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PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL TASK I

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DISCLAIMER

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ABSTRACT

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

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

In the viscosity investigation, experimentally determined values for gas viscosity of trichlorosilane and dichlorosilane are reported in the temperature range of 40°C to 200°C. Previous data are not available in this temperature range for either compound.

Chemical engineering analysis of the BCL 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; material and energy balances; and major process equipment design. Current engineering efforts are nearing completion for manpower estimate of production labor requirements for the plant.

TABLE OF CONTENTS

I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

A. SILICON TETRAFLUORIDE PROPERTIES

Analysis of process system properties was continued for silicon source materials under consideration for solar cell grade silicon production during this reporting period. Major efforts centered on property data of silicon tetrafluoride which is involved in the process operations in several alternate processes for solar cell grade silicon.

Physical Properties and Critical Constants (Table IA-1)

Physical properties are listed in Table IA-1 including the boiling point (sublimation temperature, where the vapor pressure of the solid is 760 mm Hg.). Two temperature are given in the literature for the melting point (triple point) of silicon tetrafluoride based on the work of Patnode and Papish (F37) in 1930 and the work of Pace and Mosser (F36) in 1963. The more recent work was selected as the melting point (triple point, where solid, liquid and vapor are in equilibrium).

Experimental data for the critical temperature and critical pressure of silicon tetrafluoride have been determined (F4). The critical compressibility factor, Z_c, was estimated from the Garcia'-Barcena' correlation (F39):

$$Z_c = f(T_b) - g(T_b/M) \qquad \text{(IA-1)}$$

where T_b is the normal boiling point, °K, and M is the molecular weight. When this method was applied to sulfur hexafluoride, another subliming inorganic fluoride, the calculated value of the critical compressibility factor only deviated 3.3% from the known value. From the estimated value of Z_c, the critical volume, V_c, was calculated by the rearrangement of the definition of Z_c.

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

Vapor Pressure (Figure IA-1)

The vapor pressure of silicon tetrafluoride has been determined experimentally from 50° below the sublimation point to near the critical point (F4, F36, F37, F41). The experimental data were extended to cover the liquid range (from triple point to critical point), and for the solid (below the triple point using the YSSP correlation in each case:

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

At the higher temperatures the deviation of experimental and correlation results are 2% while the average percent error was 4.8% in the lower temperature range. Much of the deviation is due to rather poor agreement in the literature for the experimenal values.

Heat of Vaporization (Figure IA-2)

Heat of vaporization data for silicon tetrafluoride are reported near the triple point (F24, F29, F36, F41, F60). Using the selected value (F36), Watson's correlation (F39) was used to extend the heat of vaporization over the entire liquid phase:

$$\Delta H_v = \Delta H_{v_1} \left[\frac{T_c - T}{T_c - T_1}\right]^n \qquad \text{(IA-4)}$$

where n = .38 and T_1 is the boiling point. The value of H_{v_1} was effectively confirmed using the Clausius-Clapyron equation and literature vapor pressure values(F36).

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

Heat capacity of the ideal gas at low pressure has been calculated from 0°C to 1200°C (F48, F63). These values, including other values covering smaller temperature ranges (F11, F24, F26, F29, F35, F60), were taken from various structural and spectral data and are in close argeement. The JANAF values (F48) were selected.

The liquid heat capacity of silicon tetrafluoride is reported near the sublimation point (F36). The values are extended over all liquid temperatures by the relationship:

$$\text{Heat Capacity x Density = Constant} \qquad \text{(IA-5)}$$

The estimated constant was 0.473. Testing of this relationship with available data (4 data points) for silicon tetrafluoride produced an average absolute deviation of 0.8% error.

Liquid Density (Figure IA-5)

Liquid density data for silicon tetrafluoride are available only within about 20 degrees of the triple point (F26, F60). The experimental data was extrapolated to the critical point by use of a modification of the Rackett equation (F65, F66):

$$\rho = \rho_c Z^{-(1-T_r)^{2/7}} \qquad \text{(IA-6)}$$

where ρ_c is the critical density, T_r is the reduced temperature, and Z is a parameter derived from available data. Comparison of the calculated and experimental values of 5 data points gave 0.57% average absolute error.

Surface Tension (Figure IA-6)

Experimental data are not available for the surface tension of silicon tetrafluoride. The Brock and Bird corresponding states technique was used to estimate the surface tension (F39):

$$\alpha = P_c^{2/3} T_c^{1/3} (0.133\ \alpha_c - 0.281)(1-T_r)^{11/9} \qquad \text{(IA-7)}$$

where α is surface tension, dynes/cm; α_c is the Riedel parameter; P_c is critical pressure, atm; T_c is the critical temperature, °K; and T_r is the reduced temperature. Application of this technique to silicon tetrachloride and sulfur hexafluoride gave results within 4% and 1% absolute deviation with experimental data, respectively.

Viscosity (Figures IA-7 and IA-8)

Experimental data for the gas viscosity of silicon tetrafluoride are available from about room temperature to above 300°C (F13, F32). The values at higher temperaures were estimated using the relationship:

$$\log \eta_G = A + BT + CT^2 \qquad \text{(IA-8\_}$$

The average absolute percentage error was 1.74% when correlated values were compared with the 28 experimental data points.

No experimental data is available for the liquid viscosity of silicon tetrafluoride. Estimates were derived applying the Letsou-Stiel high-temperature liquid-viscosity correlation (F65):

$$\eta_L \xi = (\eta_L \xi)^0 + \omega (\eta_L \xi)^1 \qquad \text{(IA-9)}$$

where the parameters $(\eta_L \xi)^0$ and $(\eta_L \xi)^1$ are functions of reduced temperature, ω is the acentric factor and $\xi = T_c^{1/6}/M^{1/2} P_c^{2/3}$. This correlations gave results within 17% and 48% absolute deviaiton for the experimental values of silicon tetracloride and sulfur hexafluoride, respectively. Since liquid viscosity estimation methods may be grossly inaccurate (F69), these values must be assumed to be order of magnitude estimates only.

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

The gaseous thermal conductivity of silicon tetrafluoride has been reported from about room temperature to 350°C (F9, F68). The experimental values were extended using a modified form of the Misic and Thodos correlation (F66):

$$\lambda_G = \frac{C_\rho}{\Gamma}(10^{-6})(14.52T_r - 5.14)^n \qquad \text{(IA-10)}$$

where n = .63, Γ is $T_c^{1/6}M^{1/2}/P_c^{2/3}$, T_r is the reduced temperature and C_p is the gaseous heat capacity. The average percentage error was less than one percent.

No liquid thermal conductivity data is available; however, values were estimated using the Sato-Riedel equation (65):

$$\lambda_L = \frac{2.64 \times 10^{-3}}{M^{1/2}} \quad \frac{3 + 20(1-T_r)^{2/3}}{3 + 20(1-T_{r_b})^{2/3}} \qquad \text{(IA-11)}$$

where M is molecular weight, T_r is the reduced temperature, and T_{r_b} is the reduced temperature at the boiling point. This correlation gave 34% error with the single experimental data point for silicon tetrachloride and 24% error for sulfur hexafluoride with the several experimental data points. There is considerable deviation of values among the several different data sources (22% maximum deviation). The present results should be taken only to represent an order of magnitude estimate.

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

Many American workers (F20, F24, F29, F34, F35, F48, F57, F58) and others (F40, F43) have reported heats of formation was well as Gibb's free energy of formation (F48, F56, F58) for the ideal gas. The JANAF values (F48) were selected.

TABLE IA-1

PHYSICAL PROPERTIES AND CRITICAL CONSTANTS OF SILICON TETRAFLUORIDE

Identification	Silicon Tetrafluoride
Formula	SiF_4
State (std. cond.)	gas (colorless)
Molecular weight, M	104.08
Boiling point, T_b, °C (sublimation point)	-95.7 (760 mm Hg)
Melting Point, T_m, °C (triple point)	-86.8 (1679 mm Hg)*Ref. F36 -90.2 (1318 mm Hg) Ref. F37
Critical Temp, T_c, °C	-14.15
Critical Pressure, P_c, atm	36.66
Critical Volume, V_c, cm^3/gr mol	165**
Critical Compressibility Factor, Z_c	0.284**
Critical Density, ρ_c. gr/cm^3	0.6308**
Acentric Factor (ω)	0.4086

