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LOW COST SOLAR ARRAY PROJECT

SILICON MATERIALS TASK

DEVELOPMENT OF A PROCESS FOR HIGH CAPACITY
ARC HEATER PRODUCTION OF SILICON FOR SOLAR ARRAYS

CONTRACT No. 954589

Quarterly Technical Report: October, 1978-February, 1979

Maurice G. Fey, Program Manager

Westinghouse Electric Corporation
Power Circuit Breaker Division
Trafford, Pennsylvania 15085



The JPL Low-Cost Solar Array Project is sponsored by the U. S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE.



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1. INTRODUCTION

1.1 Program Description

A program has been established at Westinghouse to develop a high temperature silicon production process using existing electric arc heater technology. Silicon tetrachloride and a reductant (sodium) will be injected into an arc heated mixture of hydrogen and argon. Under these high temperature conditions, a very rapid reaction is expected to occur and proceed essentially to completion, yielding silicon and gaseous sodium chloride. Techniques for high temperature separation and collection of the molten silicon will be developed using standard engineering approaches, and the salt vapor will later be electrolytically separated into its elemental constituents for recycle. Preliminary technical evaluations and economic projections indicate not only that this process appears to be feasible, but that it also has the advantages of rapid, high capacity production of good quality molten silicon at a nominal cost.¹⁻⁴

As currently envisioned, the Westinghouse program consists of a four-phase effort directed to the development and implementation of this technology. The initial phase of the program, Phase I, was an eleven-month study funded by JPL which was completed in September, 1977. While the overall program objective is to produce 1000 metric tons of high quality silicon per year on a continuous basis, Phase I was defined as a comprehensive feasibility and engineering review of the reaction process, and a formulation of the design for a test system to experimentally verify the high temperature reaction.

Phase II, currently underway, involves a multi-task approach including (1) a detailed engineering analysis of the entire process (2) design, fabrication, and assembly of the experimental system (3) experimental testing of the reduction reaction to produce silicon and (4) complementary research programs to augment the experimental

system design. The Phase II effort was initiated in October, 1977, and work is progressing on the various stages of this effort.

2. PROJECT SUMMARY

During the report period, the procurement, fabrication, and assembly activities were completed for the silicon experimental verification unit. Installation of all the subsystems was initiated and completed for all subsystems except the I&C, sodium and SiCl_4 systems. Numerous shakedown tests were conducted for the subsystems as installation was completed. The following subsystems were operated satisfactorily during the initial power tests of the arc heater-reactor assembly on gas only (no reactants): arc heaters-reactor, cooling water, gas feed system, electrical power system, effluent scrubber, gas burnoff, and a portion of the I&C system.

Two shakedown tests (without reactants) were conducted to evaluate operation of the arc heater-reactor system. The first test utilized arc heated argon only at a power level of 110 kW; while the second power test was run on the Ar-H_2 mixture at a power level of 700 kW.

The experimental stage of the project is complete for the Injection Techniques Task. A satisfactory rationalization of the laser imaging results for the particle size produced by the Sonicore sodium nozzle has been determined from further computer analysis. These results are compared with the sodium sieving data and give an overall picture of the nozzle characteristics. The silicon tetrachloride nozzle has been tested and high speed photographs indicate that its operation was successful.

A summary report of the Injection Task is being prepared and decontamination of the test chamber is in progress. Final recommendations for the sodium and silicon tetrachloride reactant systems have been relayed to the personnel responsible.

The Kinetics Task apparatus became fully operational for studying

the reduction of SiCl_4 by Na and the collection of the resulting products. Several minor modifications allowed the operating time at nominal power and design flow rates to increase continuous operation to more than 15 minutes. The product was collected on the walls of a SiC tube of 1" I.D., and experiments were completed to determine the amount of silicon collected as a function of axial location in the flow duct. The results show good agreement between experiment and theoretical predictions.

Full scale testing has been initiated for the Reaction Demonstration studies. Repeated clogging of the liquid sodium metering valve orifice has led to the abandonment of this method for monitoring the sodium flow rate. Another method which makes use of the vapor pressure-temperature characteristics of liquid sodium has been pursued, and testing at lower tube wall temperatures (1300°C) was initiated. Test products have been analyzed as being elemental silicon and sodium chloride.

3. TEST SYSTEM PREPARATION

3.1 Summary

During the report period, the procurement, fabrication, and assembly activities were completed for the silicon experimental verification unit. Installation of all the subsystems was initiated and completed for all subsystems except the I&C, sodium and SiCl_4 systems. Numerous shakedown tests were conducted for the subsystems as installation was completed. The following subsystems were operated satisfactorily during the initial power tests of the arc heater-reactor assembly on gas only (no reactants): arc heaters and reactor, cooling water, gas feed system, electrical power system, effluent scrubber, gas burnoff, and a portion of the I&C system.

3.2 System Components

3.2.1 Instrumentation & Control (J. W. George, I. N. Bova)

The procurement activity for the I&C system was completed, while the assembly-installation effort was initiated during the period. Fabrication and assembly of the three control panels (i.e., arc heater panel, sodium system panel, and the process control panel) are underway with mounting of components, switches, and internal wiring. Interconnecting wiring from panel to panel was initiated and has continued for the I&C system. Also, the pneumatic portion of the I&C system which is used for control sensing with P/E conversion at the process control panel is in progress.

Installation of the differential pressure flow controls, local flow indicators and switches, and RTD units for the cooling water system were installed and tested. Likewise, the ratio control system that

will control the injection of reactants (Na & SiCl_4) into the reactor were installed in the control panels.

Photographs of the partially completed control panels are presented in Figure 3.1 (arc heater panels), 3.2 (sodium system panels), and 3.3 (process control panel). Shakedown tests of the completed portions of both the arc heater panel and process control panel have been started.

3.2.2 Plasma Reactor/Separator (C. B. Wolf, T. N. Meyer)

In the previous report,⁴ it was noted that a thermal expansion analysis of the various reactor liner parts was made. This final analysis was necessary in order to allow for the thermal properties of the graphite materials available in the sizes and shapes required. The original analysis was based on the properties of Stackpole 2128 graphite. This material was available only in square or rectangular shapes. As a result of size and shape, the materials finally selected were Stackpole's grade 2020 and Great Lakes Carbon grade HLM. The variations in properties are attributable mainly to manufacturing methods such as isostatic pressing, molding, and extrusion. Typical thermal properties as shown in Table 3.1 illustrate these variations.

Grade HLM has the advantages that it is less expensive and is available in large sizes. The disadvantages are that less data is available and the ash content (impurity level) is somewhat higher. Thus, the HLM grade material is being used in the areas where heat transfer is lower and dimensions are not as critical. All reactor sections are being fabricated from Stackpole 2020.

