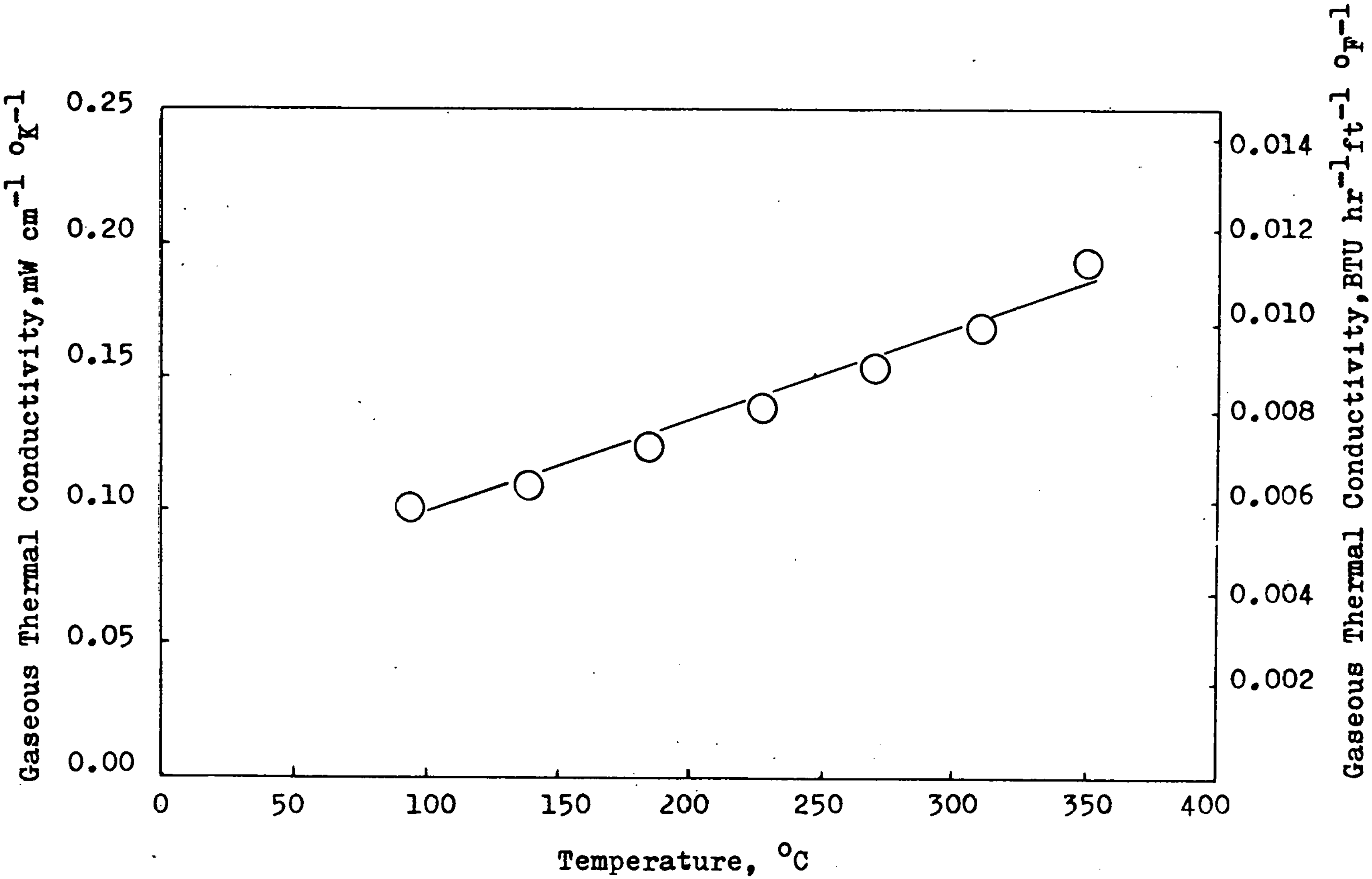

Figure IB-12 Gaseous Thermal Conductivity of Tetrachlorosilane

Table IB-10 Gaseous Thermal Conductivity Values of Tetrafluorosilane

Temperature	Gaseous Thermal Conductivity		
^oC	mW cm^{-1} $^oK^{-1}$	Cal $cm^{-1}sec^{-1}$ $^oC^{-1}$	BTU $hr^{-1}ft^{-1}$ $^oF^{-1}$
29.0	0.150	35.95×10^{-6}	8.69×10^{-3}
45.7	0.158	37.79×10^{-6}	9.13×10^{-3}
94.7	0.189	45.24×10^{-6}	10.94×10^{-3}
139.4	0.215	51.43×10^{-6}	12.43×10^{-3}
184.1	0.241	57.67×10^{-6}	13.94×10^{-3}
227.4	0.274	65.46×10^{-6}	15.83×10^{-3}
269.5	0.291	69.55×10^{-6}	16.81×10^{-3}
311.3	0.316	75.55×10^{-6}	18.26×10^{-3}
350.6	0.345	82.34×10^{-6}	19.90×10^{-3}