* Selected Value

**Estimated

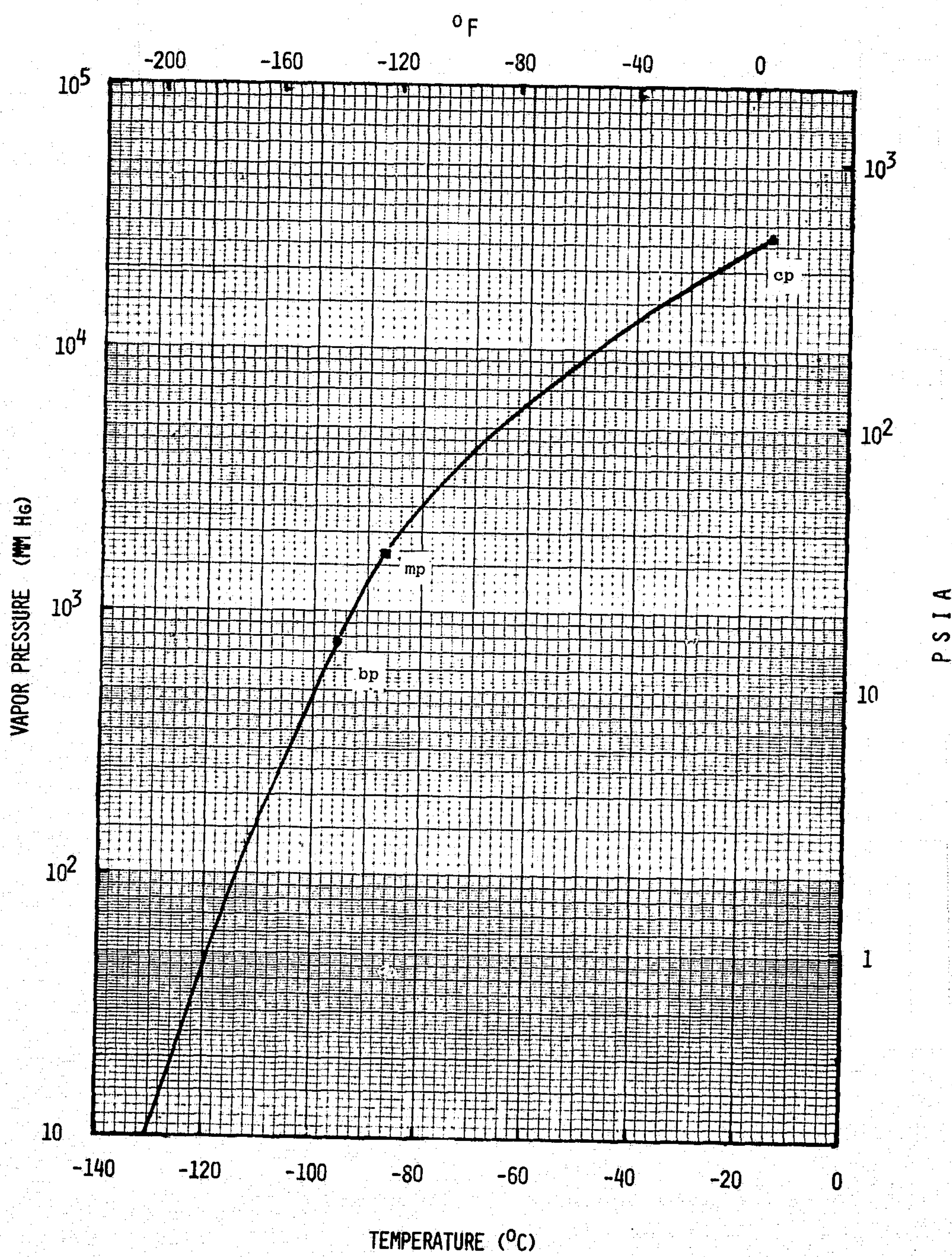

Figure IA-1. Vapor Pressure vs Temperature for Silicon Tetrafluoride

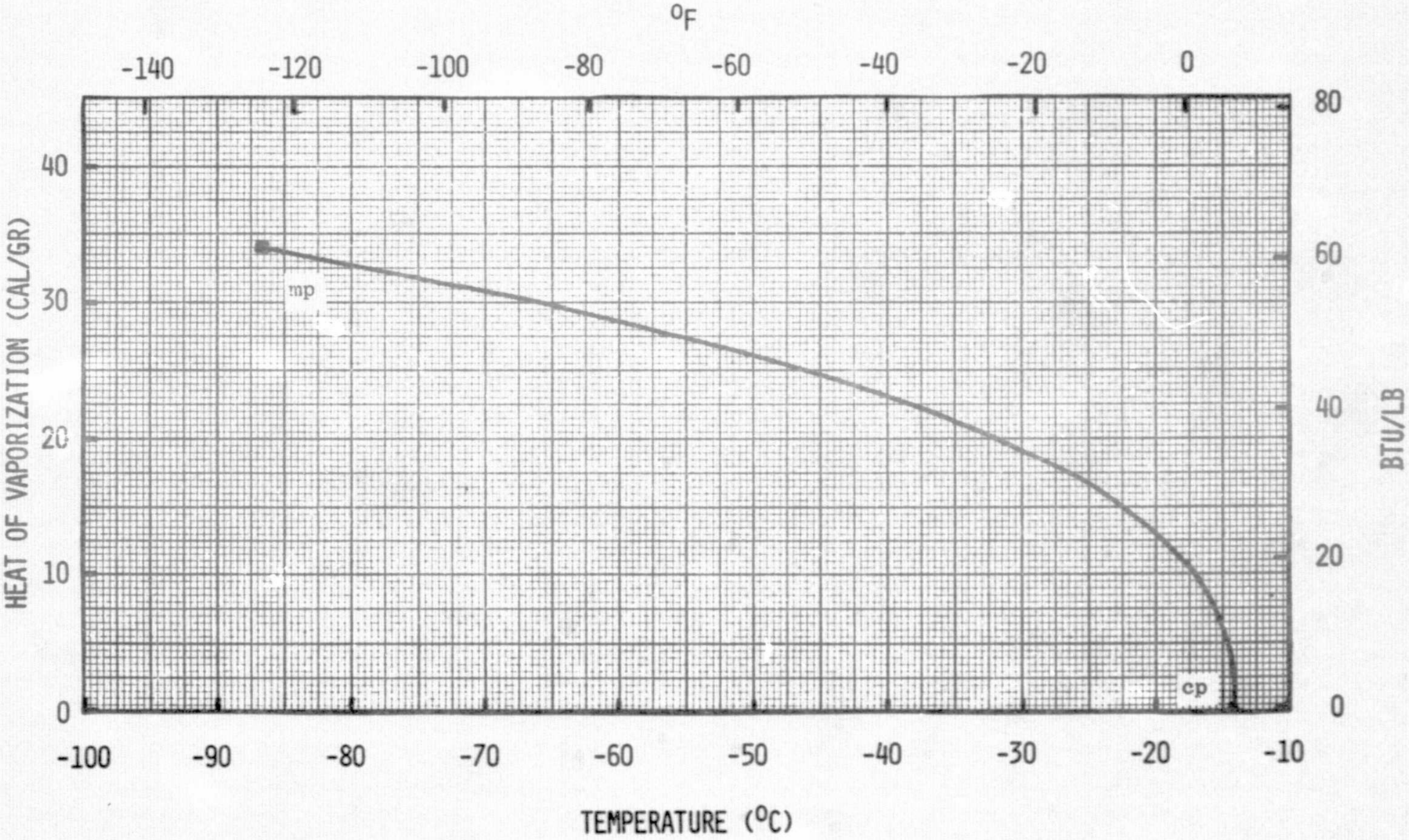

Figure IA-2. Heat of Vaporization vs Temperature for Silicon Tetrafluoride

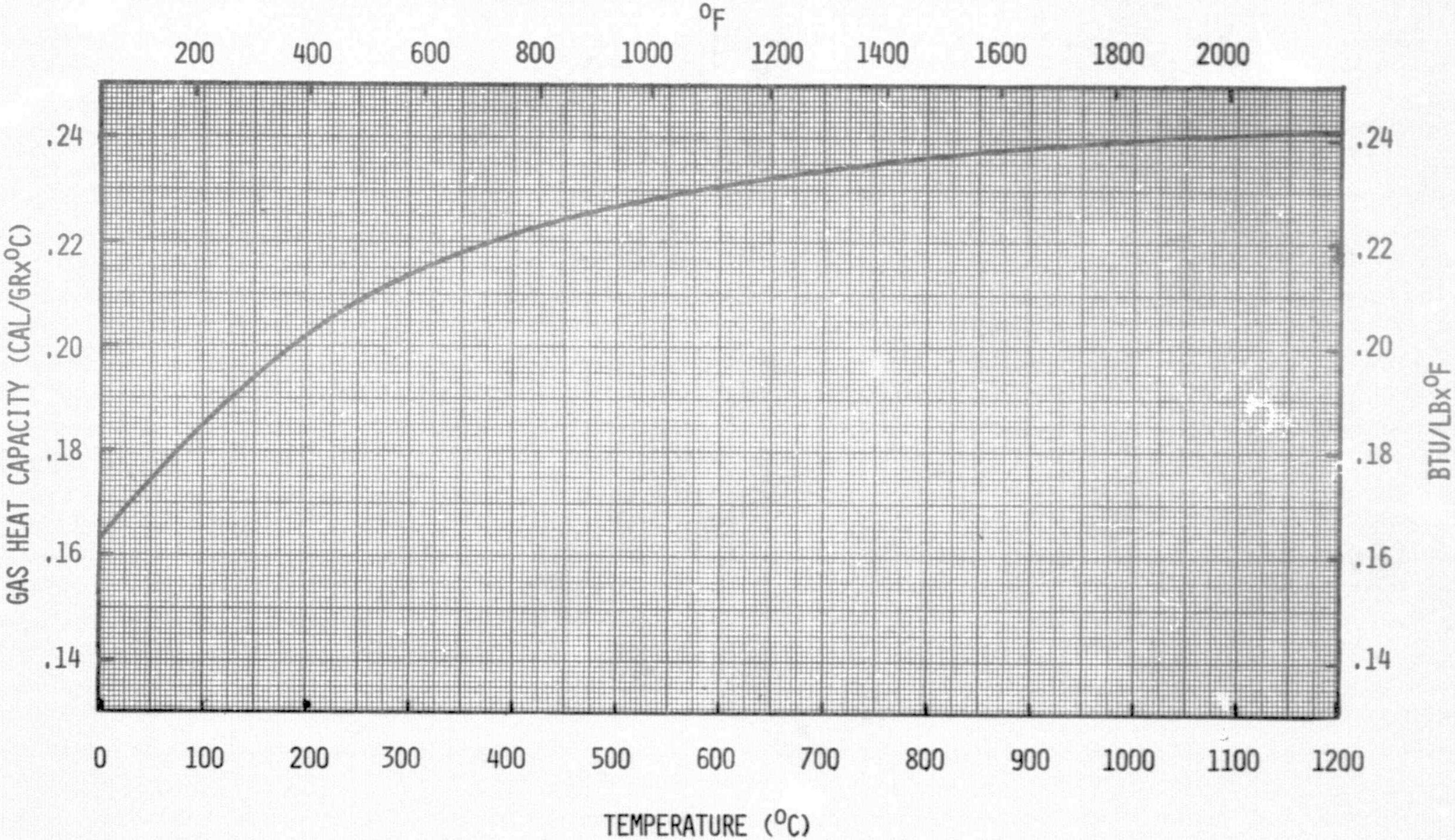

Figure IA-3. Gas Heat Capacity vs Temperature for Silicon Tetrafluoride

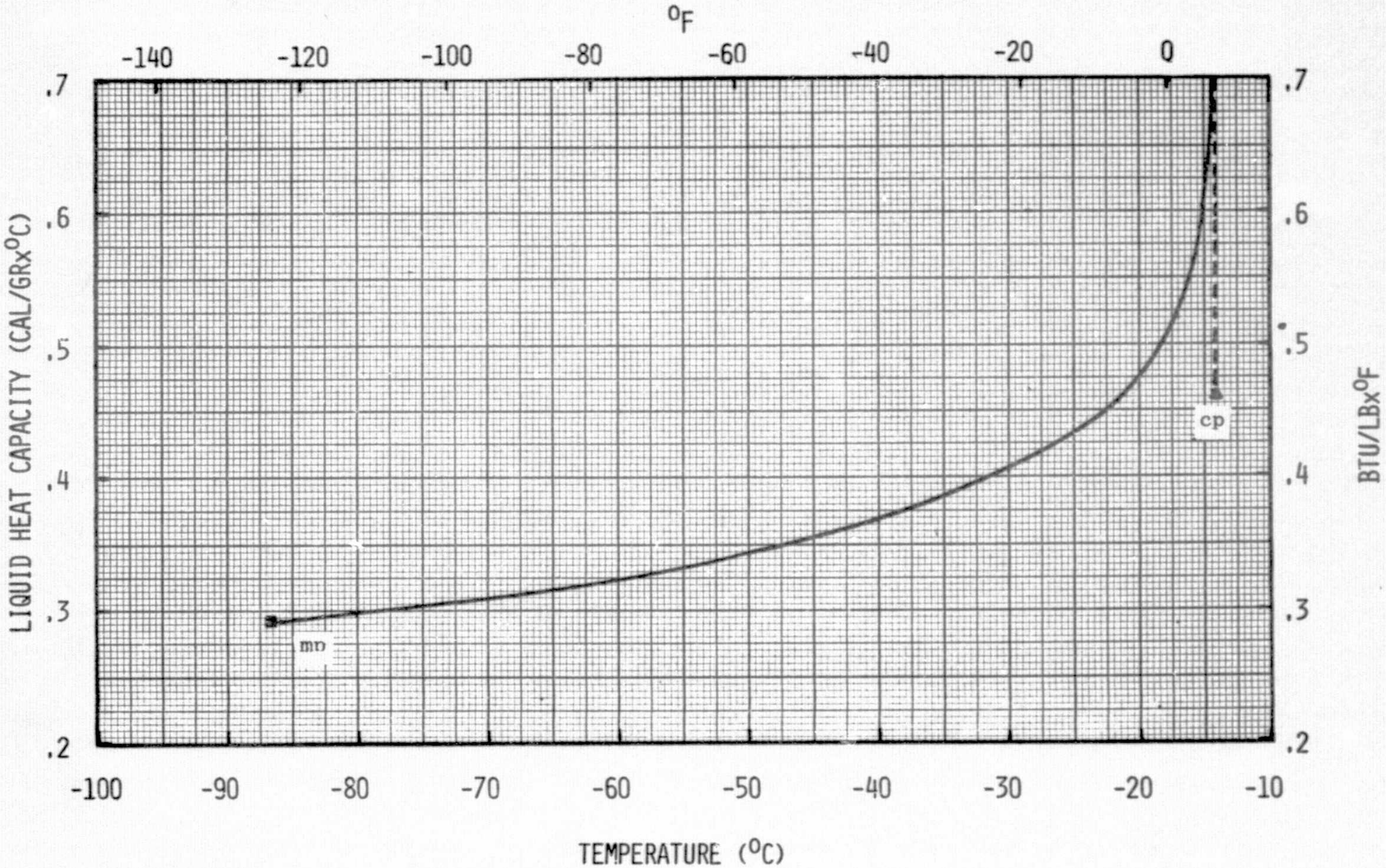

Figure IA-4. Liquid Heat Capacity vs Temperature for Silicon Tetrafluoride

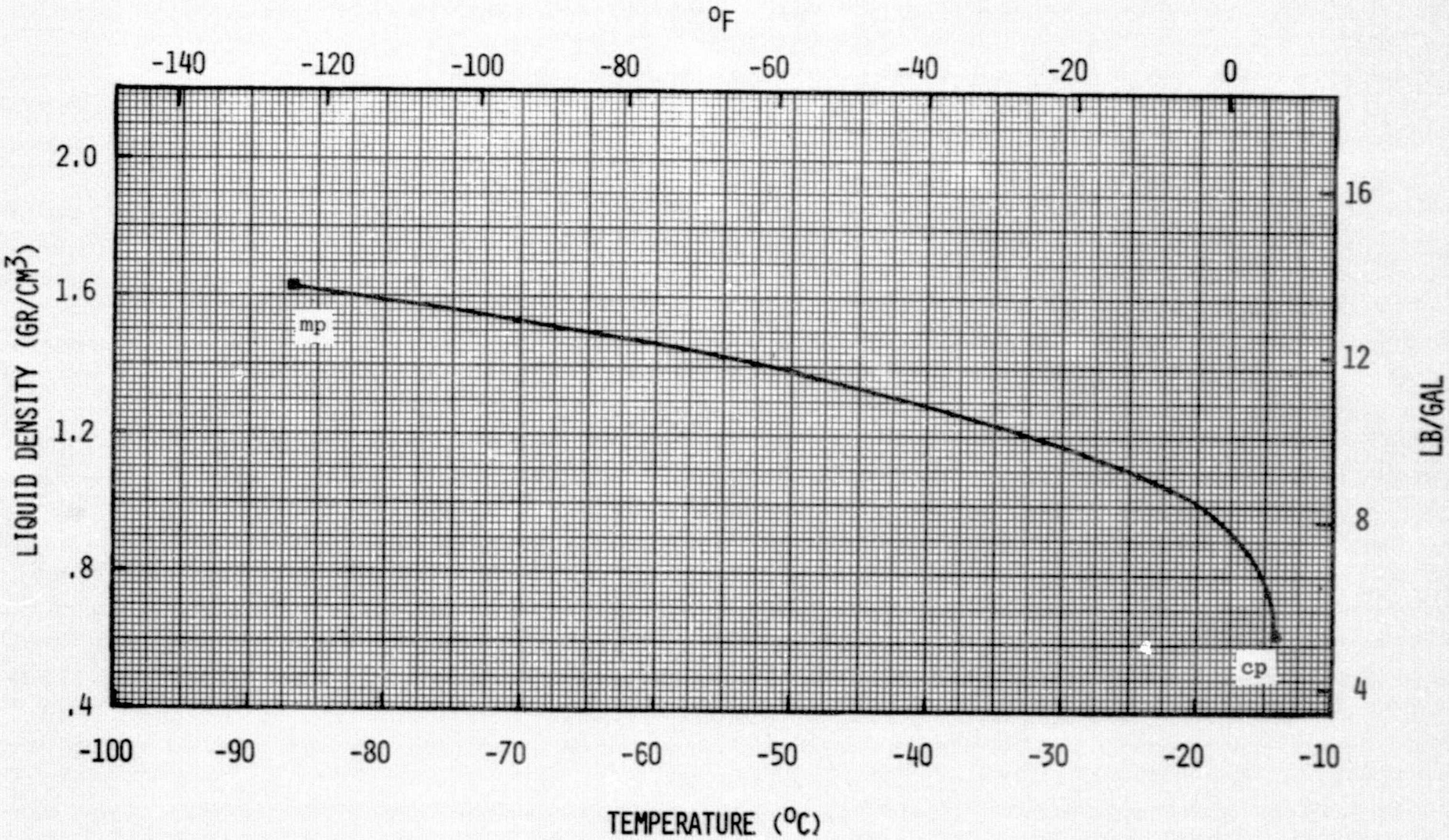

Figure IA-5. Liquid Density vs Temperature for Silicon Tetrafluoride

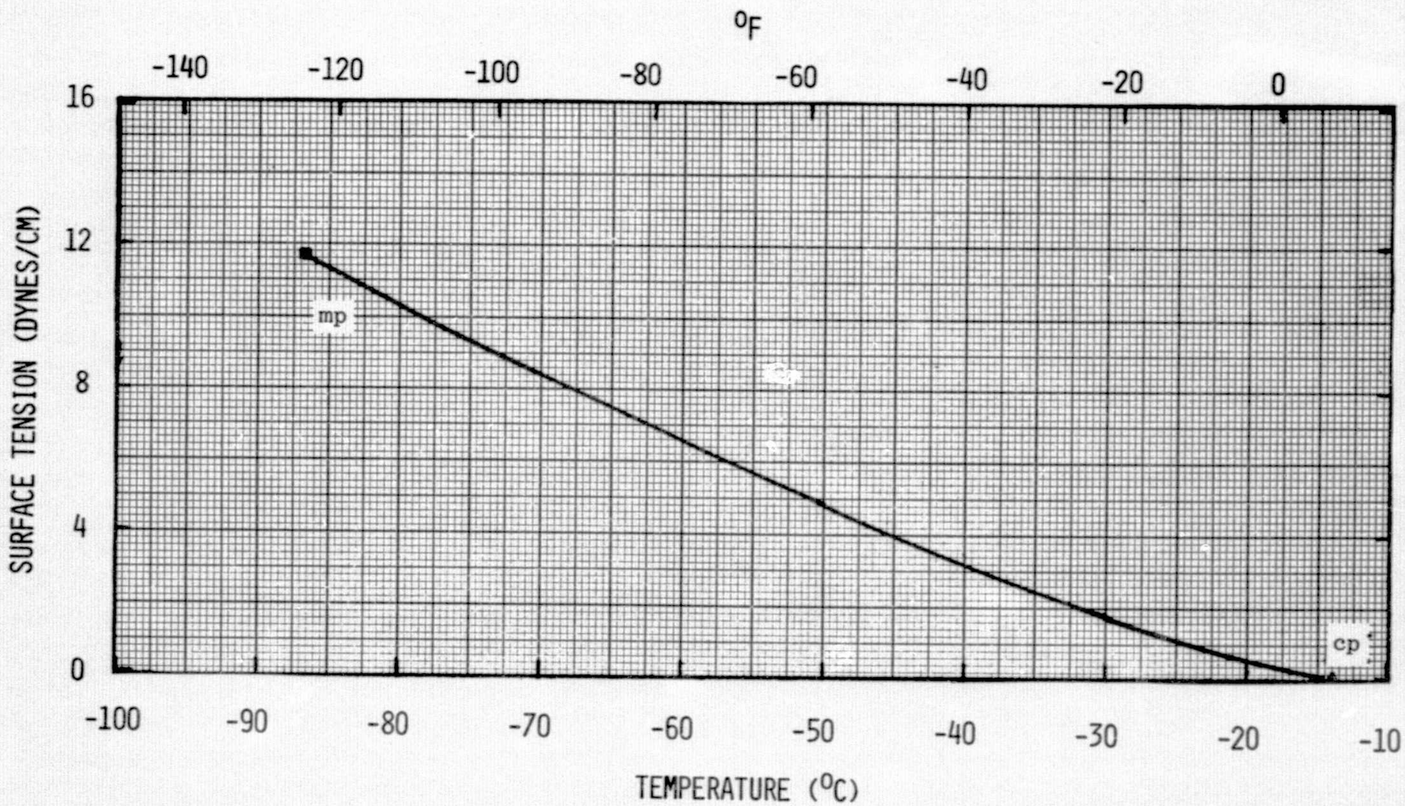

Figure IA-6--Surface Tension vs Temperature for Silicon Tetrafluoride

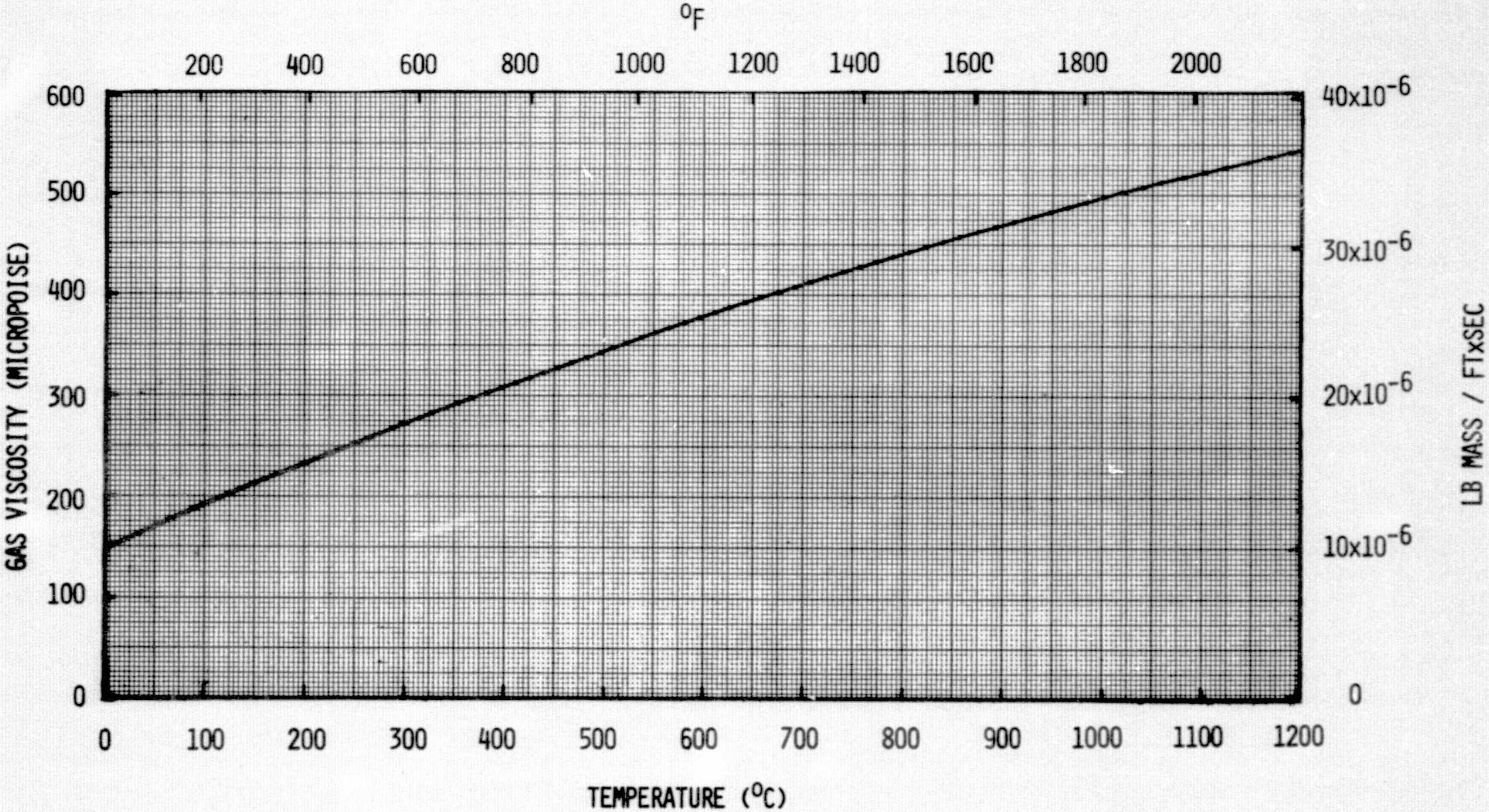

Figure IA-7--Gas Viscosity vs Temperature for Silicon Tetrafluoride

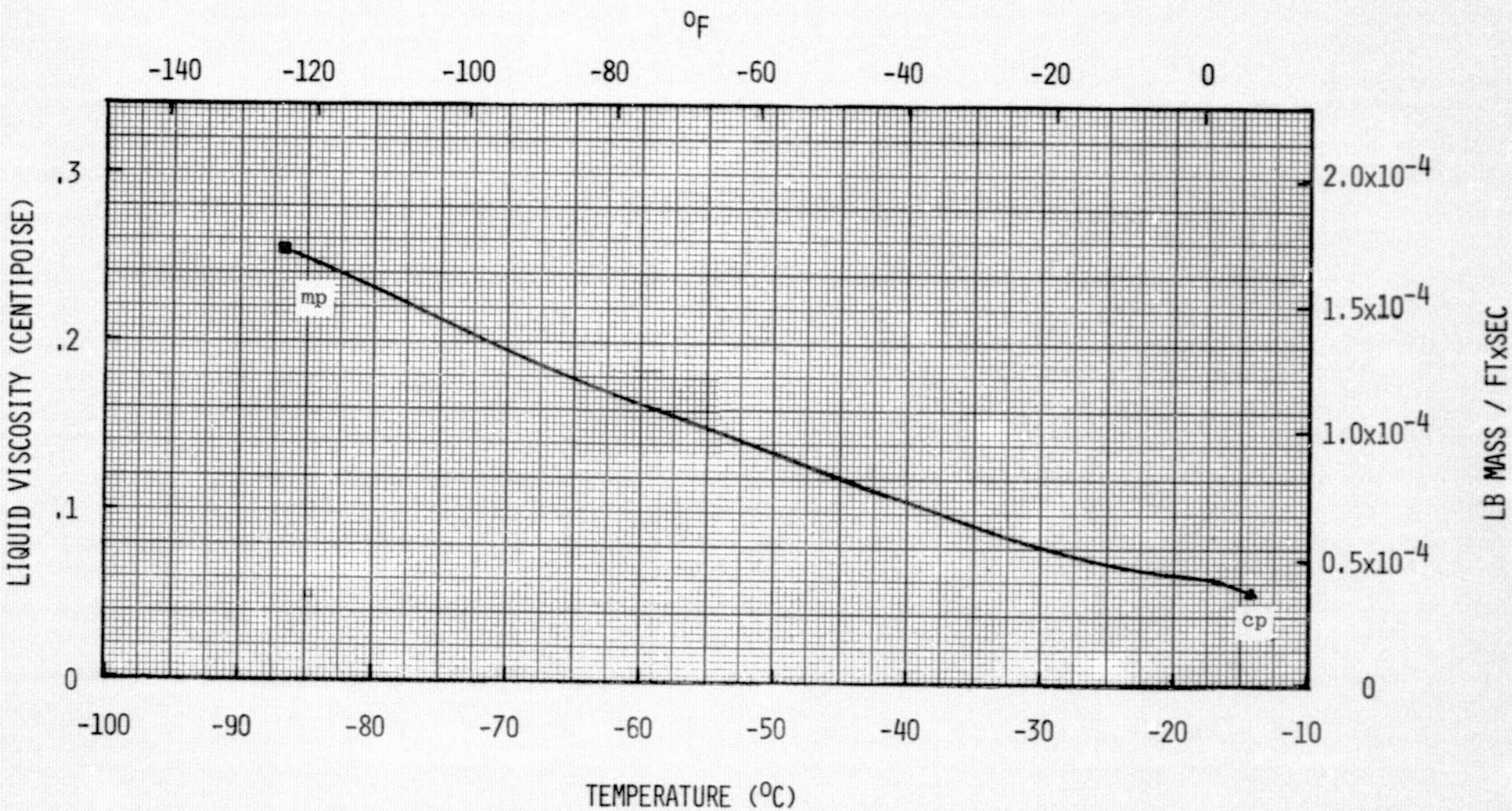

Figure IA-8--Liquid Viscosity vs Temperature for Silicon Tetrafluoride

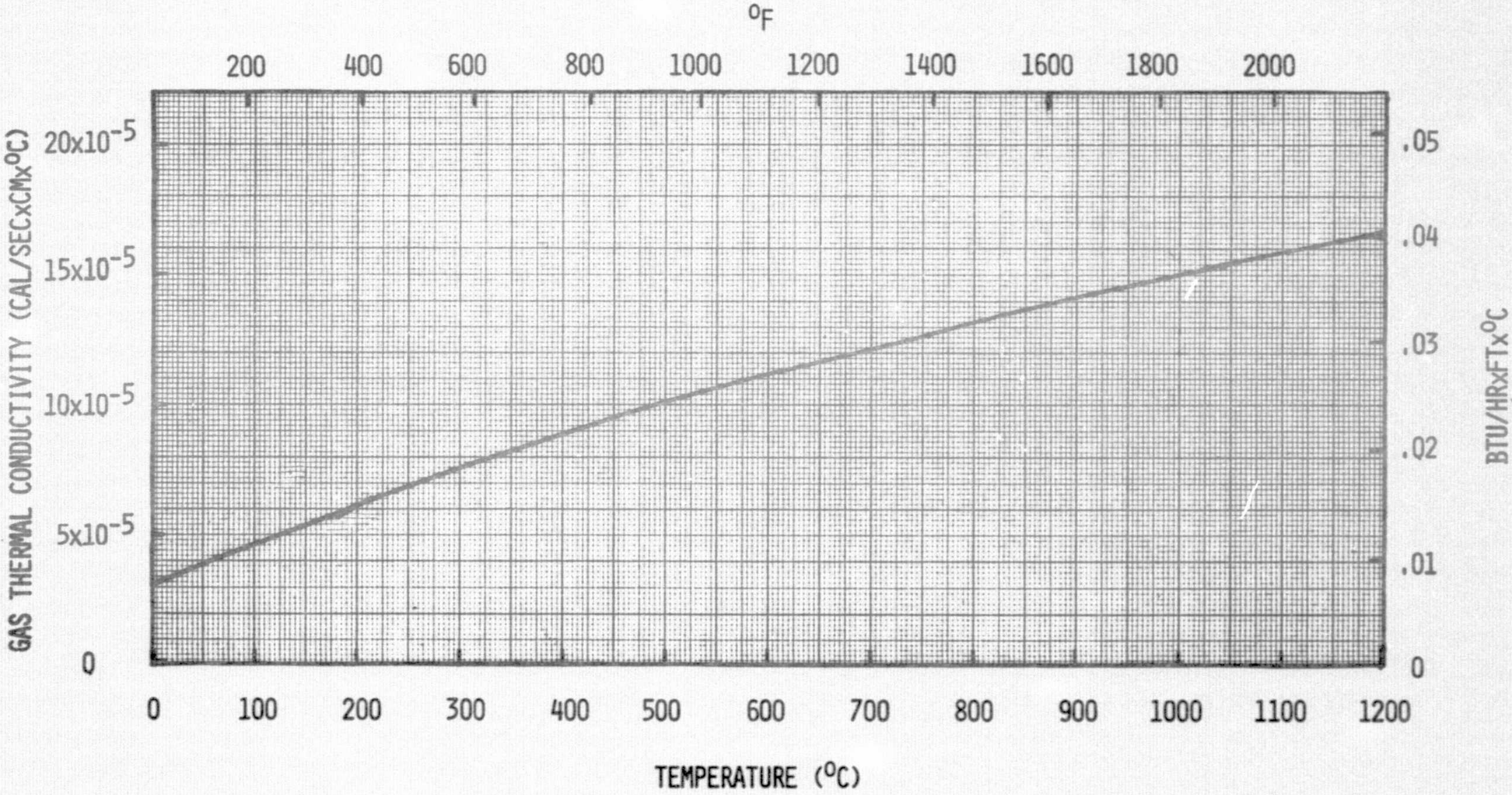

Figure IA-9--Gas Thermal Conductivity vs Temperature for Silicon Tetrafluoride

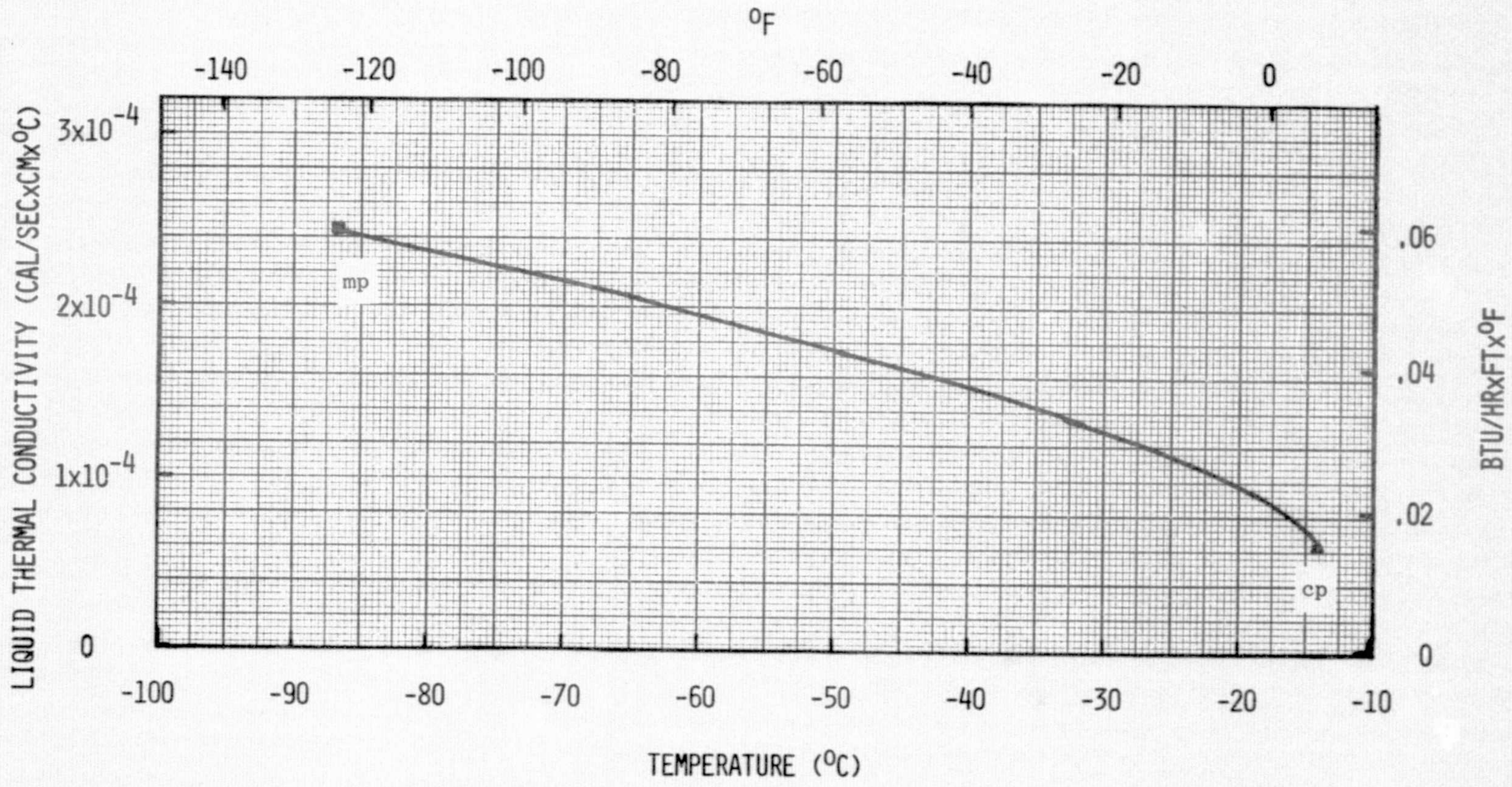