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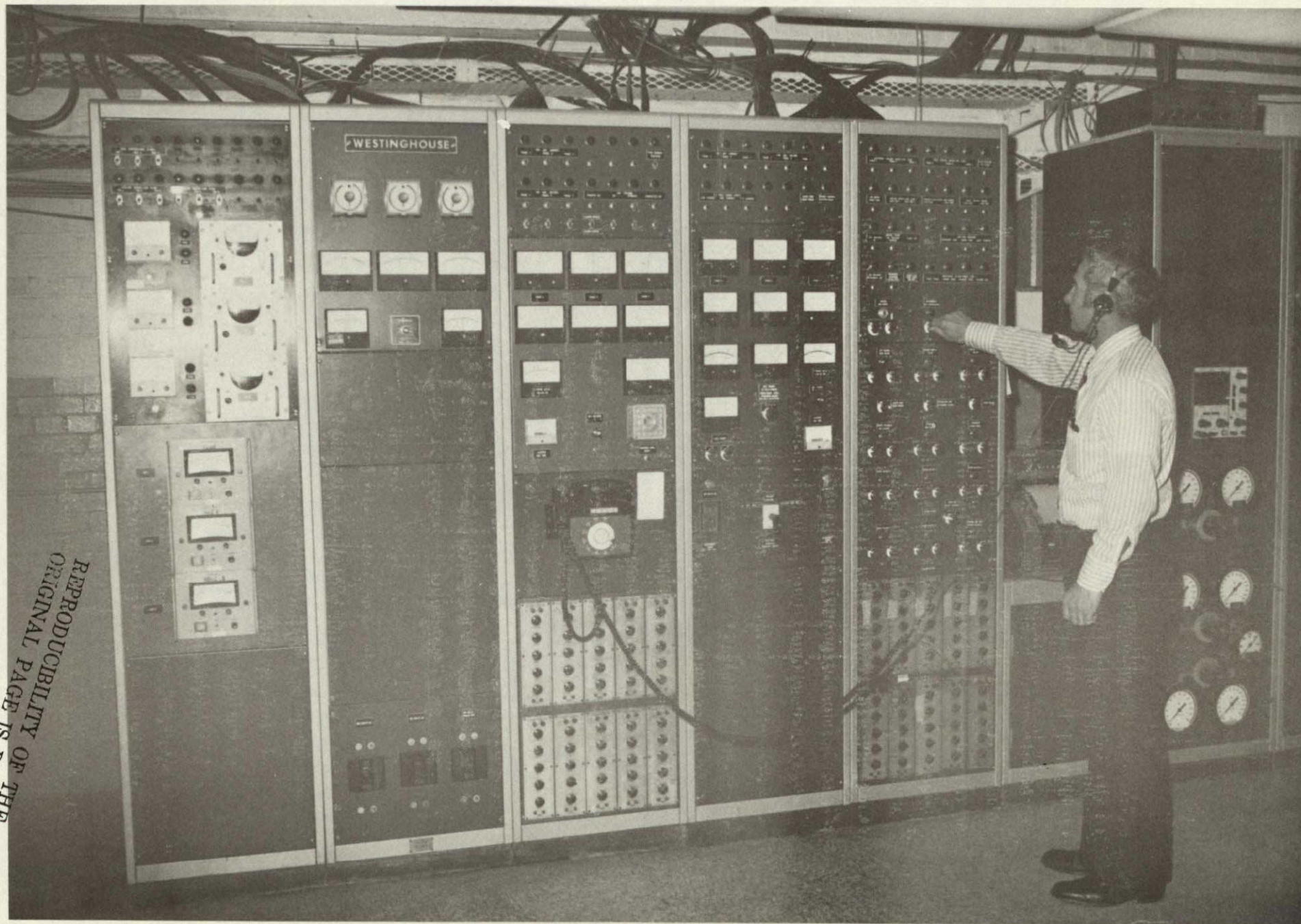