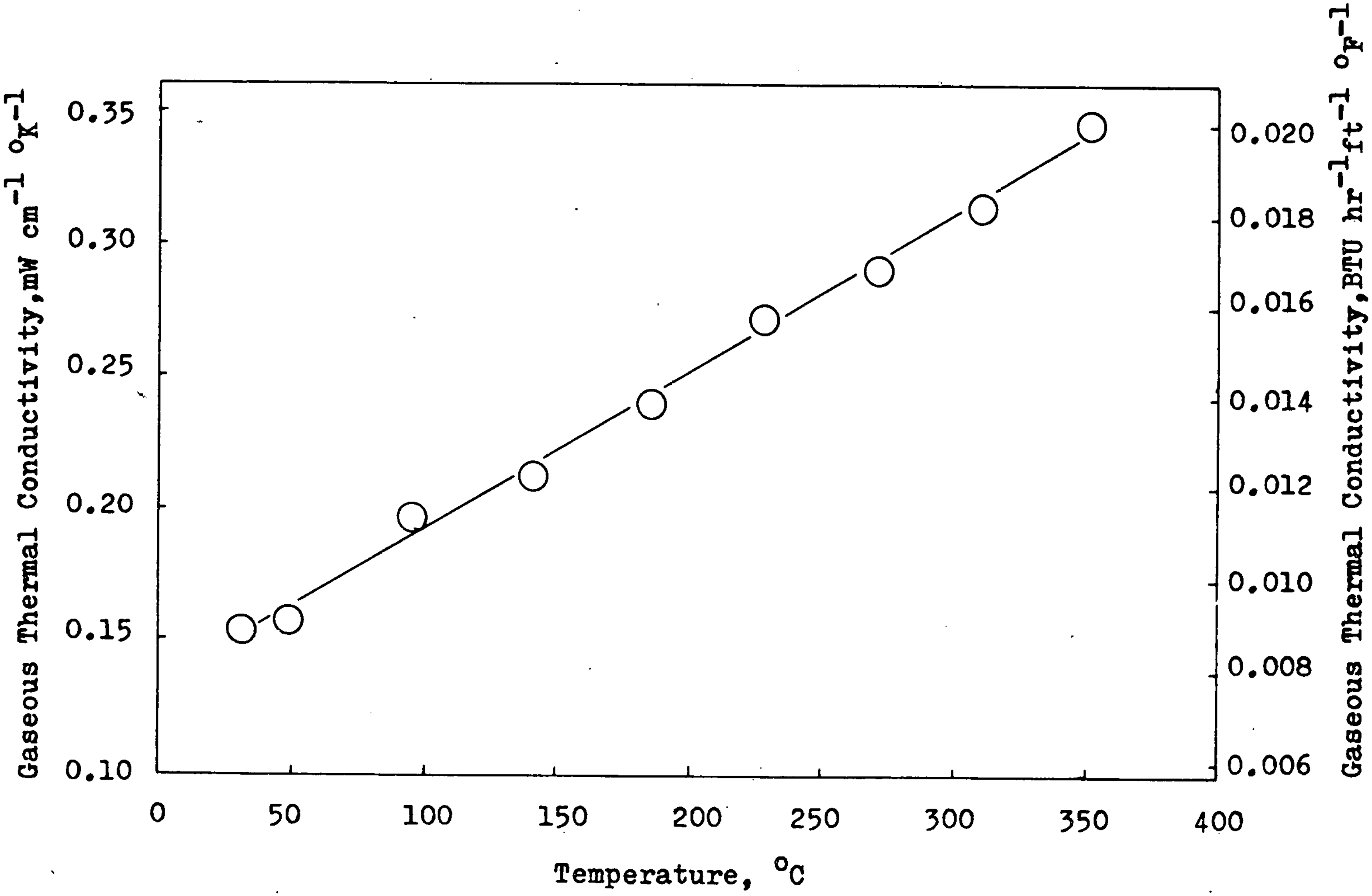

Figure IB-13 Gaseous Thermal Conductivity of Tetrafluorosilane

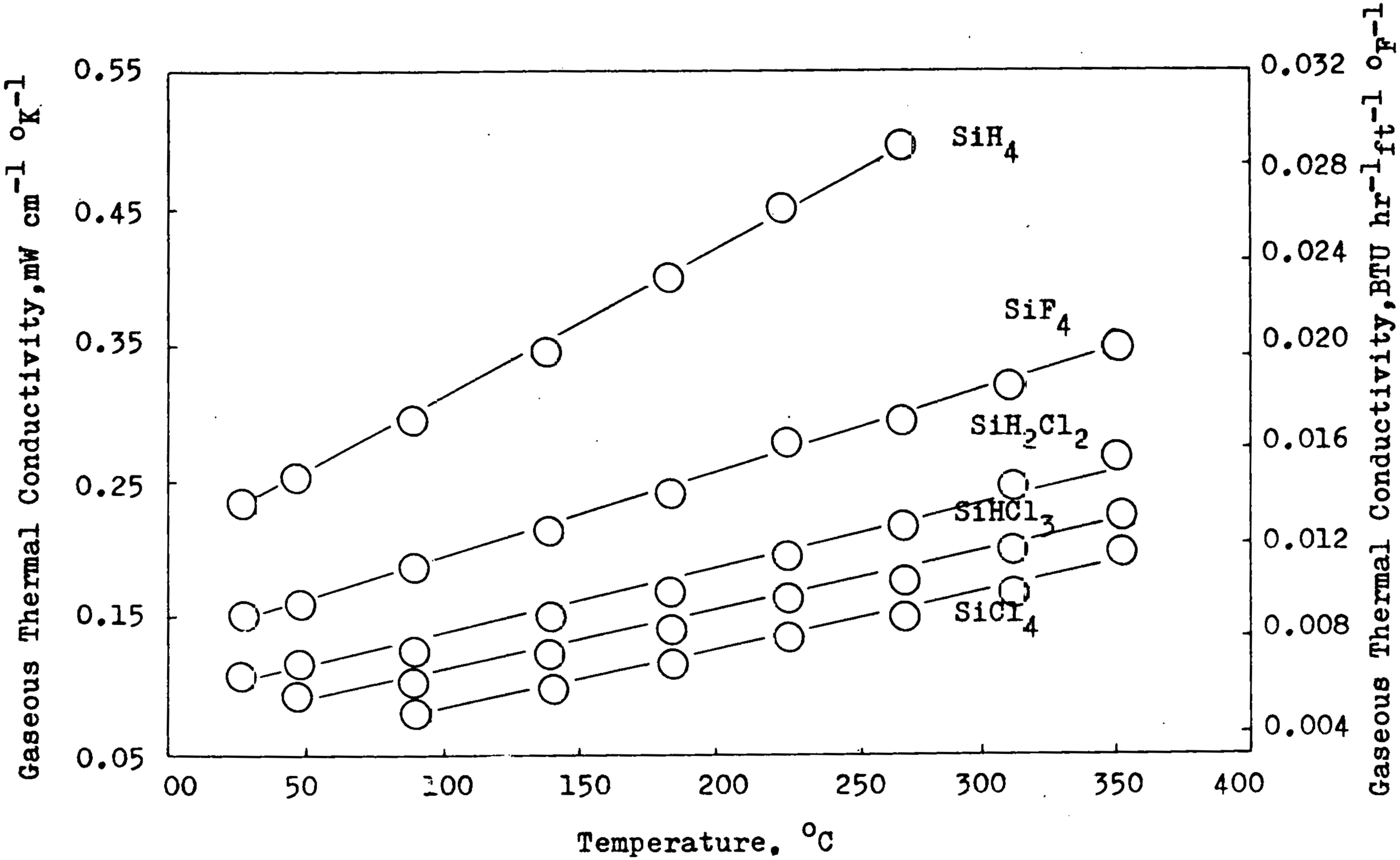

Figure IB-14 Gaseous Thermal Conductivity of Silane and Some Halogenated Silanes

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 Union Carbide silane process (Case C-Revised Process). The status, including progress since the last reporting period, for the process design is given below for key guideline items:

	Prior	Current
.Base Case Conditions	25%	95%
.Reaction Chemistry	10%	95%
.Process Flow Diagram	10%	90%
.Material Balance	10%	50%
.Energy Balance	10%	50%
.Property Data	10%	50%
.Equipment Design	0%	30%

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

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-1.1C
.Reaction Chemistry..................Table IIA-1.2C
.Redistribution Equilibrium..........Figure IIA-1.1C

In current activities, material balance, energy balance and equipment design are in progress for the mass transfer equipment. This includes engineering design of several distillation columns which separate the liquid chlorosilanes for recycle and provide the purified silane product.