Figure IA-10--Liquid Thermal Conductivity vs Temperature for Silicon Tetrafluoride

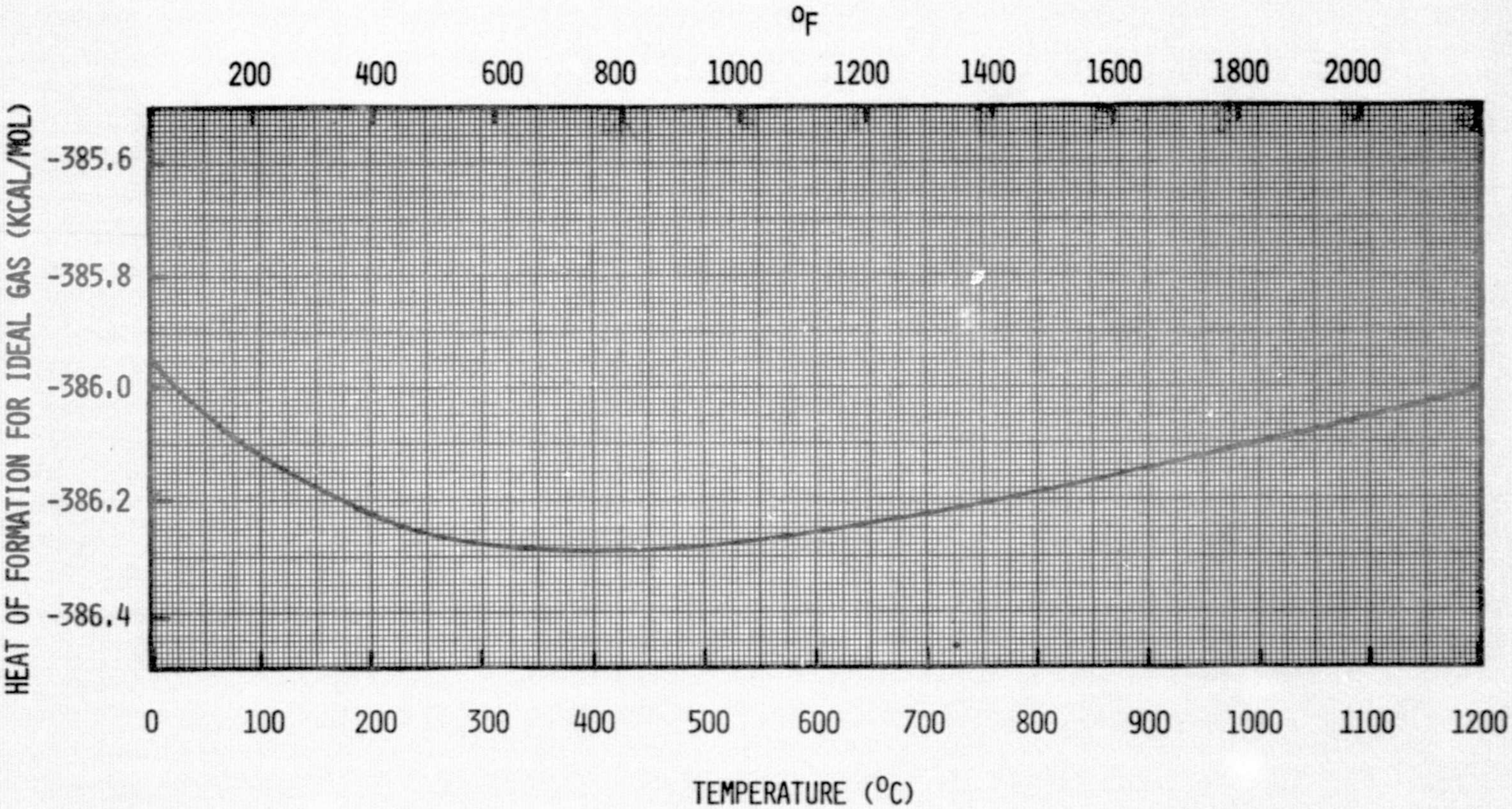

Figure IA-11--Heat of Formation vs Temperature for Silicon Tetrafluoride

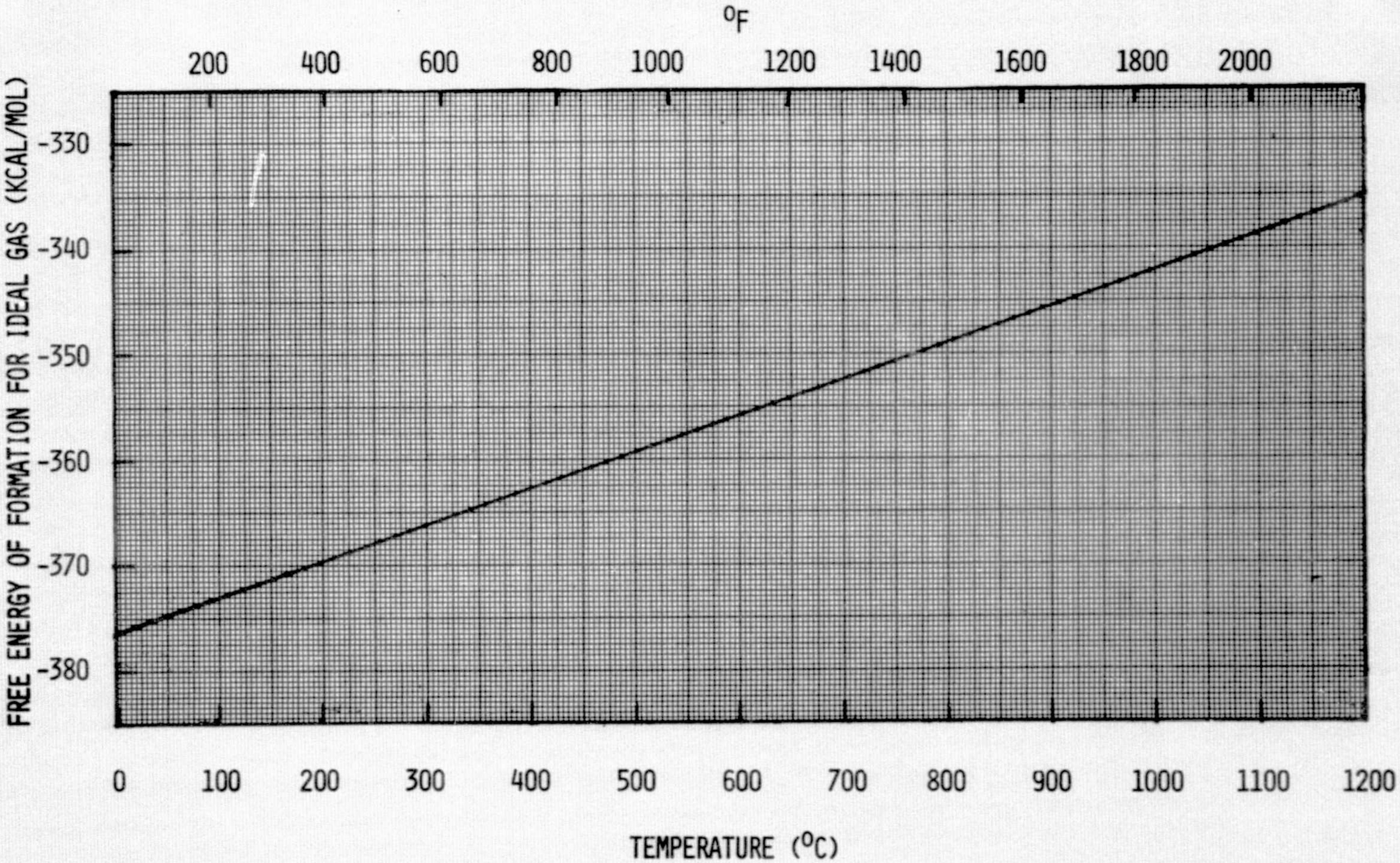

Figure IA-12--Free Energy of Formation vs Temperature for Silicon Tetrafluoride

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B. VISCOSITY INVESTIGATION

During this reporting period the experimental determination of gas phase viscosity of silicon source materials was continued.

The viscosity of trichlorosilane ($SiHCl_3$) has been determined between 40°C and 200°C (table IB-12 and figure IB-17). There have been no previously reported experimental values for gas viscosity of trichlorosilane in the temperature range of the study. Values at 0°C and 31°C were reported by Tel'chuk and Tubyanskaya (ref. 46).

The viscosity of dichlorosilane (SiH_2Cl_2) has been determined between 40°C and 200°C (table IB-13 and figure IB-18). The sample of dichlorosilane used for the measurements was semiconductor grade obtained from Union Carbide Corporation. There have been no previously