Figure 3.1 - Photograph Of The Arc Heater Control Panel

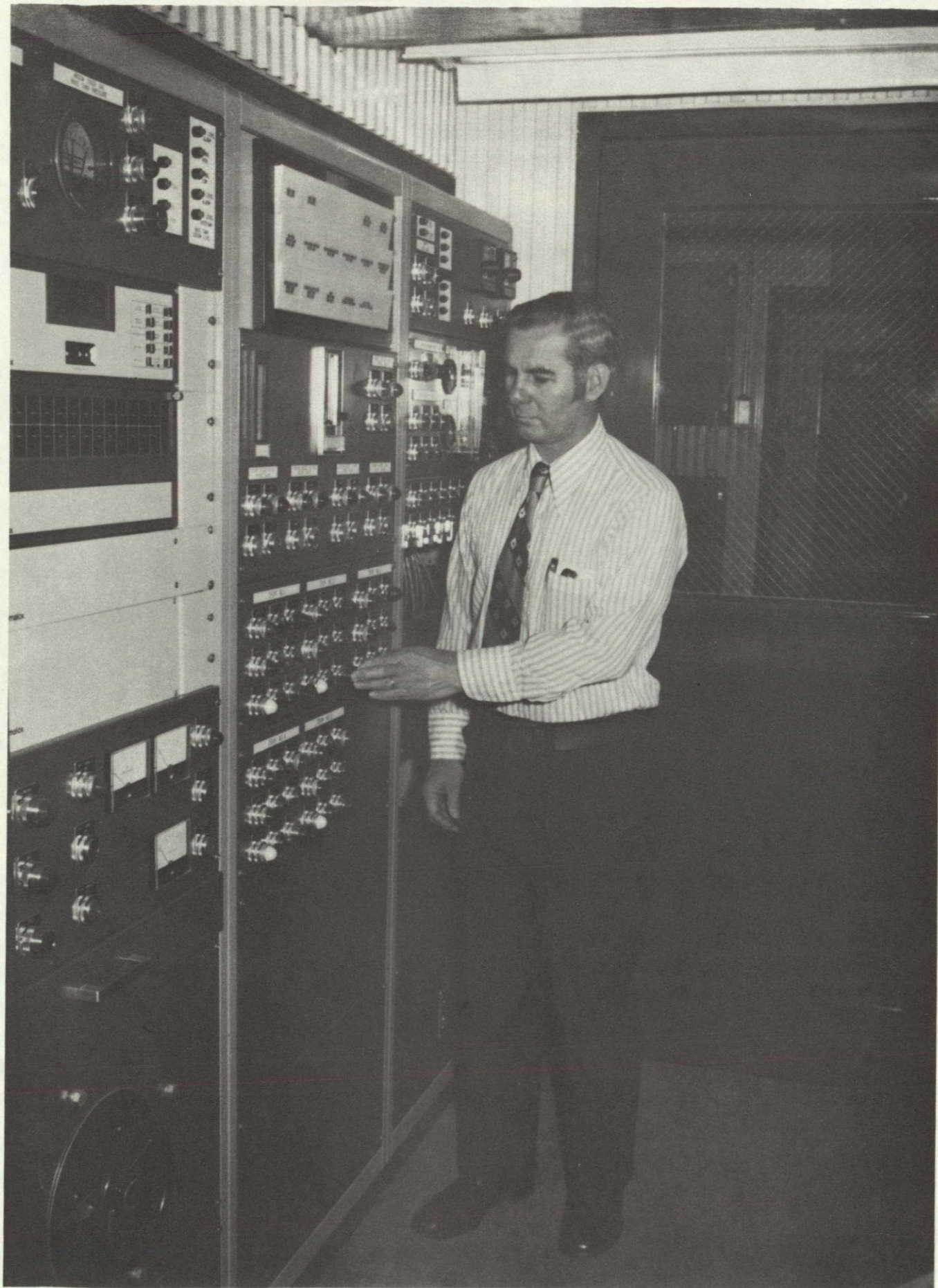


Figure 3.2 - Photograph Of The Sodium System Control Panel

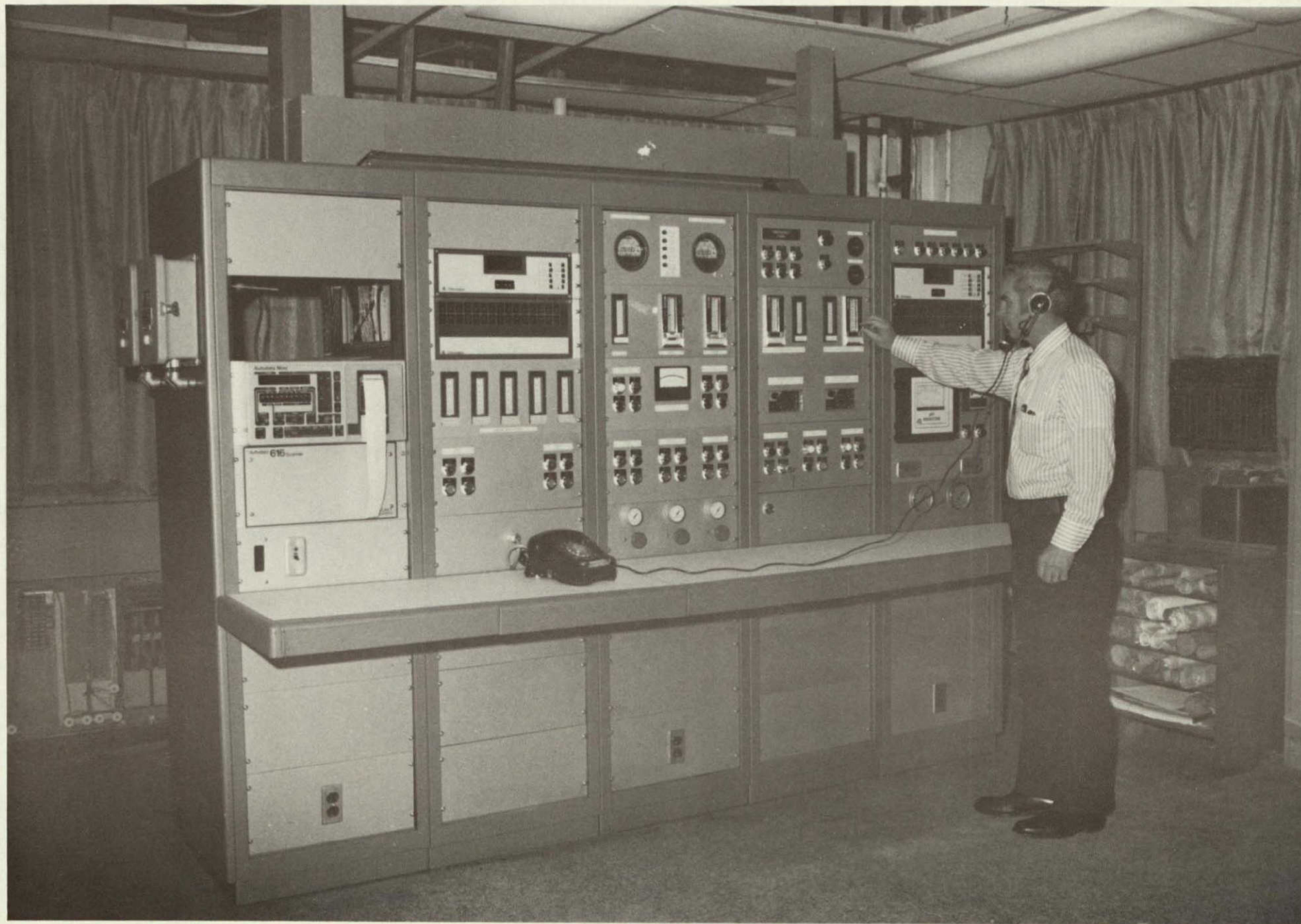


Figure 3.3 - Photograph Of The Process Control Panel

Table 3.1

Thermal Properties Of Candidate Graphites

<u>Graphite Type</u>	<u>Temp. (^oC)</u>	<u>Coefficient Of Thermal Expansion</u>		<u>Thermal Conductivity</u>
		<u>Axial (1/^oC)</u>	<u>Radial (1/^oC)</u>	<u>Watts/cm ^oC)</u>
Stackpole 2128 (Isostatic Pressed)	25	4.1×10^{-6}	4.1×10^{-6}	.63
	600	5.3×10^{-6}	5.3×10^{-6}	1.11
	1000	5.8×10^{-6}	5.8×10^{-6}	.78
	1500	6.6×10^{-6}	6.6×10^{-6}	.38
Stackpole 2020 (Molded)	25	3.3×10^{-6}	2.5×10^{-6}	.61
	600	3.8×10^{-6}	3.3×10^{-6}	.45
	1000	4.2×10^{-6}	3.7×10^{-6}	.35
	1500	4.6×10^{-6}	4.1×10^{-6}	.26
Great Lakes Carbon HLM (Extruded)	25	2.7×10^{-6}	2.7×10^{-6}	1.38 (With Grain)
	600	*	*	*
	1000	*	*	*
	1500	*	*	*

*Data Not Available

The objective of the liner design is to maintain a silicon skull wall of reasonable thickness throughout the reactor and cyclone sections. The variables under control are the liner materials and the gas gap between the liner and the water cooled shell. All reactor and cyclone parts are being internally coated with pyrolytic graphite which serves as a seal to the graphite and provides an additional thermal resistance. In the reactor sections, where the heat transfer rates are high, it is important that the tolerances of the gas gap are minimized. Thus, the bores of the shells and the outer surface of the graphite pieces are machined to close tolerances. Thermal expansion of mating parts are taken into account to minimize the gap without over stressing the parts due to differential thermal expansion.

Heat flow requirements of the various sections have been established by prior calculations.^{3, 4} The results are shown pictorially in Figure 3.4.

The approach to the problem of optimizing the liner design utilized an iterative series of calculations to determine the effects of gas gaps, material properties, and pyrolytic layer thickness upon the skull wall thickness. A program was used to compute the temperature profile and equilibrium silicon thickness of the various reactor sections as a function of the thermal conductivities, thickness of materials, and heat transfer rate. In addition, the program includes the temperature profile of the steel outer shell. When the average temperature of the steel shell and graphite liner are known, the differential thermal expansion is determined; thus dimensions of the graphite liners can be established to allow for the differential thermal expansion.

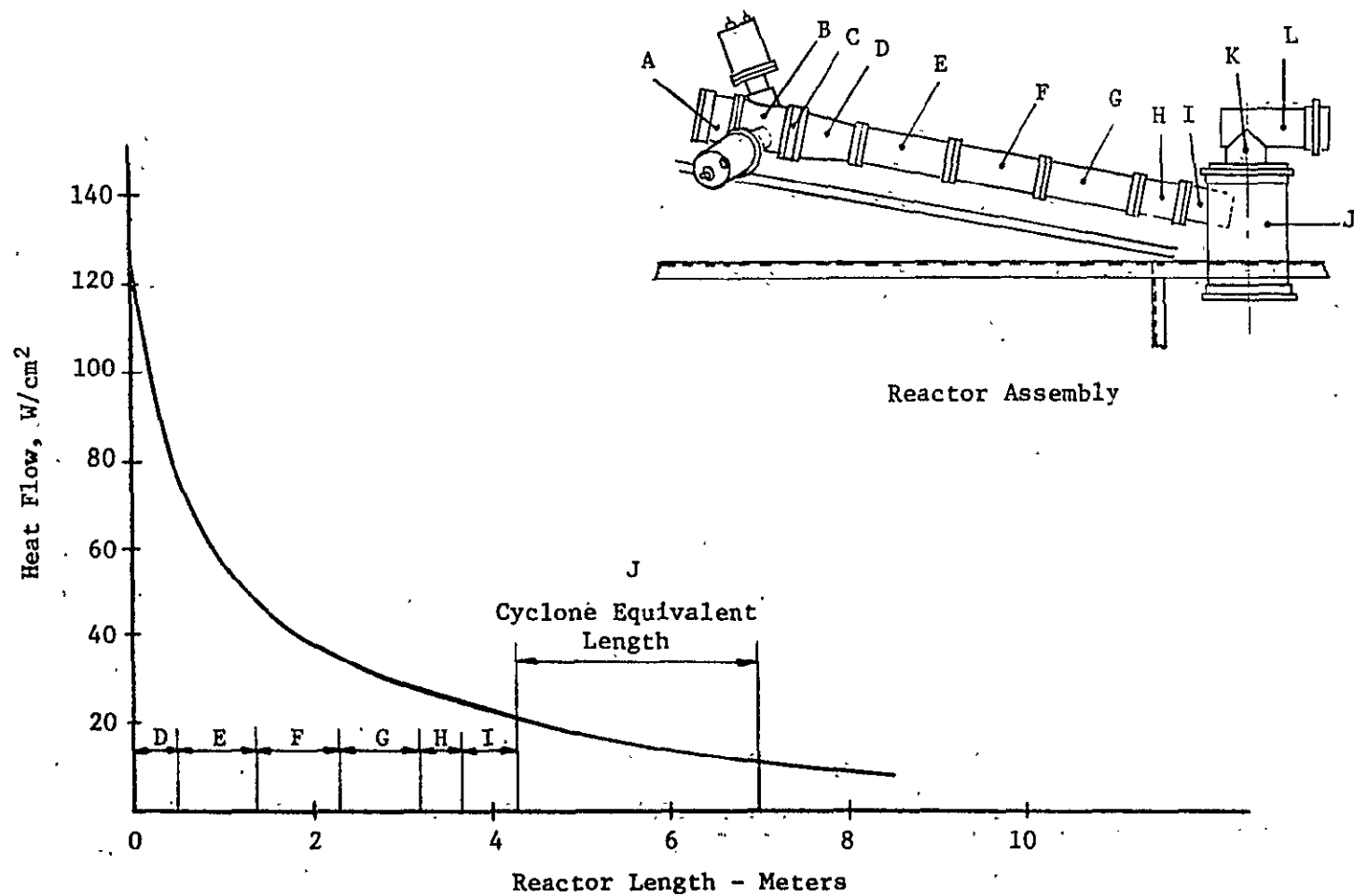


Figure 3.4 - Heat Flow vs. Reactor Length

A relationship is used to establish the thermal profile and silicon equilibrium thickness (see Figure 4.2 of reference 4 for example) within the reactor sections. Radiative heat transfer in the gas gap is taken into account. Figure 3.5 illustrates a portion of the reactor cross section. As shown, heat flow rate varies as a function of the inner skull radius relative to the solution for the 15 cm internal skull diameter solution (see Figure 4.2 of reference 4).