CASE C

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

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

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

O Plan
◐ In Progress
● Complete

CASE C

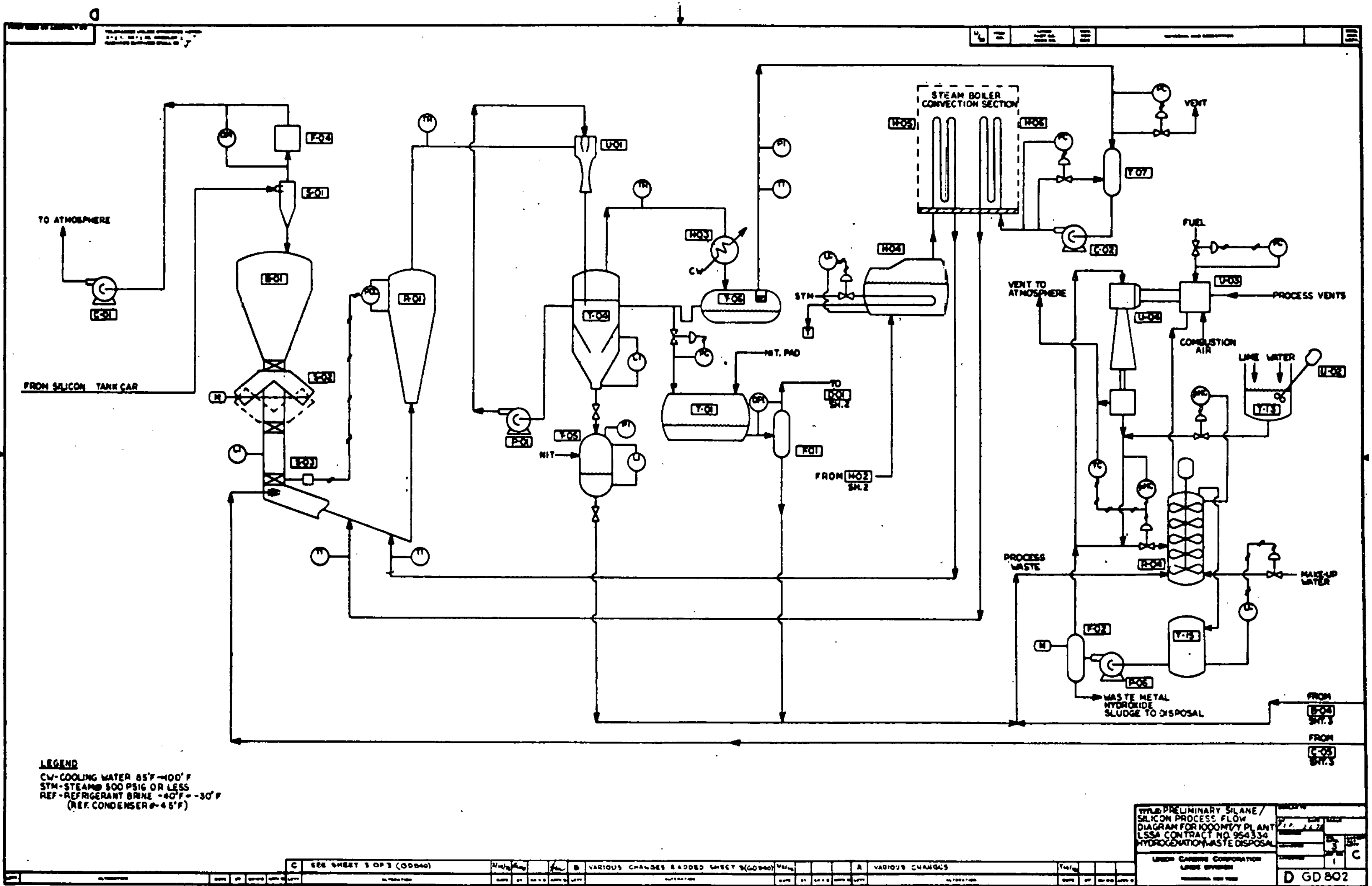

Figure IIA-1.0C Process Flow Sheet for Silane Process - CASE C (Revised Process)