reported experimental values for the gas phase viscosity of dichlorosilane. One set of calculated values have been reported (ref. 47) in the temperature range of 0°C to 300°C. These calculated values agree with the experimental values determined in this study with deviations of less than ± 2% from 40°C to 200°C.

Table IB-12

Viscosity of Gaseous Trichlorosilane

Temperature $^{\circ}C$	Viscosity micropoise	Nsm^{-1}	$lb_m s^{-1} ft^{-1}$
40	122.0	12.20×10^{-6}	8.20×10^{-6}
60	125.9	12.59×10^{-6}	8.46×10^{-6}
80	134.8	13.48×10^{-6}	9.06×10^{-6}
100	140.8	14.08×10^{-6}	9.46×10^{-6}
150	157.3	15.73×10^{-6}	10.57×10^{-6}
200	177.2	17.72×10^{-6}	11.91×10^{-6}

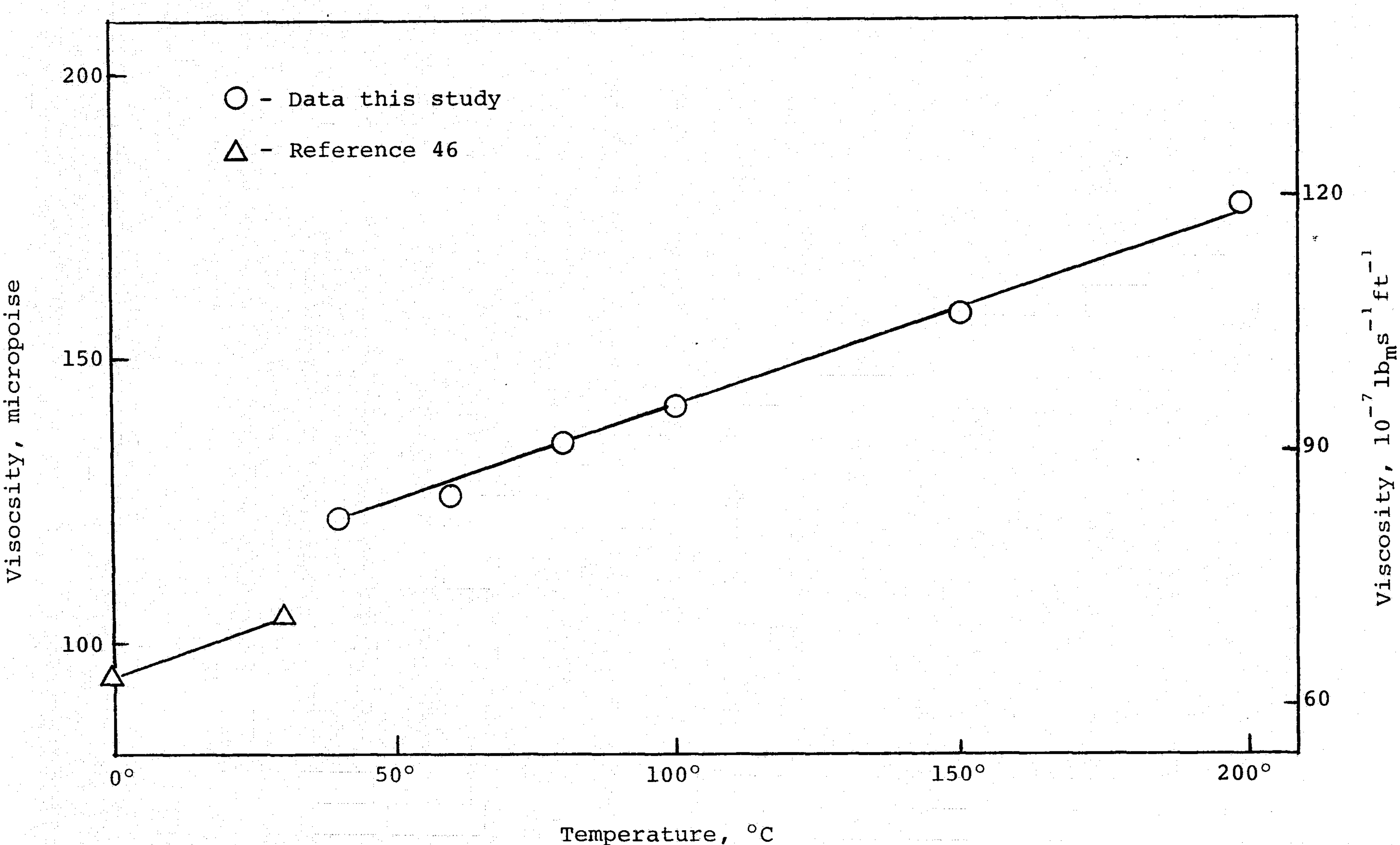


Figure IB-17. Viscosity of Gaseous Trichlorosilane

Table IB-13

Viscosity of Gaseous Trichlorosilane

Temperature °C	Viscosity micropoise	Viscosity Nsm^{-1}	Viscosity $lb_m s^{-1} ft^{-1}$
40	118.7	11.87×10^{-6}	7.98×10^{-6}
60	125.3	12.53×10^{-6}	8.42×10^{-6}
80	134.6	13.46×10^{-6}	9.05×10^{-6}
100	140.2	14.02×10^{-6}	9.42×10^{-6}
150	163.5	16.35×10^{-6}	10.99×10^{-6}
200	181.9	18.19×10^{-6}	12.22×10^{-6}