Having established the temperature profile of a particular reactor section, the differential thermal expansion of the liner and steel shell can be determined so that the outside machined diameter and length of the graphite liners can be fixed.

Similar programs were used to determine the temperature profiles and thermal expansions for both the arc heater plenum and cyclone sections. However, a silicon skull does not exist in the arc heater plenum. In the case of the cyclone, the rate of heat flow to the wall depends upon the inlet conditions rather than the inner radius of the silicon skull. The inner skull radius of silicon in the cyclone depends upon the convective heat flux in nearly a proportionate manner (i.e., a 10% change in the wall heat flux creates roughly a similar change in the internal skull radius). The desired skull thickness can be controlled by controlling the gas temperature and flow through the cyclone. Ultimately, the cyclone can be relined to operate at a given internal diameter for the desired operating temperature and flow rate.

Both the procurement and fabrication activities were completed for the reactor/separator system. The

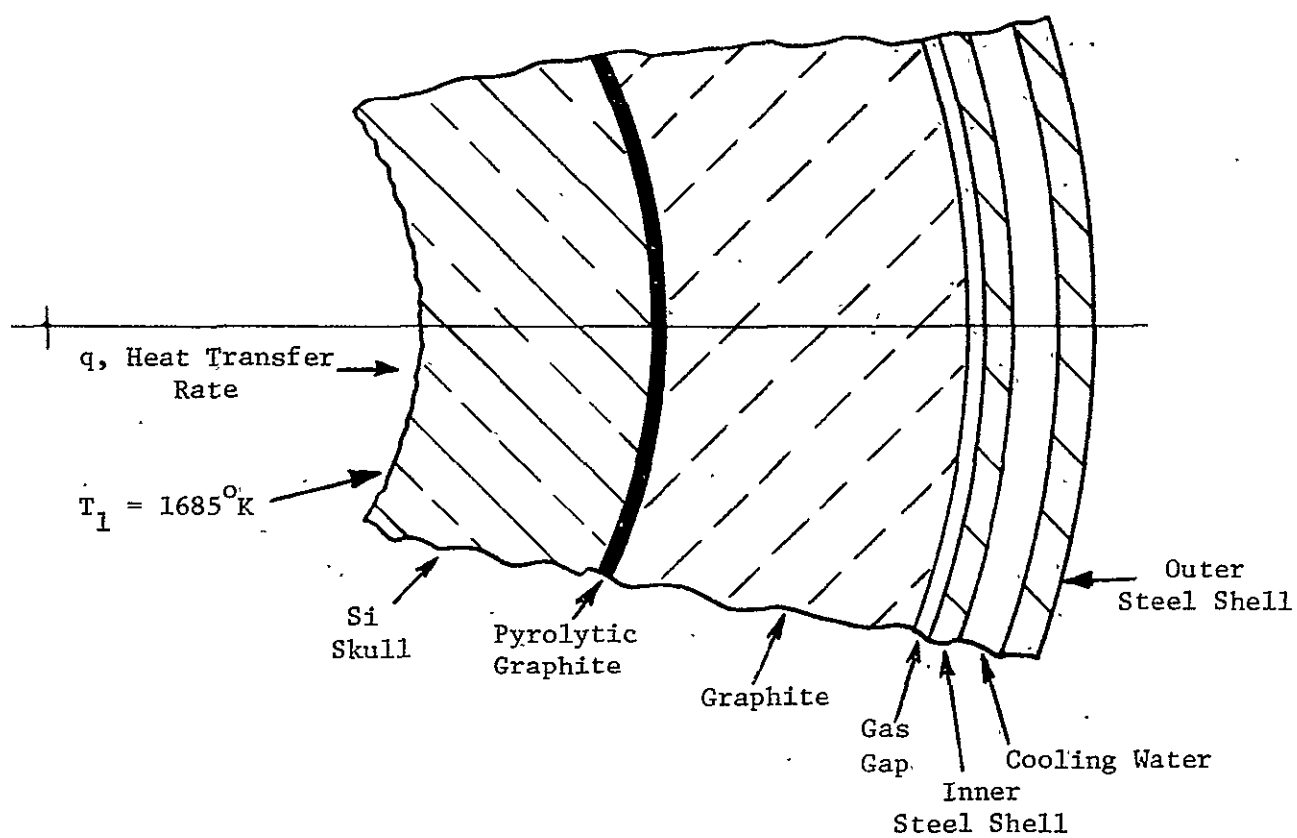


Figure 3.5 - Cross Section Of A Typical Reactor Section

nickel plated-reactor shells, graphite liners, and the cyclone-separator unit were all received and assembled onto the reactor support frame (see Figure 3.11). Drawings which depict the assembly of the reactor shells and respective liners are presented in Figures 3.6-3.10.

Installation of the arc heater-reactor system was initiated with the reactor assembly and arc heaters installed, cooling water piping attached, gas system piping connected and electrical leads coupled. The arc heaters were tested for electrical integrity and water pressure checked. Figure 3.11 presents a view of the arc heater-reactor assembly mounted on the frame within the test cell. Key reactor system components are labeled on the figure. One of the three-phase arc heaters from the reactor is presented in Figure 3.12 along with its protective outer cover shown in the left background.

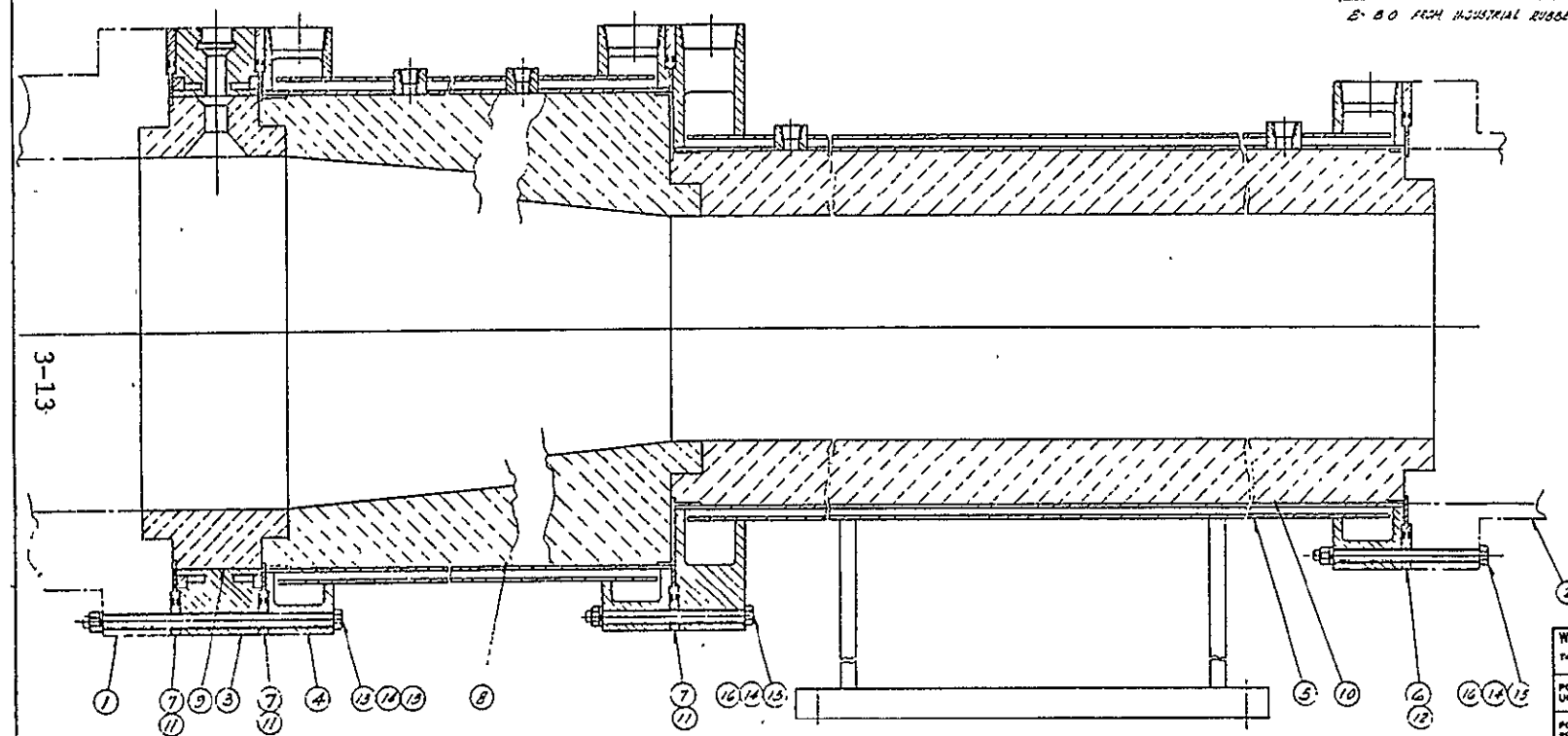
Operational shakedown tests of the arc heater-reactor were initiated for the cooling water, arc heater gases, and arc heater ac and dc power. Power tests of the arc heaters and reactor assembly with Ar-H₂ gas only (no reactants) were conducted (see Section 4 - Testing).