CASE C

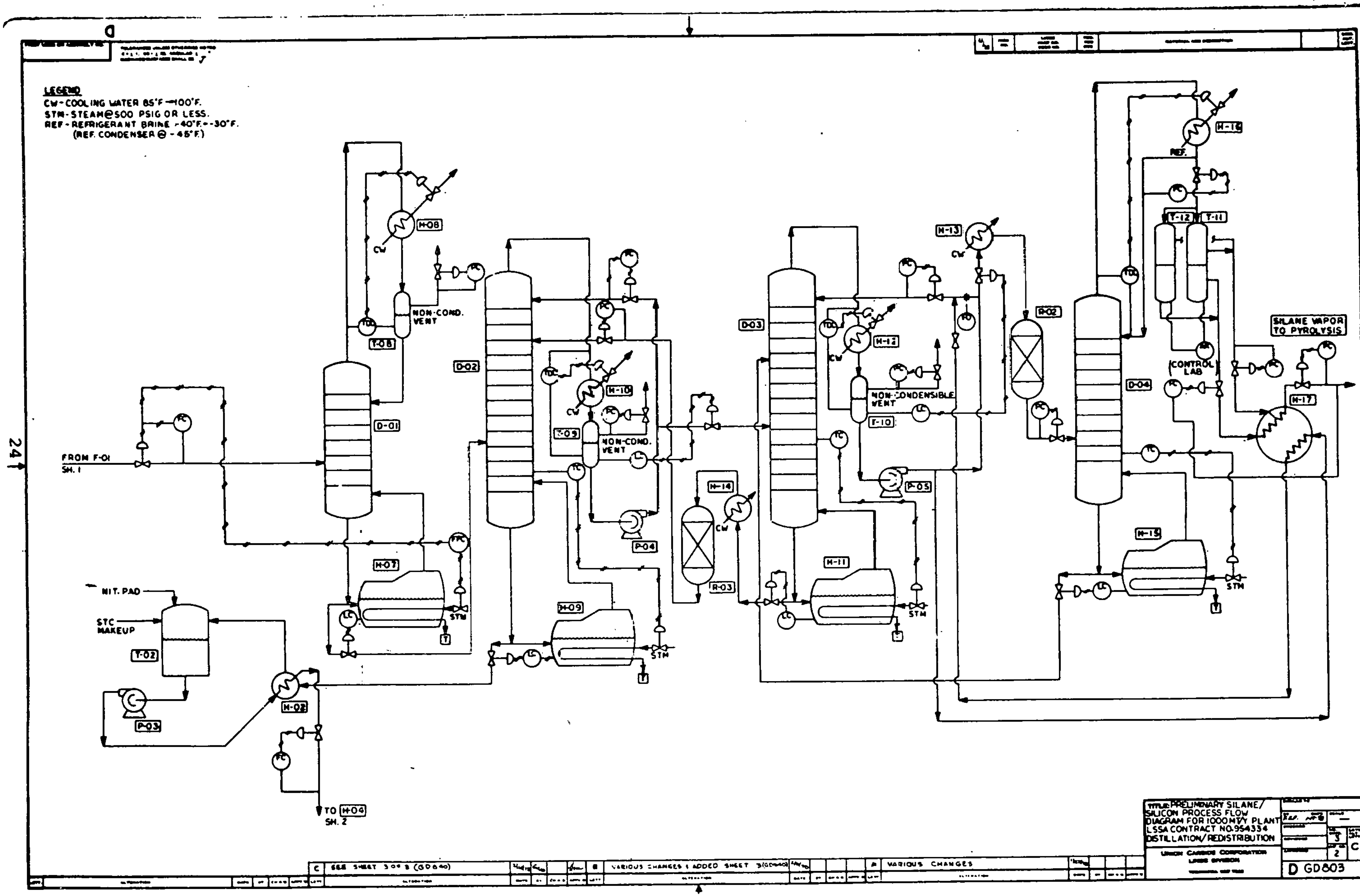

Figure IIA-1.0C (Continued)

24

CASE C

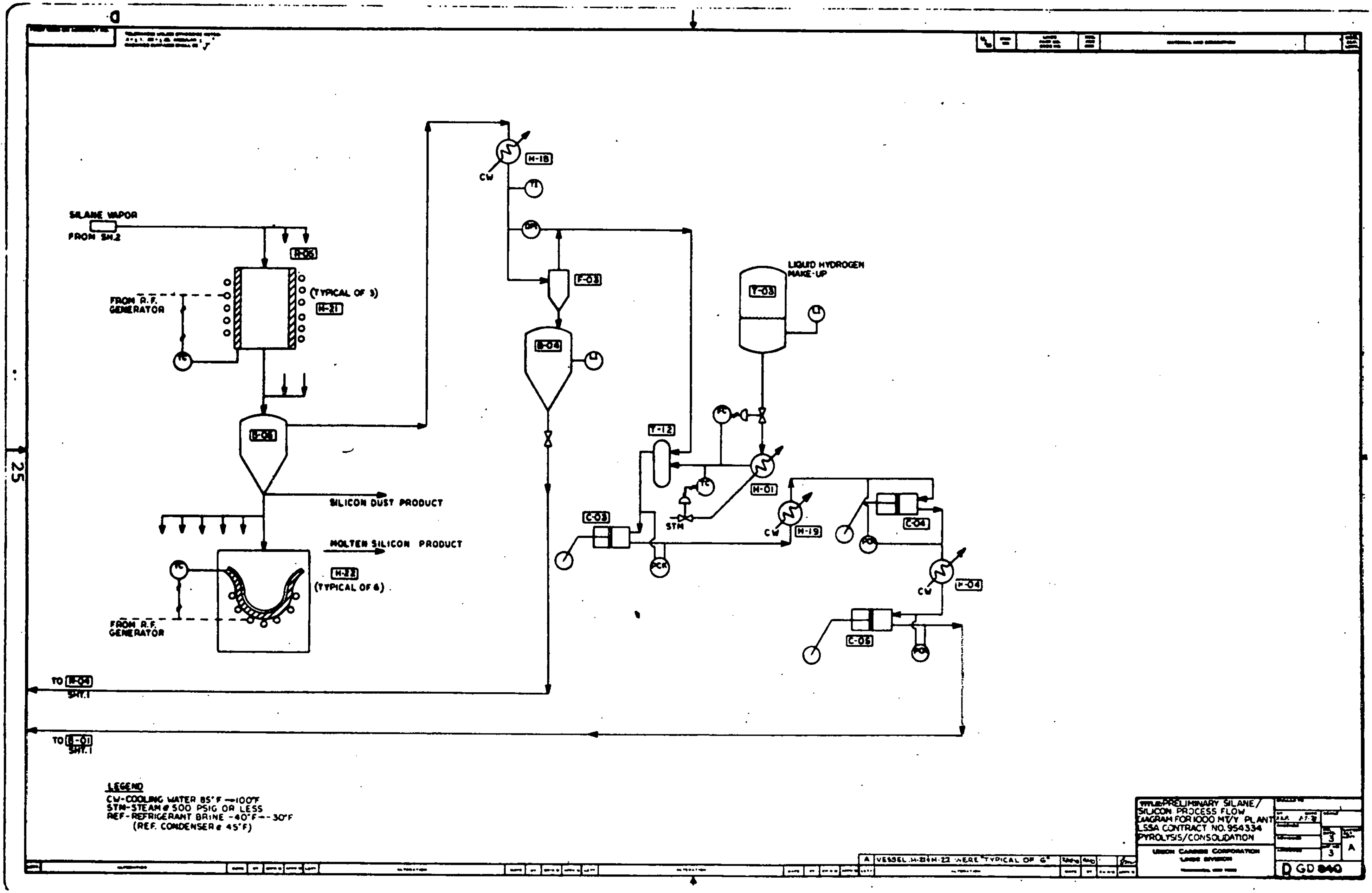

Figure IIA-1.0C (Continued)