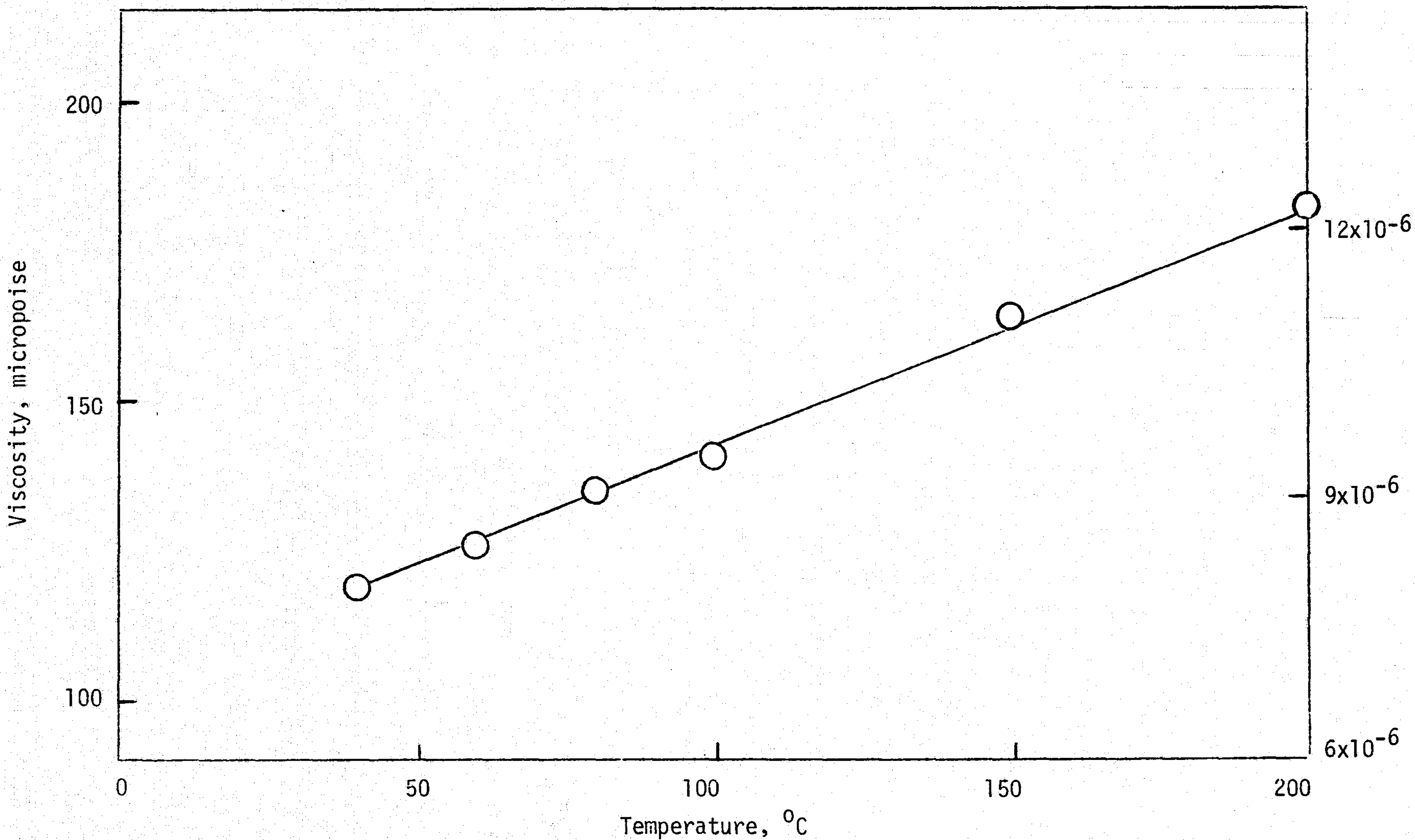


Figure IB-18. The Viscosity of Gaseous Dichlorosilane as a Function of Temperature

II. CHEMICAL ENGINEERING ANALYSIS (TASK 2)

A. BCL PROCESS (BATTELLE COLUMBUS LABORATORIES)

Major efforts were continued during this reporting period on the preliminary process design for the BCL 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	40%	95%
.Reaction Chemistry	40%	95%
.Process Flow Diagram	30%	90%
.Material Balance	20%	90%
.Energy Balance	10%	90%
.Property Data	10%	90%
.Equipment Design	10%	90%
.Production Labor	0%	80%

The detailed status sheet is shown in Table IIA-1.0 to present the items that make up the preliminary process design. The process flowsheet is given in Figure IIA-1.0 for the BCL process.

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

.Base Case Conditions...............Table IIA-1.1
.Reaction Chemistry.................Table IIA-1.2
.Raw Material Requirements..........Table IIA-1.3
.Utility Requirements...............Table IIA-1.4
.Major Process Equipment............Table IIA-1.5

In current activities for the preliminary design, engineering efforts are being devoted to manpower estimates of production labor required to operate the plant. These results from the preliminary process design will be used in economic analysis.

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

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	◐
1. Size	◐
2. Type	◐
3. Materials of Construction	◐
8a. Major Technical Factors	◐
(Potential Problem Areas)	◐
1. Materials Compatibility	◐
2. Process Conditions Limitations	◐
3. Additional	◐
9. Production Labor Requirements	◐
1. Process Technology	◐
2. Production Volume	◐
10. Forward for Economic Analysis	O

O Plan
◐ In Progress
● Complete

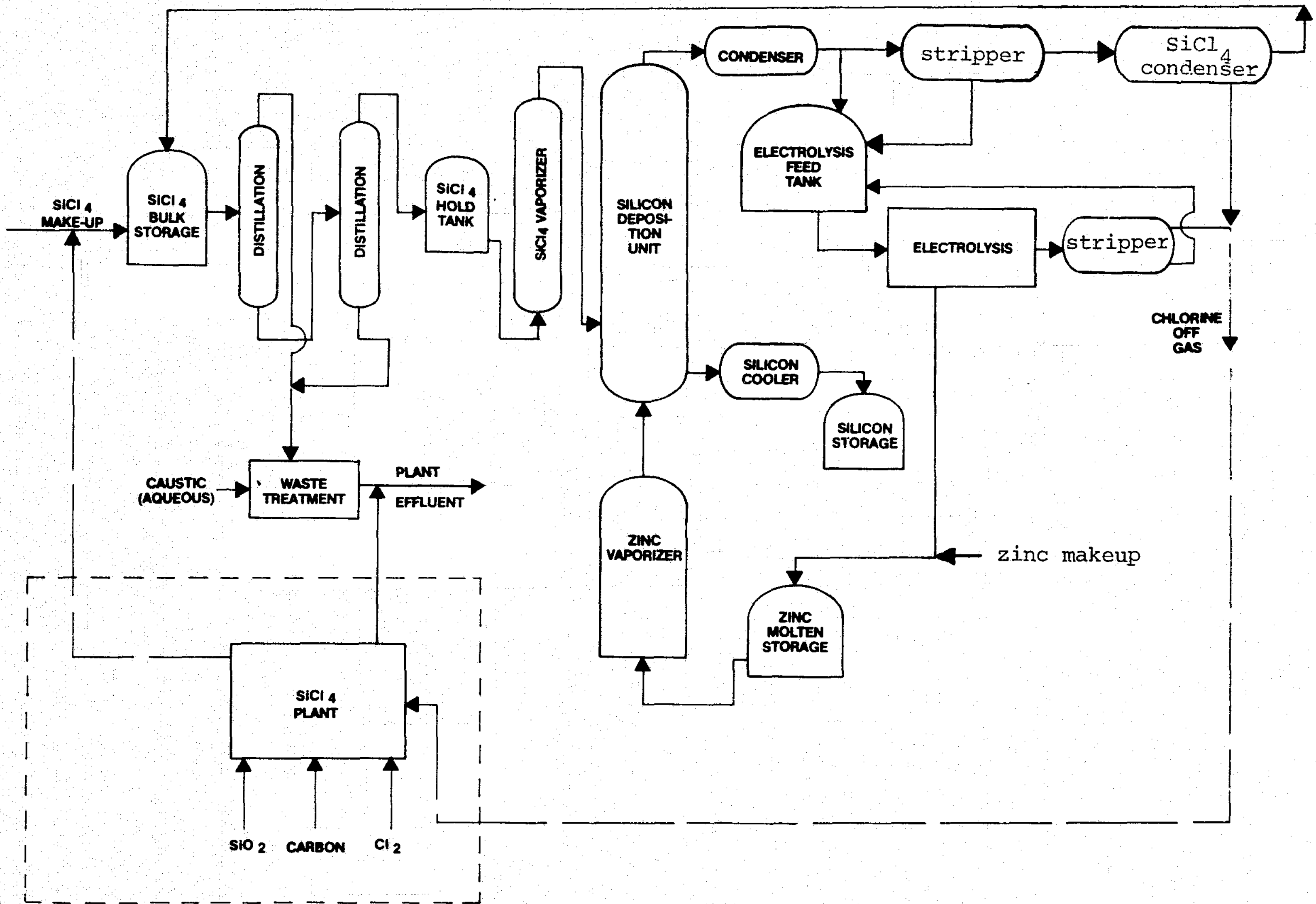


Figure IIA-1.0 Process Flow Sheet for BCL Process (Battelle Columbus Laboratories)

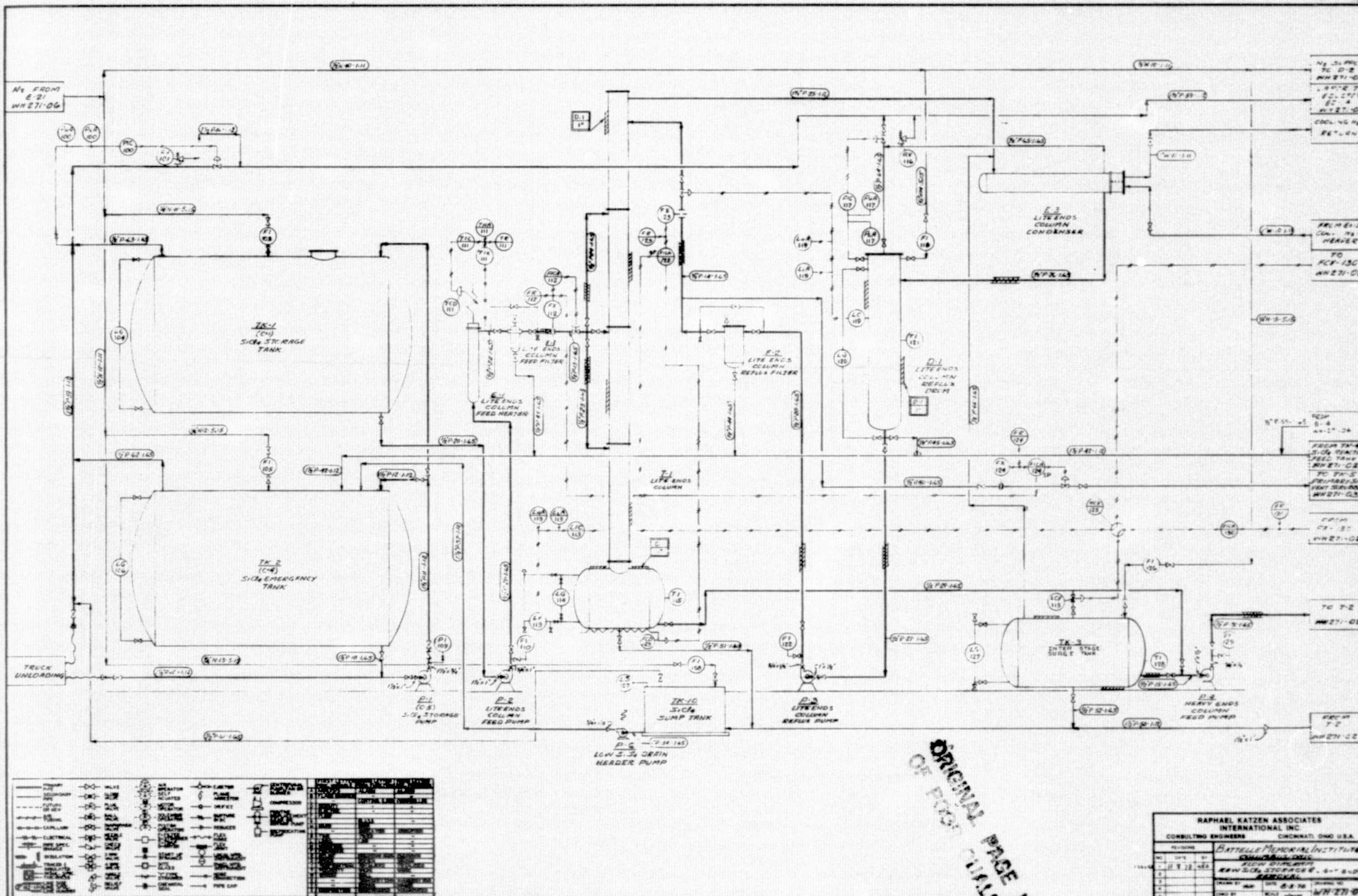


Figure IIA-1.0 (Continued)