3.2.3 SiCl₄ System (R. E. Witkowski, P. A. Ciarelli, G. C. Burrow)

For this quarter the studies were directed to the task areas summarized below. They include efforts towards SiCl₄ storage tank fabrication and delivery, injection system assembly, and determining a method of SiCl₄ chemistry quality control.

Drawings for the SiCl₄ storage tanks were finalized

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TITLE					DATE	
PCB RING REPAIR SILICON RING SET					REV. 1	
SICL ₄ INJECTOR & REACTOR RING SET					REV. 2	
7351D57					REV. 3	
ITEM	NOTE	PART NAME	DEF	(SIZE) REFERENCE INFORMATION	MAF. & QTY CODE	QTY
1		ASSY		SILICON RING & REACTOR	100-0000	1
2		FLAT		REACTOR	100-0000	1
3		RING		REACTOR	100-0000	1
4		REACTOR W.C.		REACTOR	100-0000	1
5		FLAT (H)		REACTOR	100-0000	1
6		FLAT RING		REACTOR	100-0000	1
7		FLAT RING		REACTOR	100-0000	1
8		FLAT RING		REACTOR	100-0000	1
9		FLAT RING		REACTOR	100-0000	1
10		FLAT RING		REACTOR	100-0000	1
11		FLAT RING		REACTOR	100-0000	1
12		FLAT RING		REACTOR	100-0000	1
13		FLAT RING		REACTOR	100-0000	1
14		FLAT RING		REACTOR	100-0000	1
15		FLAT RING		REACTOR	100-0000	1
16		FLAT RING		REACTOR	100-0000	1

E. B. BO FECH INDUSTRIAL RUBBER PRODUCTS CO (PARICA SEALS)

Westinghouse Electric Corporation

TITLE PCB RING REPAIR SILICON RING SET

SICL₄ INJECTOR & REACTOR RING SET

PCB SPI OR SPECIFIED MPQ. TOLERANCES UNLESS SHOWN ON FACE OF DRAWING

PCB SPI OR SPECIFIED GENERAL PRODUCT SPECIFICATIONS, PRACTICES

Sign Off Location

DATE

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REV. 2

REV. 3

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Figure 3.7 - Assembly Drawing Of The SiCl₄ Injector Ring And Reactor Sections

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2	ASST	102	102	102	102
3	ASST	103	103	103	103
4	ASST	104	104	104	104
5	ASST	105	105	105	105
6	ASST	106	106	106	106
7	ASST	107	107	107	107
8	ASST	108	108	108	108
9	ASST	109	109	109	109
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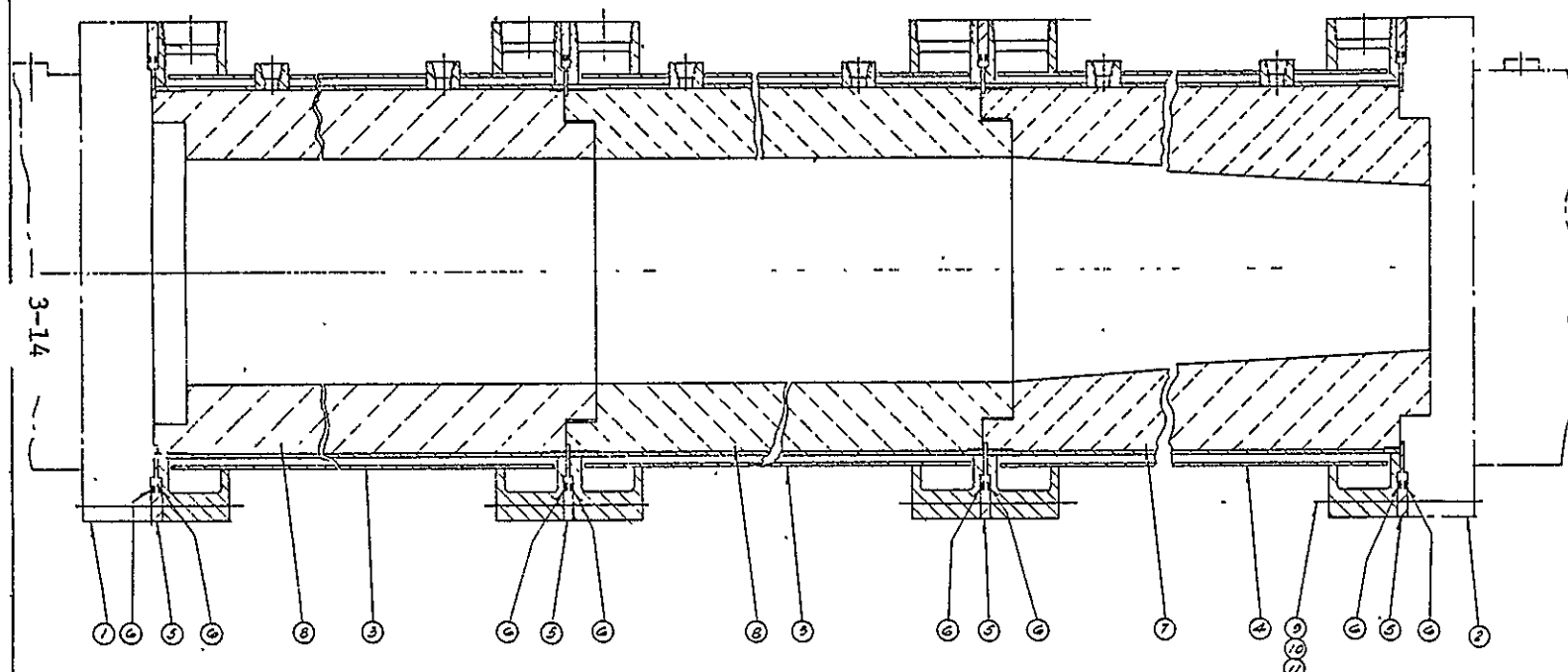
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Figure 3.8 - Assembly Drawing Of Reactor Sections