CASE C

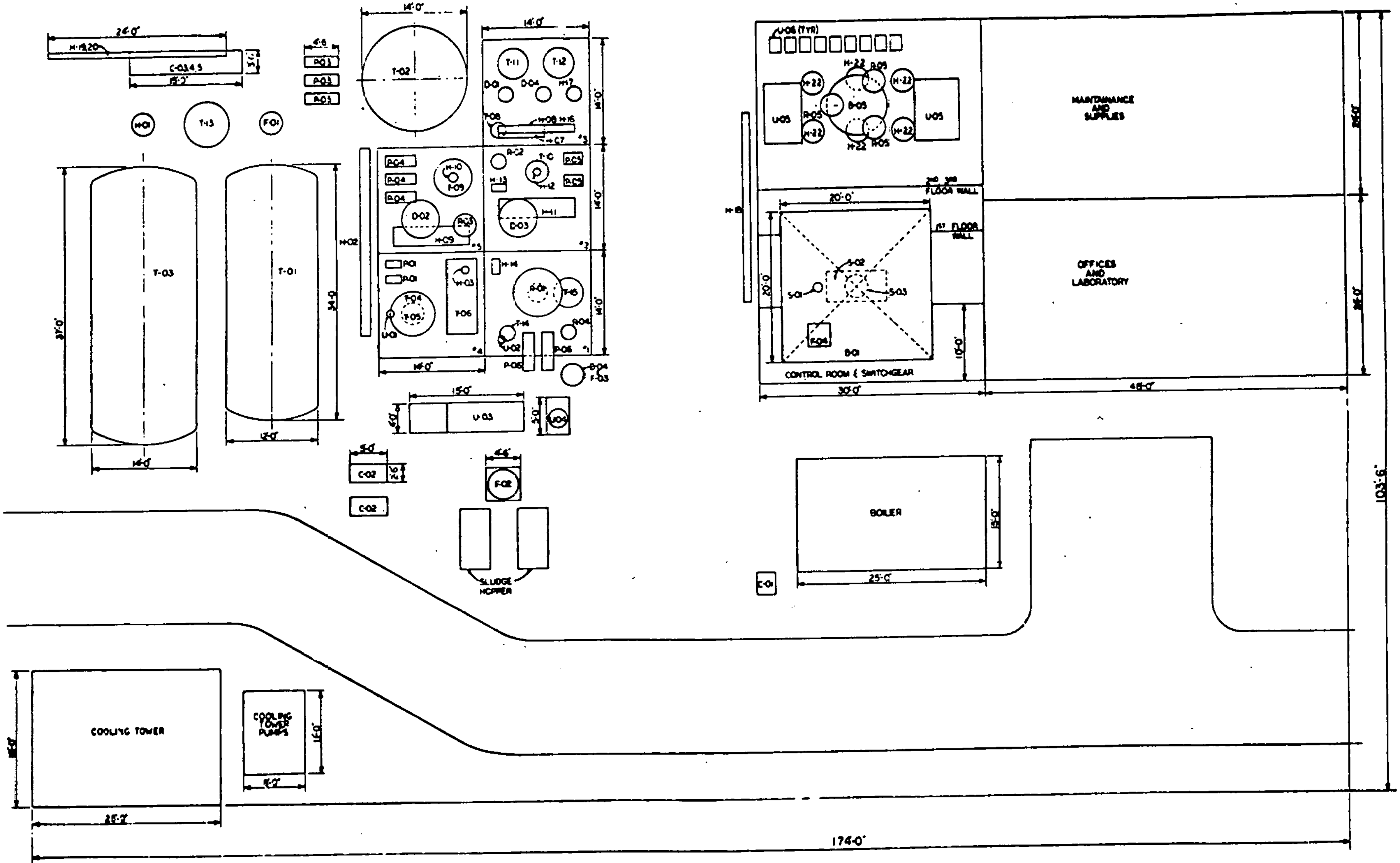

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Figure IIA-1.0C (Continued)

CASE C

TABLE IIA-1.1C

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

1. Plant Size
   - Silicon produced from silane
   - 1000 metric tons/year of silicon
   - Solar cell grade silicon

2. Hydrogenation Reaction
   - Metallurgical grade silicon, hydrogen, and recycle silicon tetrachloride (TET) used to produce trichlorosilane (TCS)
   - Copper catalyzed
   - Fluidized bed
   - 500PC, 154.7 psia
   - 20% to 22.5% conversion of $SiCl_4$ (example)

3. TCS Redistribution Reaction
   - TCS from hydrogenation produces dichlorosilane (DCS)
   - Catalytic redistribution of TCS with tertiary amine ion exchange resin
   - Liquid phase 85 psia, 140°F
   - Conversion a function of inlet concentration (Union Carbide equilibrium)
   - Conversion from pure TCS feed is about 9.5% to DCS (example)

4. DCS Redistribution Reaction
   - DCS produces SiH_4 (silane)
   - Catalytic redistribution of DCS with tertiary amine exchange resin
   - Liquid phase 510 psia, 140°F
   - Conversion a function of inlet concentration (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
   - Final purification by distillation
   - Designed to remove trace impurities (B_2H_6, example)