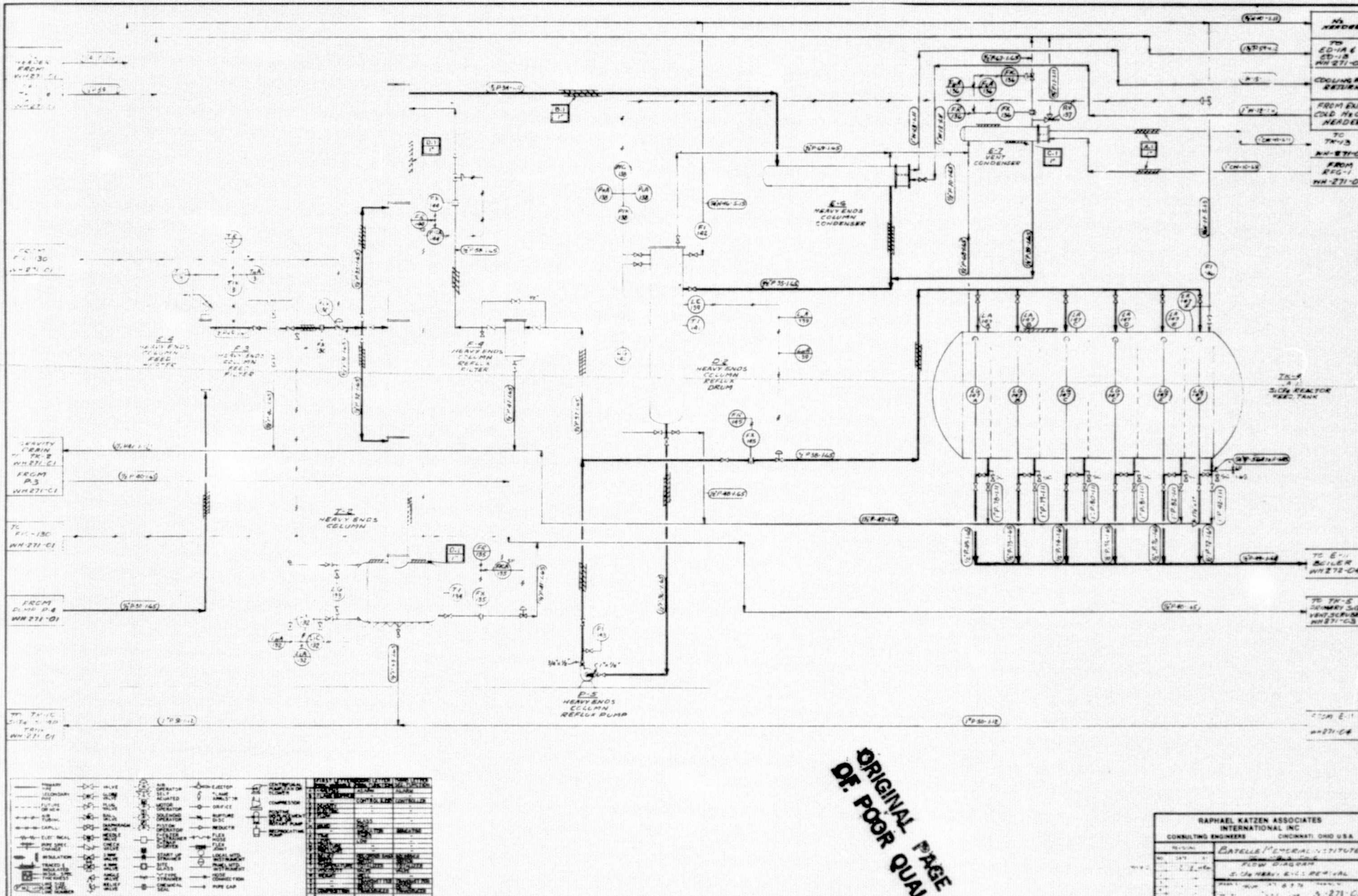


Figure IIA-1.0 (Continued)

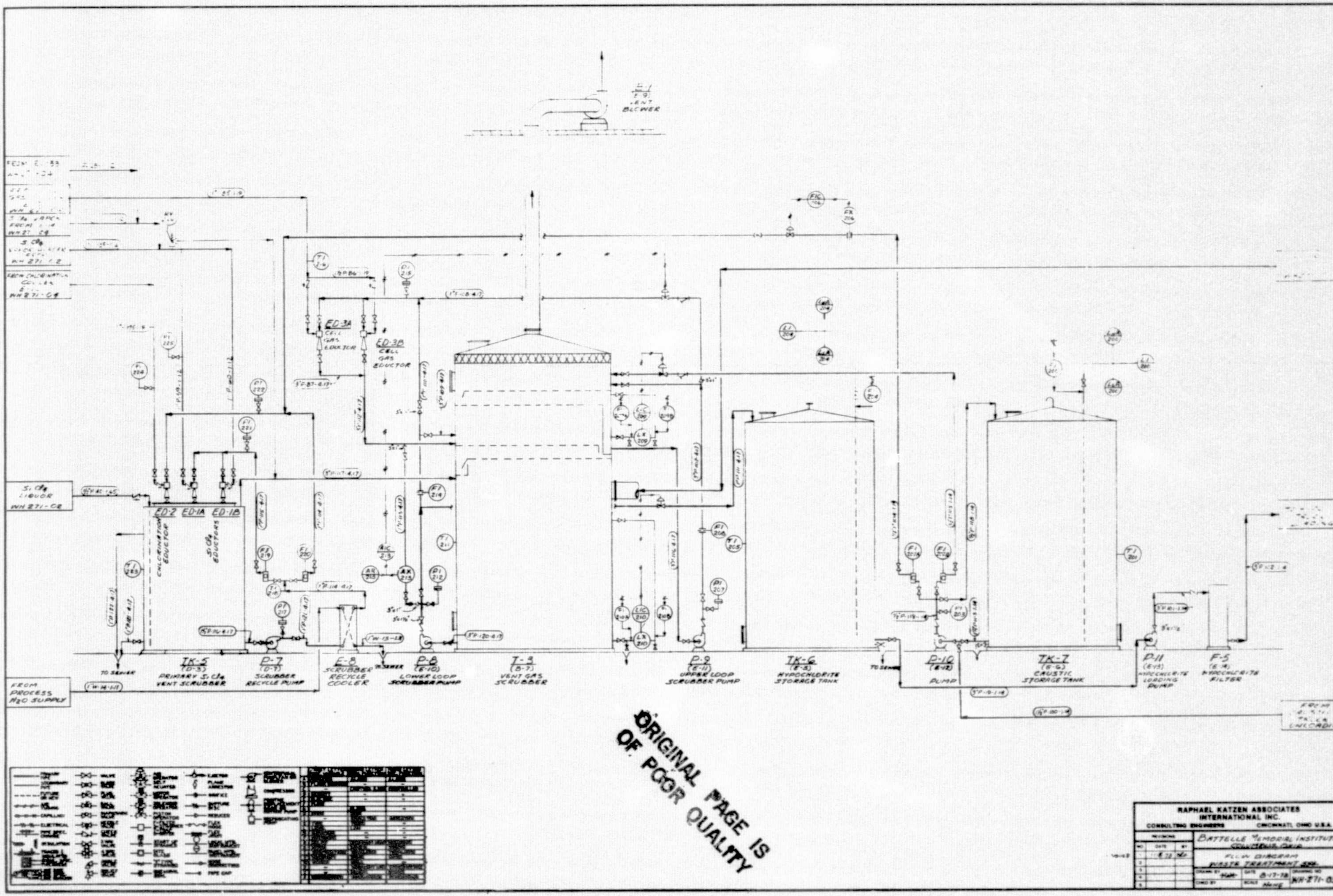


Figure IIA-1.0 (Continued)

Note: For 50 MT/yr Facility only

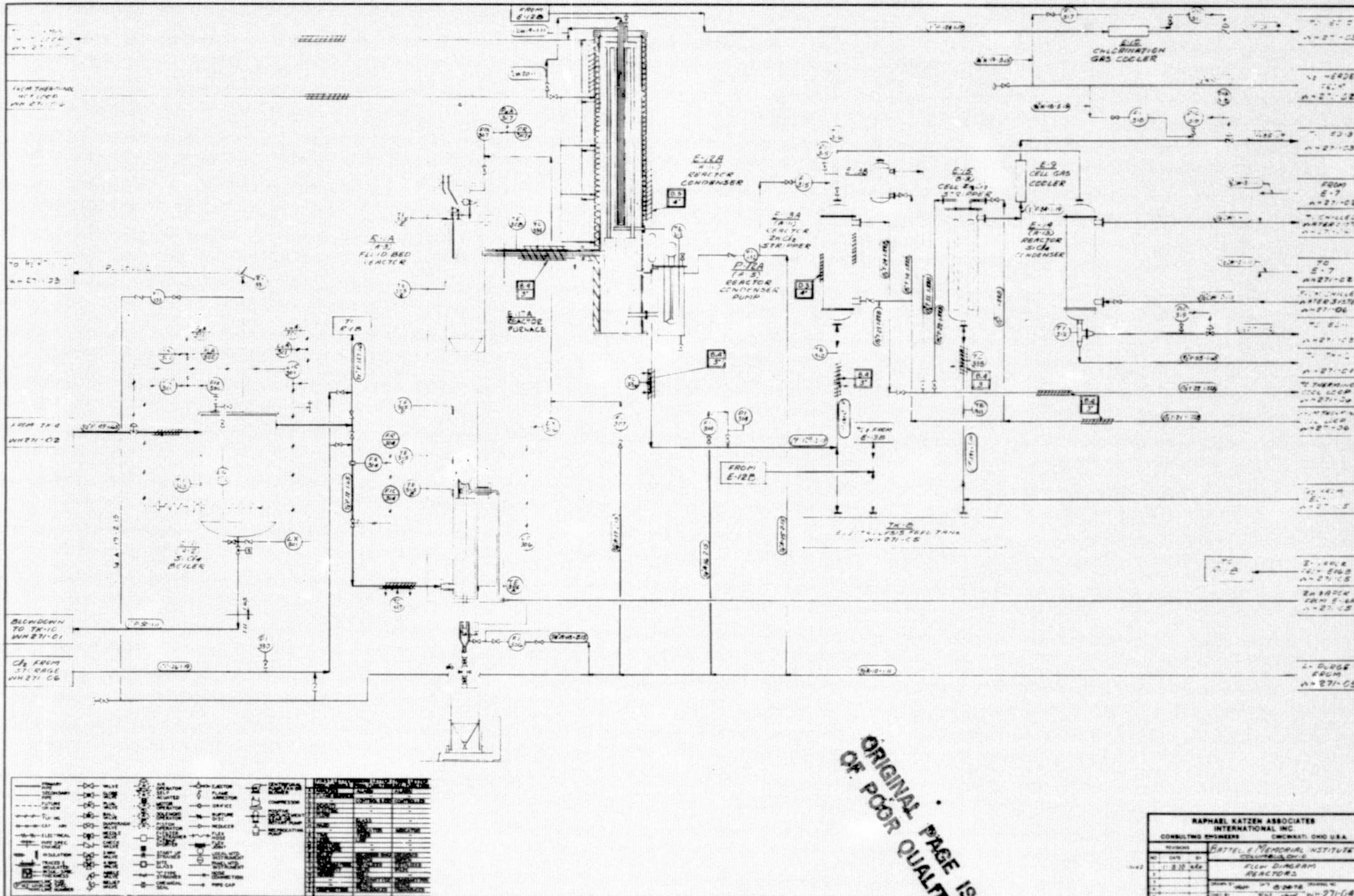


Figure IIA-1.0 (Continued)

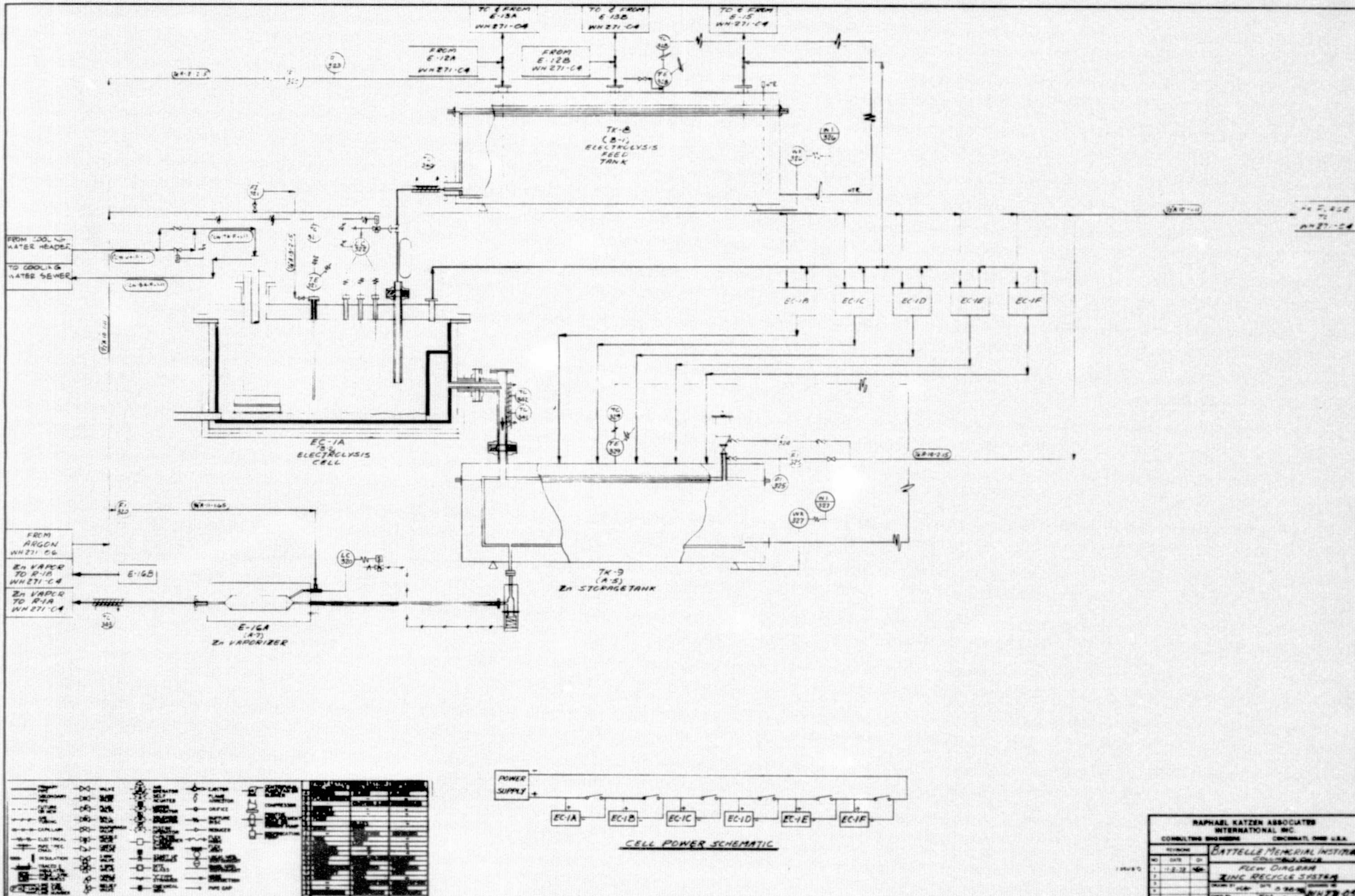


Figure IIA-1.0 (Continued)