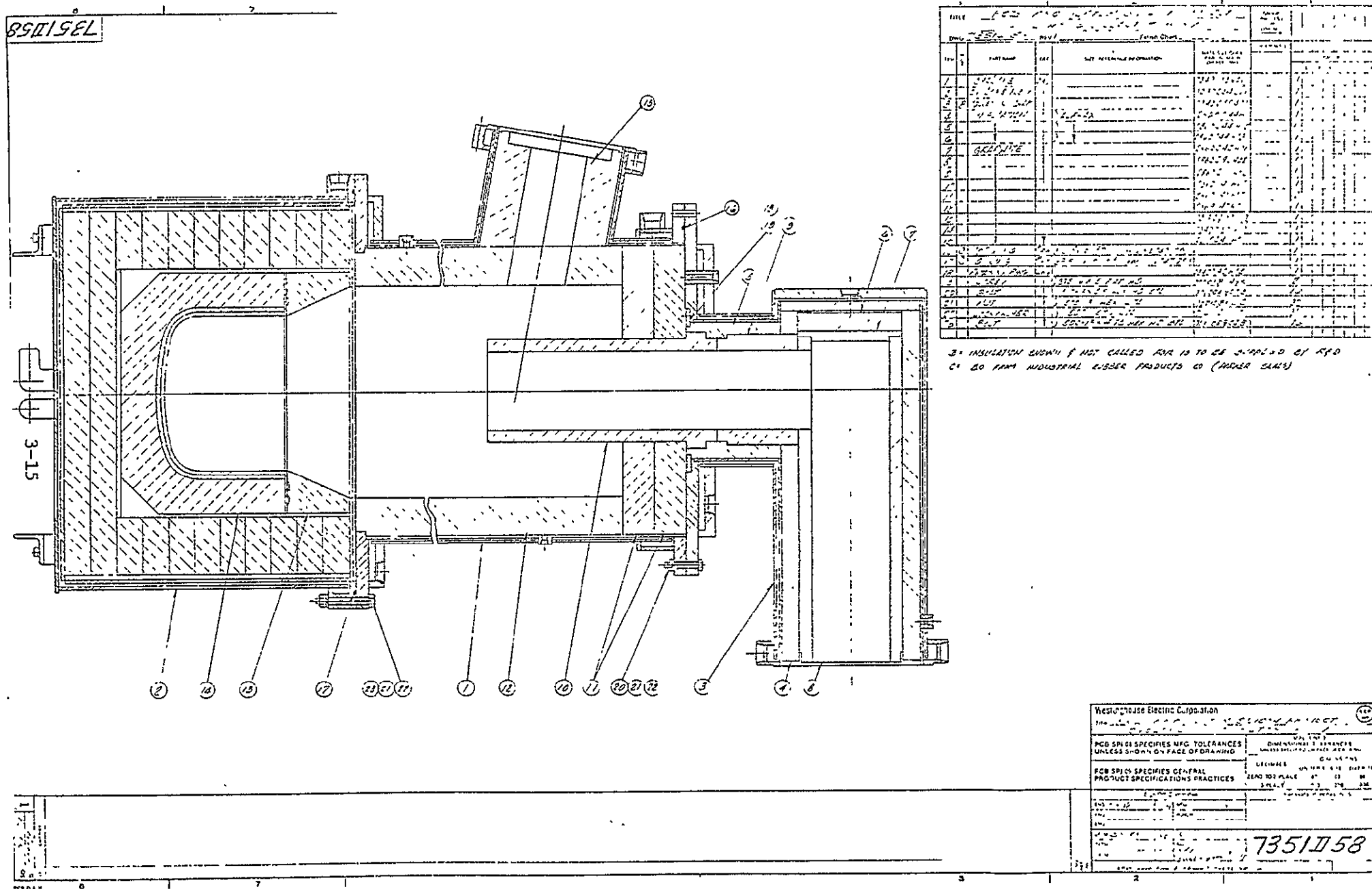
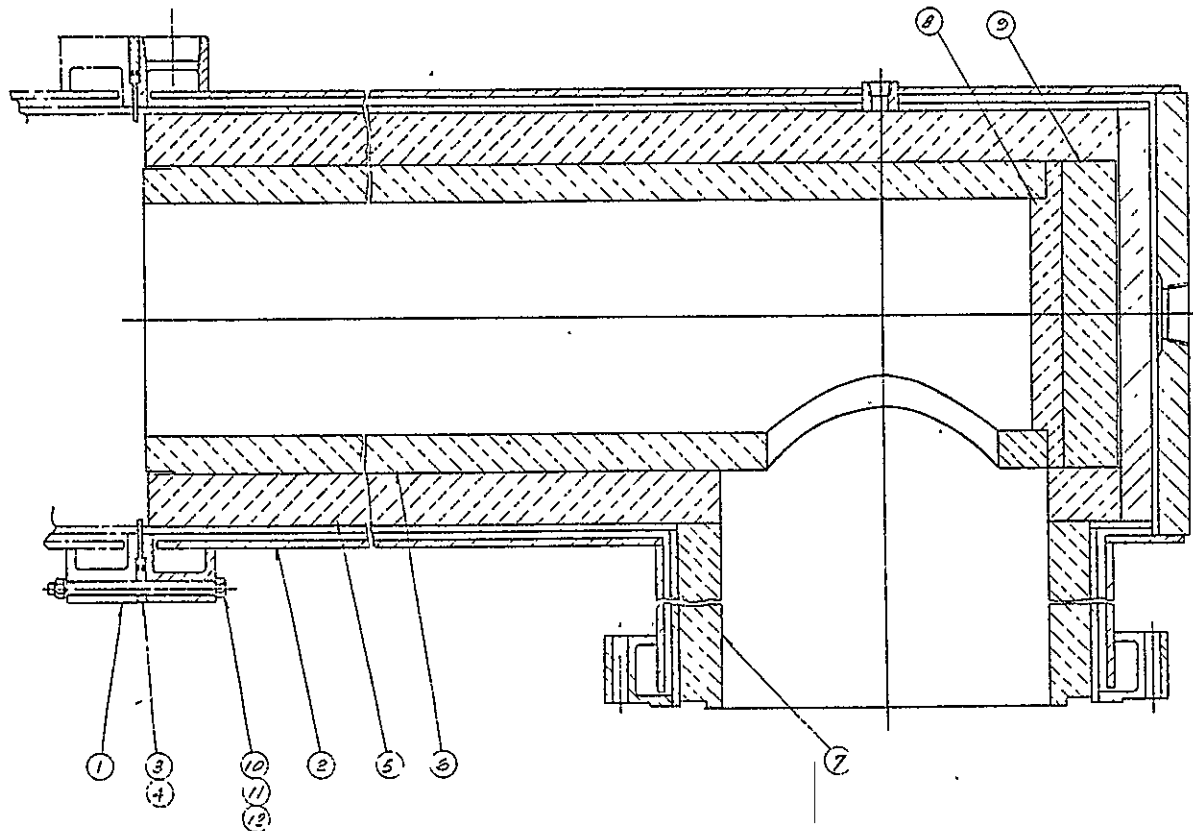


Figure 3.9 - Assembly Drawing Of Silicon Cyclone And Collector

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TITLE PCB ARC WELDER SLIDE-IN MODIFIED				DATE		BY	
REV				DATE		BY	
DWG 7351756				DATE		BY	
ITEM	QTY	PART NAME	QTY	DATE		BY	
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2	1	FLANGE	1	DATE		BY	
3	1	FLANGE	1	DATE		BY	
4	1	FLANGE	1	DATE		BY	
5	1	FLANGE	1	DATE		BY	
6	1	FLANGE	1	DATE		BY	
7	1	FLANGE	1	DATE		BY	
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Figure 3.10 - Assembly Drawing Of Gas Scrubber Inlet Section