7. Operating Ratio
   - Approximately 85% utilization (on stream time)
   - Approximately 7445 hour/year production

8. Storage Consideration
   - Feed materials (several week supply, approx. 1 month)
   - Product (two shifts storage)
   - Process (several hours to 1 shift)

CASE C

TABLE IIA-1.2C

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

1. Hydrogenation Reaction

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

2. Trichlorosilane Redistribution Reaction

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

3. Dishlorosilane Redistribution Reaction

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

Note

1. Reaction 1 Product contains H_2, $SiCl_4$, $SiHCl_3$, SiH_2Cl_2 (trace), other trace chlorides
2. Reaction 2 Product conatins $SiHCl_3$, $SiCl_4$, SiH_2Cl_2, SiH_3Cl
3. Reaction 3 Product contains SiH_2Cl_2, $SiHCl_3$, $SiCl_4$, SiH_3Cl, SiH_4

CASE C

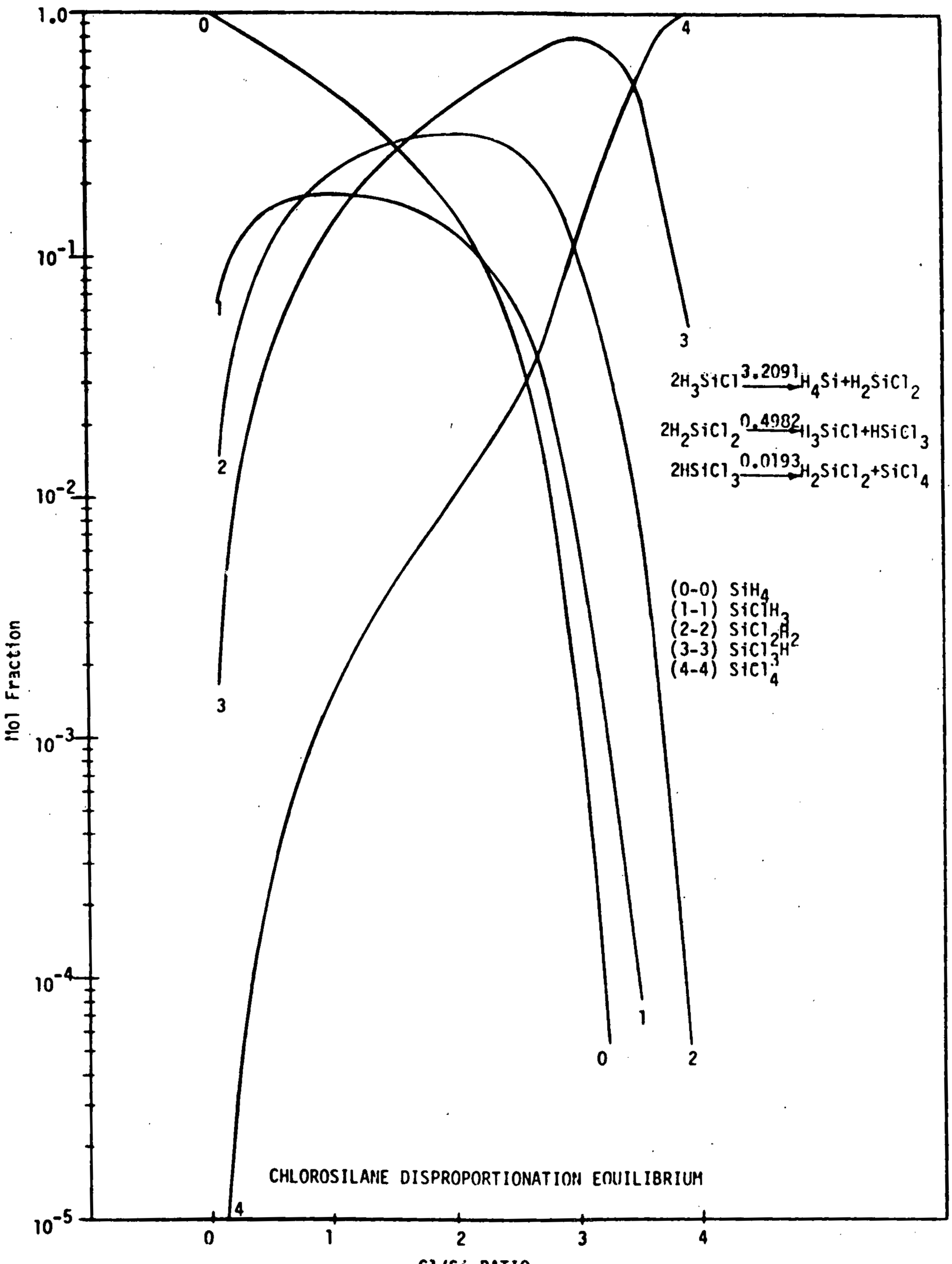

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Figure IIA-1.1C Redistribution Equilibrium For Silane Process-CASE C
(Provided by Union Carbide)

B. OTHER PROCESS

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

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

III. SUMMARY - CONCLUSIONS

The following summary-conclusions are made as aresult of major activities accomplished during this reporting period.