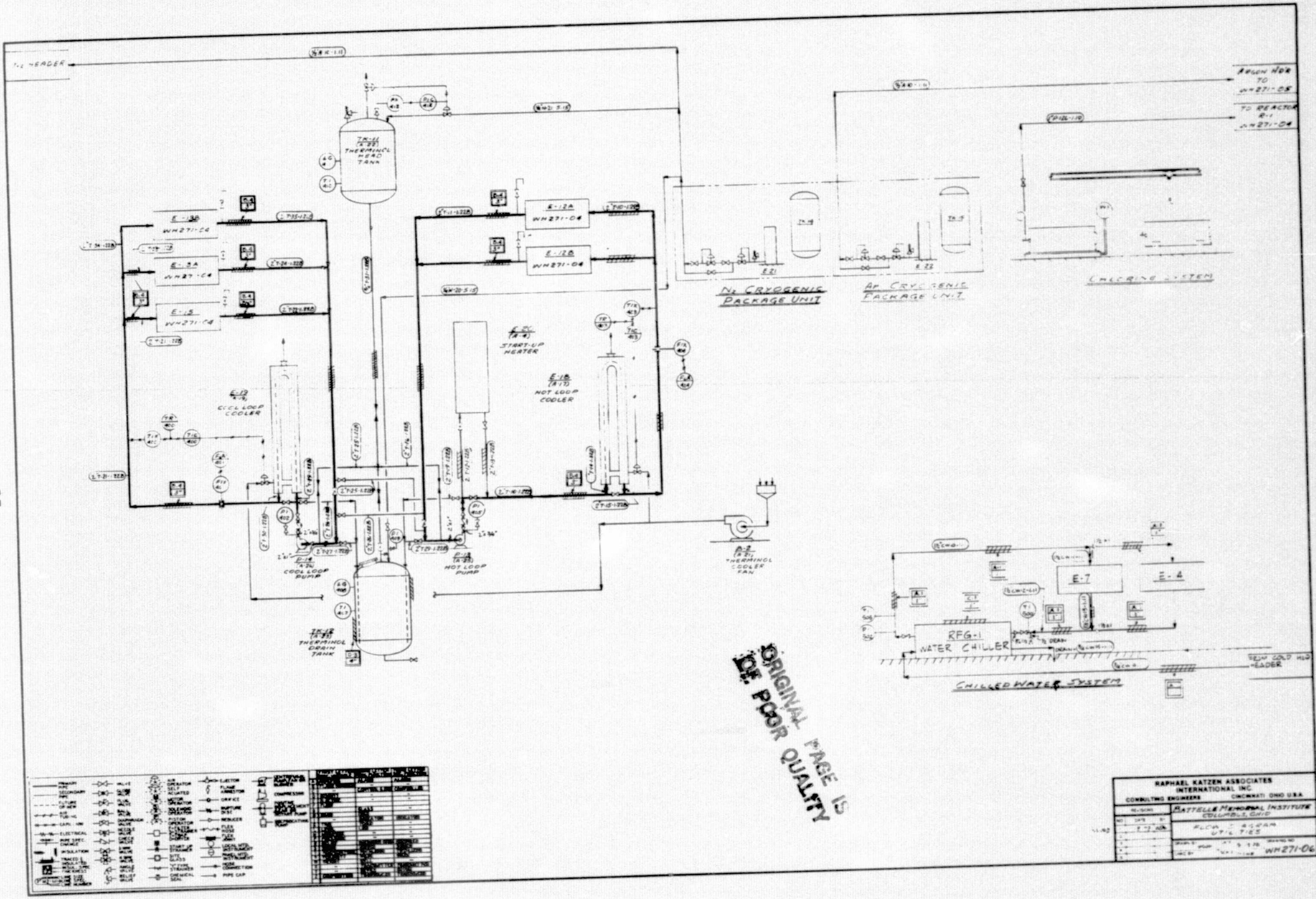
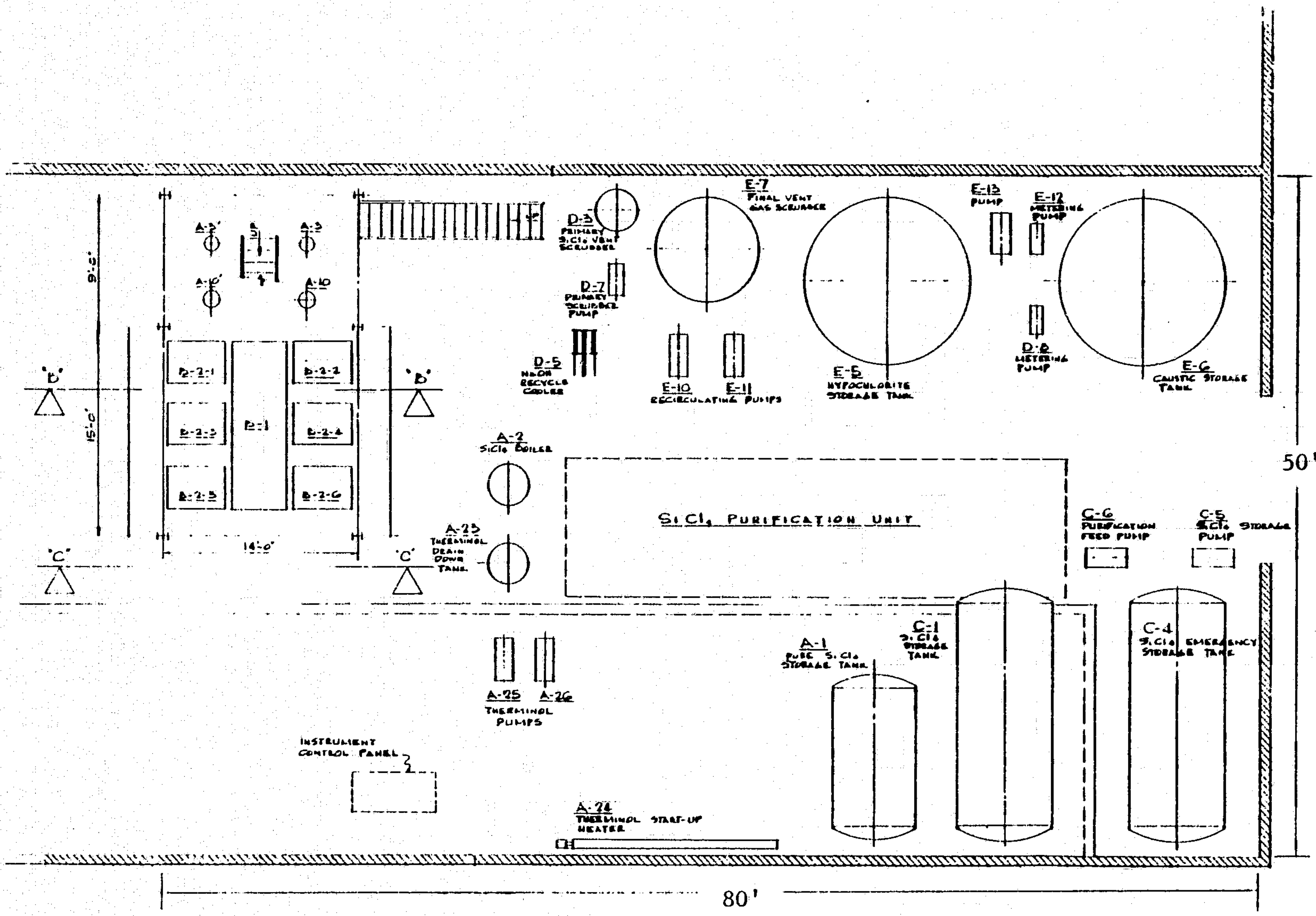


Figure IIA-1.0 (Continued)



PLOT PLAN

Figure IIA-1.0 (Continued)

TABLE IIA-1.1

BASE CASE CONDITIONS FOR BCL PROCESS (BATTELLE COLUMBUS LABORATORIES)

1. Plant Size
   - silicon produced from silicon tetrachloride (TET)
   - 1000 metric tons/yr of silicon
   - solar cell grade silicon
   - solid phase product form (granules)
2. Light End Distillation
   - purification of TET by distillation
   - remove 4% chlorosilanes as the light end
   - 80°C, 10 psig

3. Heavy End Distillation
   - purification of TET by distillation
   - remove 4% impurities as the heavy end
   - 92% over-all yield of TET from both distillations
   - 80°C, 10 psig

4. TET Vaporizer
   - to supply TET vapor for deposition reactor
   - by power input (resistance heater)
   - hold at constant level and constant pressure
   - 164°F

5. Deposition Reactor
   - reduce TET by zinc to produce silicon
   - deposit on pure silicon seed
   - fluid bed
   - 927°C (1700°F, 1 atm)
   - 63% conversion of TET to silicon

6. Reactor Condenser
   - to condense gases from reactor ($ZnCl_2$, unreacted Zn and $SiCl_4$ gases)
   - partial condensation
   - using therminol 66 as the coolant
   - 927°C inlet temperature and 350°C outlet temperature

7. Reactor $ZnCl_2$ Stripper
   - work as partial condenser
   - to condense $ZnCl_2$ gas from $SiCl_4$ gas
   - operating at the temperature right above $ZnCl_2$ melting point (318°C), 350°C
   - using therminol 66 as the coolant

8. Cell $ZnCl_2$ Stripper
   - operates as partial condenser
   - to condense $ZnCl_2$ gas from Cl_2 and $SiCl_4$ gases
   - operating at the temperature right above $ZnCl_2$ melting point (318°C), 350°C
   - using therminol 66 as the heat exchange medium

9. Reactor $SiCl_4$ Condenser
   -condense $SiCl_4$ gas for recycle
   -antifreeze as the coolant
   -350°C inlet temperature, 20°F outlet temperature.

10. Electrolysis
   -electrolytic recovery of Zn from $ZnCl_2$
   -Cl_2 gas is by product
   -95% Zn recovery
   -500°C, approx. 1 atm

11. Zinc Vaporizer
   -to vaporize Zinc
   -by induction heating
   -927°C, approx. 1 atm.

12. Wastes Treatment
   -to scrub and neutralize $SiCl_4$ and chlorosilane gases
   -caustic solution used to neutralize

13. Operating Ratio
   -approximately 80% utilization (on stream time)
   -approximately 7,000 hr/yr production

14. Storage Considerations
   -feed material (two week supply)
   -product (two shifts storage)
   -process (several hours)

TABLE IIA-1.2
REACTION CHEMISTRY FOR BCL PROCESS (BATTELLE COLUMBUS LABORATORIES)

1. Silicon Deposition

$2Zn + SiCl_4 \rightarrow Si\downarrow + 2ZnCl_2$

2. Electrolysis

$ZnCl_2 \xrightarrow{\text{electroly.}} Zn(l) + Cl_2\ (g)$

3. Waste Treatment

$SiCl_4 + 2H_2O \xrightarrow{aq} SiO_2 + 4HCl(aq)$

$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O$

or

$Ca(OH)_2(aq) + 2HCl(aq) \rightarrow CaCl_2(aq) + 2H_2O$

3a. Waste Treatment (50 MT/yr unit)

$Cl_2(g) + 2NaOH(aq) \rightarrow NaOCl(aq) + NaCl(aq) + H_2O$

Table IIA-1.3

Raw Material Requirements for
BCL Process (Battelle Columbus Laboratories)

Raw Material	Requirements lb/KG of Silicon
1. Silicon Tetrachloride, $SiCl_4$	15.33
2. Zinc, Zn	0.54
3. Caustic (50%), NaOH(aq)	3.75
or	
Lime (99%), $Ca(OH)_2$	1.75
4. Argon	3.1 SCF*
5. Nitrogen	7.6 SCF*
6. Chlorine, Cl_2 (by-product)	11.12

*Estimate from BCL

TABLE IIA-1.4 UTILITY REQUIREMENTS FOR BCL PROCESS (BATTELLE COLUMBUS LABORATORIES)

Utility/Function		Requirements/Kg of Silicon Product
1. Electricity		29.40 Kw-hr
1. Low Voltage D.C. for Electrolysis	(20.51)	
2. Zinc Vaporizer Induction Heated	(4.37)	
3. Preheat Section of Deposition Unit Induction Heated	(1.12)	
4. Electrolysis Feed Tank Heater	(0.24)	
5. Molten Zinc Storage Heater	(0.10)	
6. $SiCl_4$ Vaporizer	(0.53)	
7. Pumps, Blowers	(2.53)	
2. Steam (50 PSIA)		9.67 pounds
1. #1 Purification Column Calandria	(4.59)	
2. #2 Purification Column Calandria	(4.30)	
3. Caustic Storage Heating	(0.29)	
4. #1 Purification Column Preheater	(0.49)	
3. Cooling Water		34.49 Gallons
1. #1 Purification Column Condenser	(16.94)	
2. #2 Purification Column Condenser	(15.88)	
3. Purified Tet Cooler	(1.67)	
4. Process Water		10.48 Gallons
1. Diluent for Waste Treatment	(10.48)	
5. Refrigeration		2.38 MBtu
1. Reactor $SiCl_4$ Condenser (H-11)	(1.28)	
2. $SiCl_4$ Vent Condenser (H-07)	(1.10)	

TABLE IIA-1.5

LIST OF MAJOR PROCESS EQUIPMENT
FOR BCL (BATTELLE COLUMBUS LABORATORIES)

Equipment	Function	Size/Type	Material of Construction	Capacity Ratio to 1000 MT/yr
PROCESS TOWER AND INTERNALS				
1. D-01 Light End Distillation Column	To purify $SiCl_4$	8" dia. x 21', packed 13.5'	Column, CS/packing, SS	20
2. D-02 Heavy End Distillation Column	To purify $SiCl_4$	8" dia. x 21', packed 13.5'	Column, CS/packing, SS	20
3. A-01 Primary $SiCl_4$ Vent Scrubber	To scrub $SiCl_4$ vent gas	3' dia. x 4'4" T/T, 225 gal/flat bottom	FRP	1
4. A-02 Final $SiCl_4$ Vent Scrubber	To scrub $SiCl_4$ vent gas	7'6" dia. x 17'4" T/T/ 4 pp trays, Teflon dimister	FRP	1
HEAT EXCHANGER				
5. H-01 L.E. Column Feed Heater	To preheat feed to D-01	2' dia. x 5', 15,013 Btu/hr / external heater	CS	20
6. H-02 L.E. Column Reboiler	Reboiler of D-01	2' dia. x 3', 51,522 Btu/hr / external heater	CS	20
7. H-03 L.E. Column Condenser	Total condenser of D-01	47,430 Btu/hr/shell-tube H.E.	CS	20
8. H-04 H.E. Column Feed Heater	To preheat feed to D-02	2' dia. x 5', 14,331 Btu/hr/external heater	CS	20

TABLE IIA-1.5 (Continued)

9.	H-05 H.E. Column Reboiler	Reboiler of D-02	2' dia. x 3', 56,641 Btu/hr/external heater	CS	20
10.	H-06 H.E. Column Condenser	Total condenser of D-02	52,292 Btu/hr/shell-tube H.E.	CS	20
11.	H-07 $SiCl_4$ Vent Condenser	Condense $SiCl_4$ from vent gas	38 ft^2, 18,000 Btu/hr/shell-tube H.E.	CS	20
12.	H-08 $SiCl_4$ Vaporizer	To provide $SiCl_4$ vapor to reactor	2.75' dia. x 3' T/T, 13,648 Btu/hr/resistance heater	CS	20
13.	H-09 Reactor Condenser	To condense by products from reactor	14' dia. x 6.4', 126,237.2 Btu/hr	Graphite W/SS shell	20
14.	H-10 Reactor $ZnCl_2$ Stripper	To condense $ZnCl_2$ gas	12 ft^2, 2,652 Btu/hr/shell-tube H.E., finned U-tube	316 SS	20
15.	H-11 $SiCl_4$ Condenser	To condense $SiCl_4$ gas for recycle	6,401 Btu/hr (x 4.62** = 29.573 Btu/hr)	316 SS	20
16.	H-12 Cell $ZnCl_2$ Stripper	To condense $ZnCl_2$ vapor	9,841.4 Btu/hr/shell-tube, H.E. (x 0.32**)	Inconel 600	20
17.	H-13 Therminol Cooler (cold circuit)	To cool Therminol 66	68 ft^2, 11,000 Btu/hr/shell-tube H.E., 500 psia	CS	20
18.	H-14 Therminol Cooler (hot circuit)	To cool Therminol 66	262 ft^2, 120,000 Btu/hr/shell-tube H.E., 500 psia	CS	20

* The operation conditions were changed from 171°F ~ 32°F to 662°F ~ 20°F.