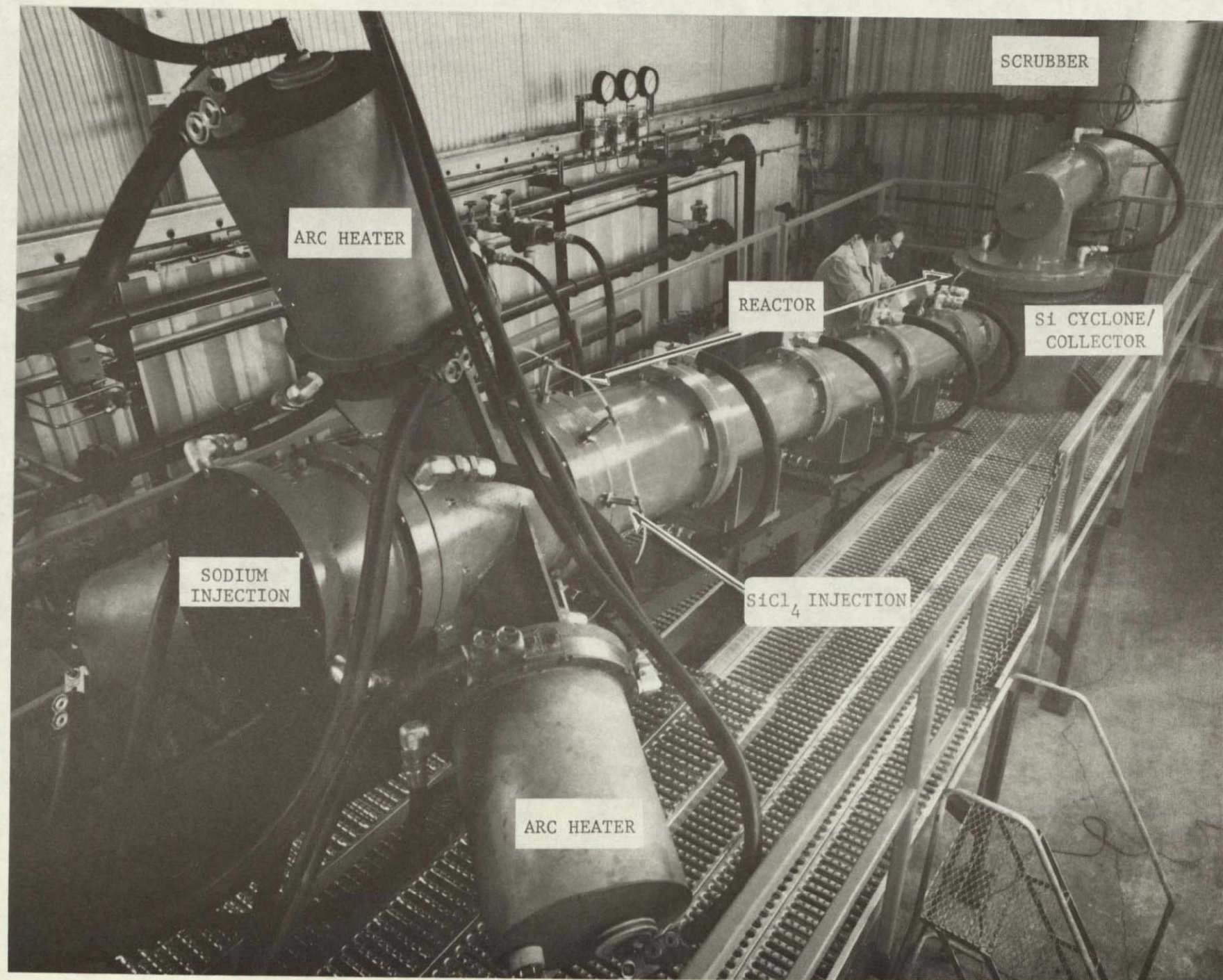


Figure 3.11 - Photograph Of The Arc Heater-Reactor For Experimental Silicon Production.

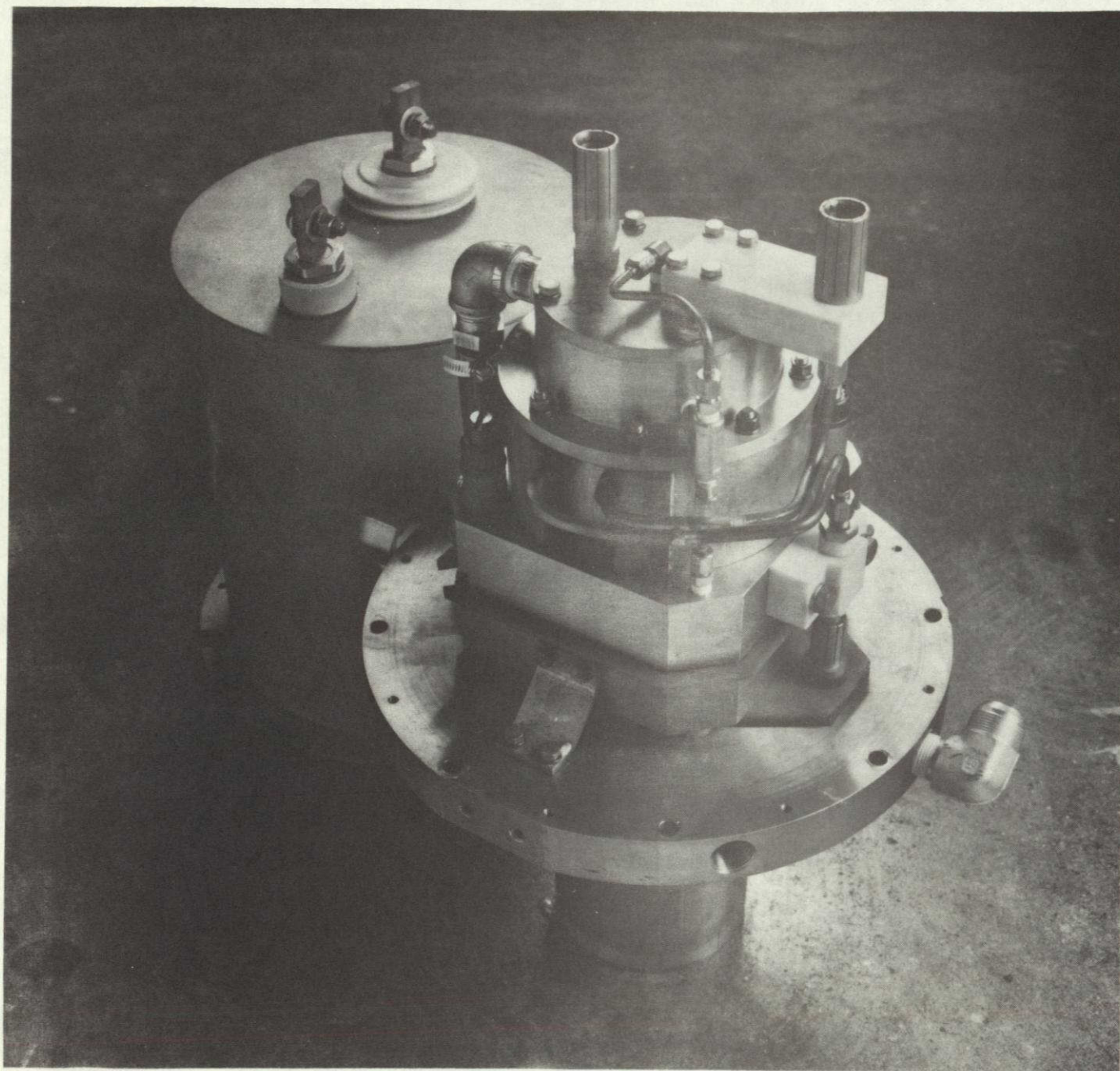


Figure 3.12 - Photograph Of The Westinghouse Arc Heater Used For The Silicon Process Experimental Verification Unit

with Buffalo Tank making changes and additions per Westinghouse recommendations. Surface preparation and application of the recommended polyurethane paint for the tank's outside finish were determined. The requirements for tank cleanup and drying prior to shipment were also reviewed with the vendor. Fabrication of the tanks was completed and delivery was made to the Arc Heater Lab. All tank fittings have been prepared and helium leak checked. The preparation of all tank nozzle attachments was also completed. The SiCl_4 storage tanks were installed in the storage building, cleaned, and prepared for SiCl_4 filling (see Figure 3.13).

With the continued assembly of the SiCl_4 injection module, major components were installed. The SiCl_4 loop accumulator was disassembled, cleaned, and rebuilt. It has been installed in the module along with the positive displacement pump, main control valve, and dump tank. All instruments were installed and plumbing is nearly complete in the module control panel. Figures 3.14 and 3.15 detail the status of the control panel components and injection module components.