1. Task 1

Analysis of process system properties was continued for silicon source materials. Property data for silicon tetrachloride are reported for critical constants (temperature, pressure, volume, compressibility factor); vapro pressure; heat of vaporization; gas heat capacity and liquid heat capacity. Silicon tetrachloride is the source material in several processes under consideration for solar cell grade silicon production.

The experimental determination of gaseous thermal conductivity values for silicon source materials is now finished with final values being reported in the temperature range 25°C to 350°C. Thermocouples used to monitor the temperture of the thermal conductivity cell were calibrated and these calibrated temperture values used to obtain final thermal conductivity values.

2. Task 2

Chemical engineering analysis of the Union Carbide silane process (Case C-Revised Process) was continued with primary efforts being devoted to the preliminary process design. Status and progress are reported for base case conditions, process flow diagram, reaction chemistry and equipment design. Current engineering design is in progress for the several distillation columns which separate the liquid chlorosilanes and provide prufied silane product.

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.

Initiate preliminary investigation for the measurement of gaseous viscosity of silicon source materials.

2. Task 2

Continue preliminary process design of the Union Carbide silane process (Case C-Revised Process).

3. Task 3

Initiate cost analysis of the Union Carbide silane process (Case C-Revised Process).

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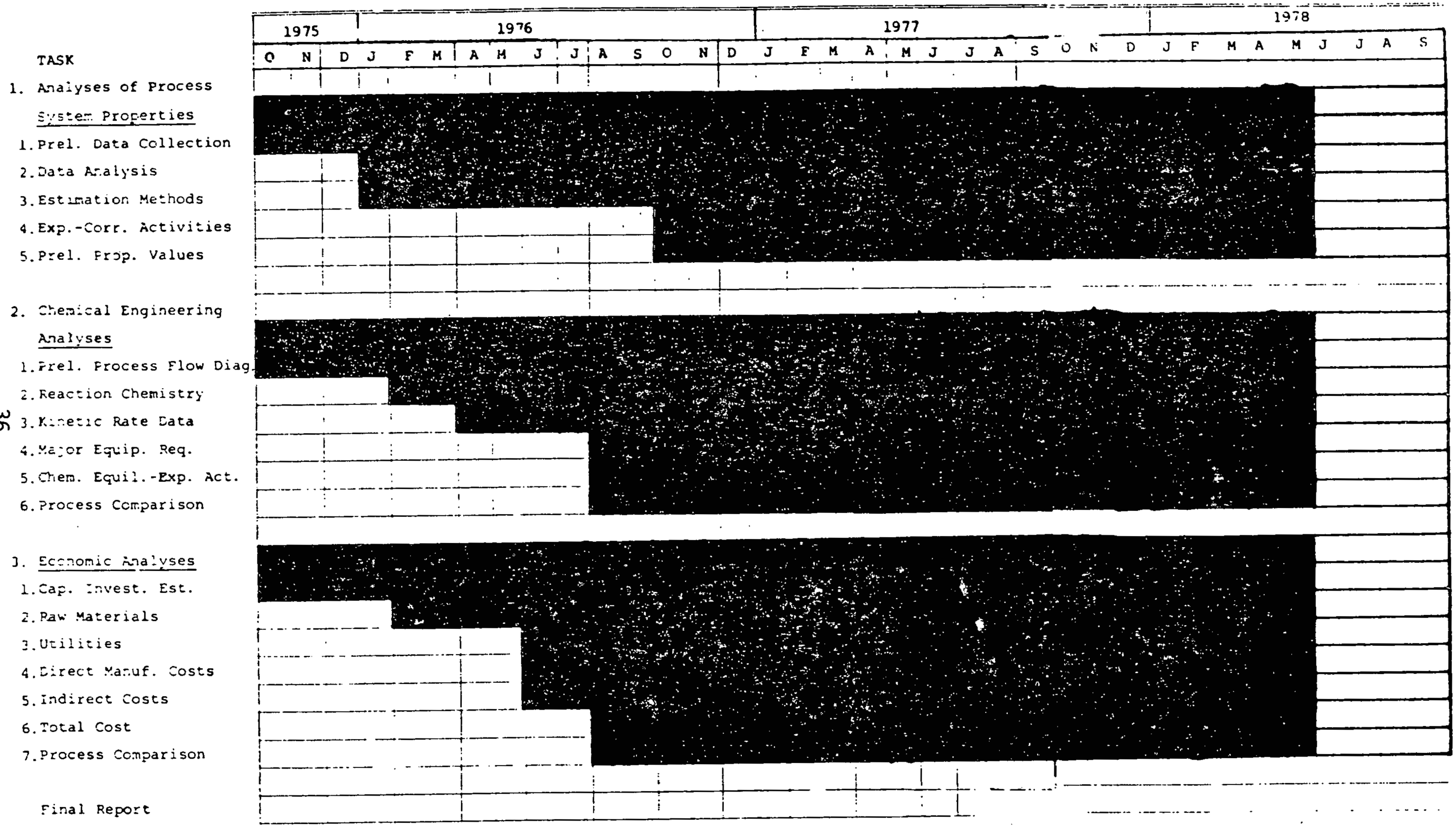

PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL TASK I

JPL Contract No. 954343

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