** The operation conditions have been changed from ΔT = 855°F to 270°F.

TABLE IIA-1.5 (Continued)

19.	H-15 Start-up Heater	Therminol start-up heater	98,950 Btu/hr/U-tube 15', resistance heater	CS	20
20.	H-16 Silicon Product Cooler	To cool the Si product from reactor	5,735 Btu/hr	SiC	20

PROCESS AND STORAGE VESSELS

21.	T-01 $SiCl_4$ Storage Tank	Storage/feed to purification	7' dia. x 16' T/T/ 4,600 gal	CS	20
22.	T-02 $SiCl_4$ Emergency Storage Tank	Storage/feed to purification	7' dia. x 16' T/T/ 4,600 gal	CS	20
23.	T-03 L.E. Column Reflux Drum	To hold distillate for reflux	12" dia. x 4'/23 gal	CS	20
24.	T-04 Surge Tank	Surge Tank for D-01 bottom	3' dia. x 4'/200 gal	CS	20
25.	T-05 Sump Tank	Sump for purification unit	3' dia. x 4'/200 gal	CS	20
26.	T-06 H.E. Column Reflux Drum	To hold distillate for reflux	12" dia. x 4'/23 gal	CS	20
27.	T-07 Pure $SiCl_4$ Storage Tank	Storage/feed to $SiCl_4$ Vaporizer	6' dia. x 10' T/T/ 1900 gal	CS	20
28.	T-08 Electrolysis Feed Tank	Storage/feed $ZnCl_2$ to electrolysis cell	50" x 158" x 38"H/ 7" graphite TH	Graphite/304 SS	20
29.	T-09 Molten Zinc Storage Tank	Storage/feed to Zinc vaporizer	W/heater 68,242 Btu/hr	Graphite/304 SS	20
30.	T-10 Therminol Head Tank	Storage Therminol	1.5' dia. x 3.75' T/T/ 49.6 gal	CS	20

TABLE IIA-1.5 (Continued)

31.	T-11 Thermino1 Drain Down Tank	To store drained Therminol	2.75' dia. x 3' T/T/ 133 gal	CS	20
32.	T-12 Chlorine Supply Tank	To supply chlorine gas	1 1/2' dia. x 3'/ 37.62 gal	CS	20
33.	T-13 Caustic Storage Tank	Storage Caustic	12' dia. x 14'6" T/T/ 12,000 gal	FRP	1

PUMPS WITH DRIVERS

34.	P-01 Purification Feed Pump	To feed $SiCl_4$ to storage tank	30 gpm, 31' head/ centrifugal, 1 1/2 hp	CS	20
35.	P-02 L.E. Column Feed Pump	To supply $SiCl_4$ to preheater	28.9 gph, Δp = 72 psia/ 0.5 hp.	CS	20
36.	P-03 L.E. Column Relux Pump	D-01 Reflux	51.7 gph, Δp = 23 psia/ 0.5 hp.	CS	20
37.	P-04 Surge Tank Pump	To supply $SiCl_4$ to H.E. Column	29.4 gph, Δp = 53 psia/ 0.5 hp.	CS	20
38.	P-05 Sump Pump	To pump $Sicl_4$ to emergency tank	30 gpm, 31' head/ centrifugal, 1 1/2 hp.	CS	20
39.	P-06 L.E. Column Bottom Pump	To pump $SiCl_4$ to surge tank	29.4 gph, Δp = 53 psia/ 0.5 hp.	CS	20
40.	P-07 H.E. Column Relux Pump	D-02 Reflux	57.1 gph, Δp = 25 psia/ 0.5 hp.	CS	20
41.	P-08 H.E. Column Bottom Pump	To pump bottom solution to waste treatment	1.3 gph, Δp = 25 psia/ 0.5 hp.	CS	20
42.	P-09 $SiCl_4$ Vaporizer Feed Pump	To feed $SiCl_4$ to Vaporizer	15 gph, 31' head/ 1/2 hp	CS	20

TABLE IIA-1.5 (Continued)

43.	P-10 Reactor Condenser Circulating Pump	To circulate condensates	2.4 gpm, 30' head/1/2 hp	Graphite	20
44.	P-11 Cold Circuit Pump	Cold Therminol circulation	20 gpm, 85' head/centrifugal, 2 hp.	CS	20
45.	P-12 Hot Circuit Pump	Hot Therminol circulation	62 gpm, 85' head/centrifugal, 4 hp.	CS	20
46.	P-13 Primary Scrubber Recirculation Pump	Recirculation for Scrubber A-01	20 gpm, 125' head/ centrigugal, 2.5 hp.	Duriron	1
47.	P-14 Primary Scrubber Lower-loop Recirculating Pump	Circulate solution for Lower-loop of Scrubber A-02	100 gpm, 103' head/ centrifugal, 7 1/2 hp.	Duriron	1
48.	P-15 Primary Scrubber Upper-loop Recirculating Pump	Circulate solution for upper-loop of Scrubber A-02	100 gpm, 13' head/ centrifugal, 2 hp.	Duriron	1
49.	P-16 Make up NaOH Metering Pump	NaOH make up	0.9 gpm, 25' head/ centrifugal, 1/2 hp.	CS	1

FILTERS

50.	F-01 L.E. Column Feed Filter	Remove solids	29 gph, Δp = 5 psia/ 140 micron	CS	20
51.	F-02 L.E. Column Reflux Filter	Remove solids	30 gph, Δp = 5 psia/ 140 micron	CS	20
52.	F-03 H. E. Column Feed Filter	Remove solids	52 gph, Δp = 5 psia/ 140 micron	CS	20
53.	F-04 H.E. Column Relfux Filter	Remove solids	31 gph, Δp = 5 psia/ 140 micron	CS	20

54.	F-05 Therminol Cooler Blower Filter	To filter the solids from air			20

SPECIALIZED EQUIPMENTS

55.	R-01 Fluidized Bed Reactor	To reduce $SiCl_4$ to Si by Zn	1830.2 Btu/hr, 6.5" dia.	Graphite Lined / SS	20
56.	FN-01 Furnace	To preheat $SiCl_4$ gas	272,966 Btu/hr		20
57.	B-01 Seed Addition Hopper	To feed Si seed to the reactor		310 SS	20
58.	B-02 Si Product Hopper (four)	To hold Si product	6 gal	310 SS	20
59.	B-03 Zinc Hopper	To hold make up Zinc	40 gal	CS	20
60.	C-01 Therminol Cooler Blower	Therminol system air cooler blower	500 acfm fan/electric, 1 1/2 hp., 12-1/2" wheel	CS	20
61.	C-02 Scrubber Vent Blower	Suck $SiCl_4$ gas for A-01 & A-02	10,000 acfm/electric, 50 hp. 31-1/2" wheel	FRP	1
62.	E-01 Eductor	$SiCl_4$ scrubbing (Scrubber D-05)	20 gpm, Δp = 47.4 psia/ Hydraulic ejector, 1-1/2" NPT	P.V.C.	1
62.	EC-01 Electrolysis Cell	To recover Zn from $ZnCl_2$	5,000 ~6,000 amp cells	Graphite/SS	20
64.	PW-01 Power Supply	To supply power to electrolysis cell	545,933 Btu/hr.		20
65.	VP-01 Zinc Vaporizer	To provide zinc vapor to reactor	104,128.8 Btu/hr 13.5" dia. x 32"	Quartz	20

NOTE:

1. For the 1000 MT/yr plant, items 3, 4, 33, 46, 47, 48, 49, 61, and 62 are used for waste treatment of distillation wastes (light, heavy) and vent gases.

2. In the 50 MT/yr facility, these items are used for hypochlorite maufacture which is not present in the 1000 MT/yr plant.

B. OTHER PROCESSES

Other processes under consideration for solar cell grade silicon production are being monitored and are shown in the following:

1. Westinghouse Process ($Na/SiCl_4$)
2. SRI Process (Na/SiF_4, other)
3. Dow Process (C/SiO_2, other)
4. Aerochem (Na/SiF_4, $Na/SiCl_4$, Na/Graphite, etc.)

III. 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 silicon tetrafluoride: critical constants, vapor pressure, heat of vaporization, heat capacity, density, surface tension, viscosity, thermal conductivity, heat of formation and Gibb's free energy of formation.

Experimental determination of gas viscosity values of silicon source materials was continued. Data are reported for viscosity of trichlorosilane and dichlorosilane between 40°C and 200°C.

2. Task 2

Chemical engineering analysis of the BCL process was continued with primary efforts being concentrated to the preliminary process design. Status and progress are reported for base case conditions; process flow diagram; reaction chemistry; material and energy balances; and equipment design. Current engineering efforts are nearing completion for manpower estimates of production labor requirements for the plant to produce solar cell grade silicon.

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.

2. Task 2

Continue preliminary process design of the BCL process.

3. Task 3

Perform cost analysis of processes as results issue from chemical engineering alnalysis. Initiate cost analysis of the BCL process.

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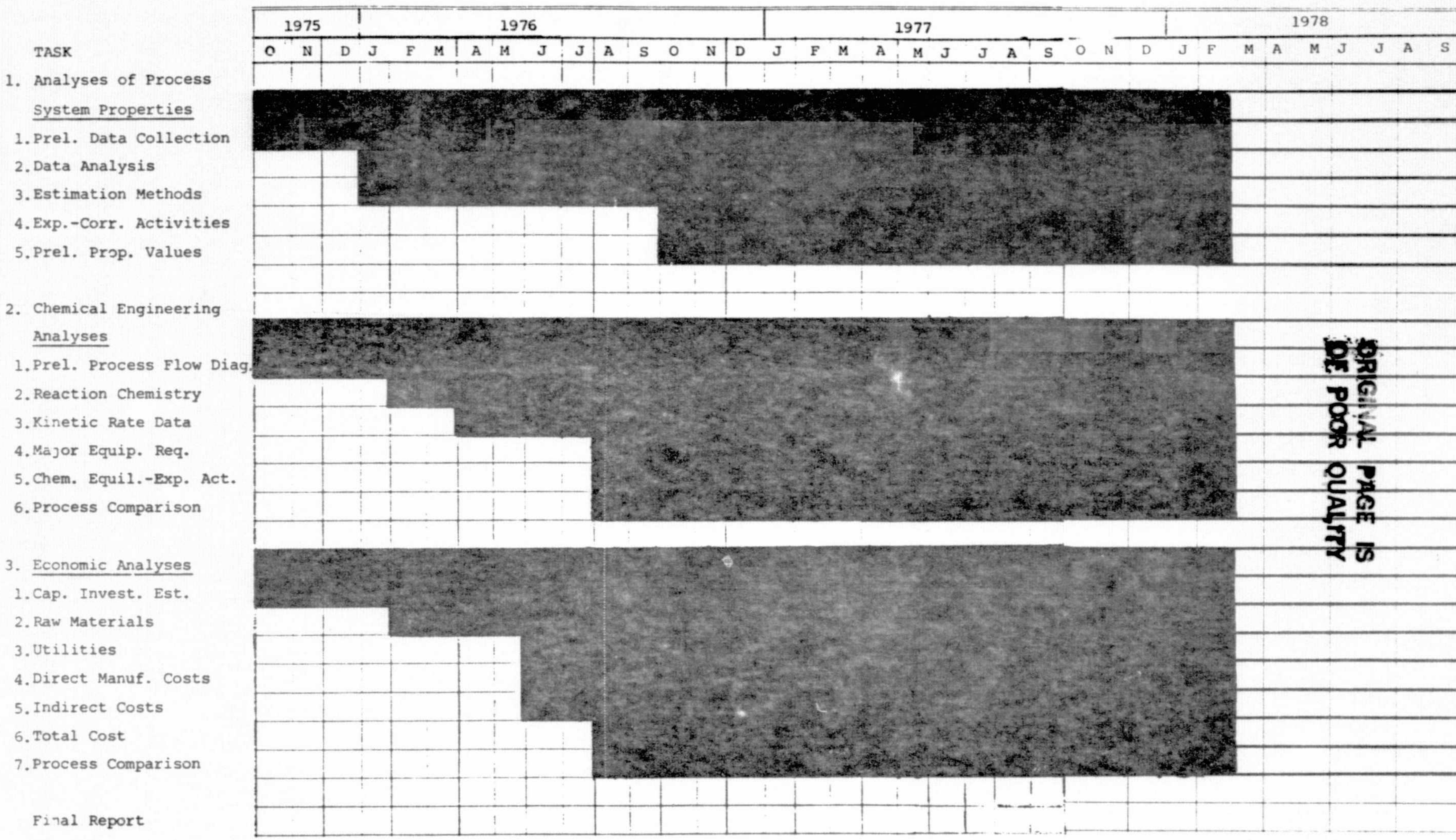
MILESTONE CHART



PROCESS FEASIBILITY STUDY IN
SUPPORT OF SILICON MATERIAL TASK I

JPL Contract No. 954343