In an effort to find alternate methods for qualifying the SiCl_4 feedstock, the Westinghouse R&D Analytical Chemistry Department has defined analytical methods for the grab sample analysis of SiCl_4 . The free chlorine will be determined by titration, free HCl spectrophotometrically (Cl^- ion), iron, chromium, and nickel by atomic absorption, the SiCl_4 boiling range - ASTM distillation, SiCl_4 assay-acid/base titration, and gas evolution by alkaline hydrolysis.

Detailed procedures for handling large SiCl_4 spills

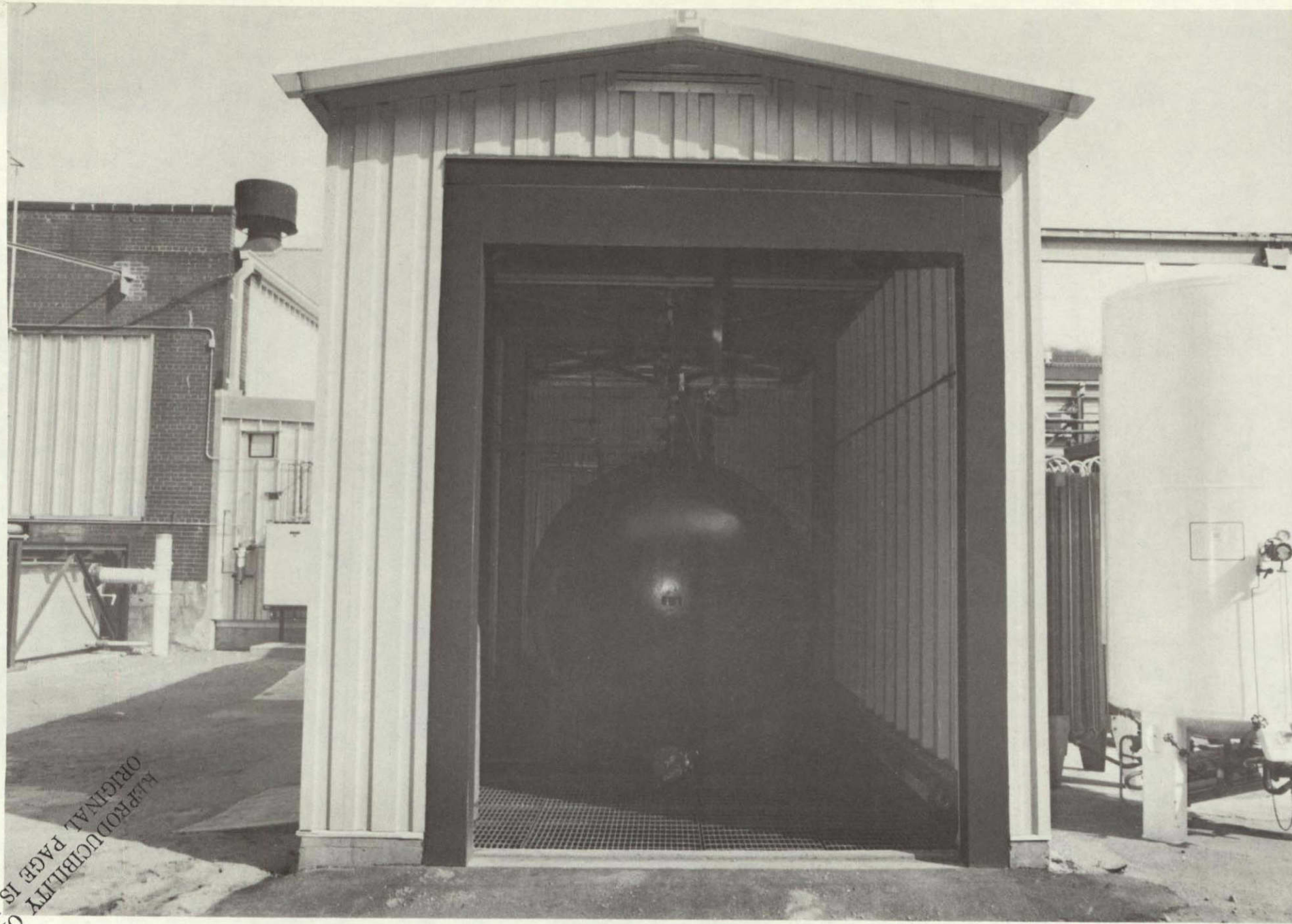


Figure 3.13 - Photograph Of The Main SiCl_4 Storage Tank Installation

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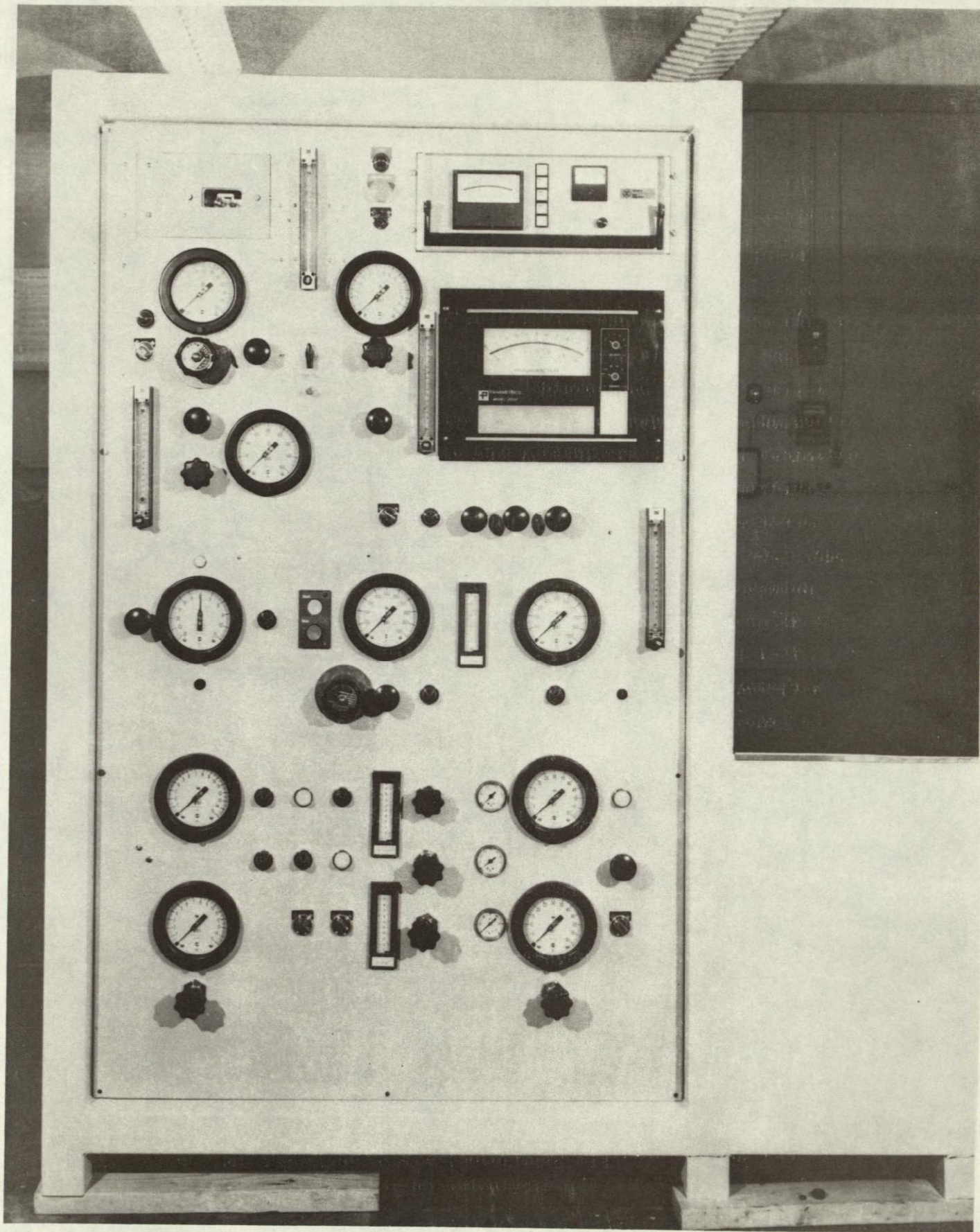


Figure 3.14 - Front Panel For The SiCl₄ Injection Module

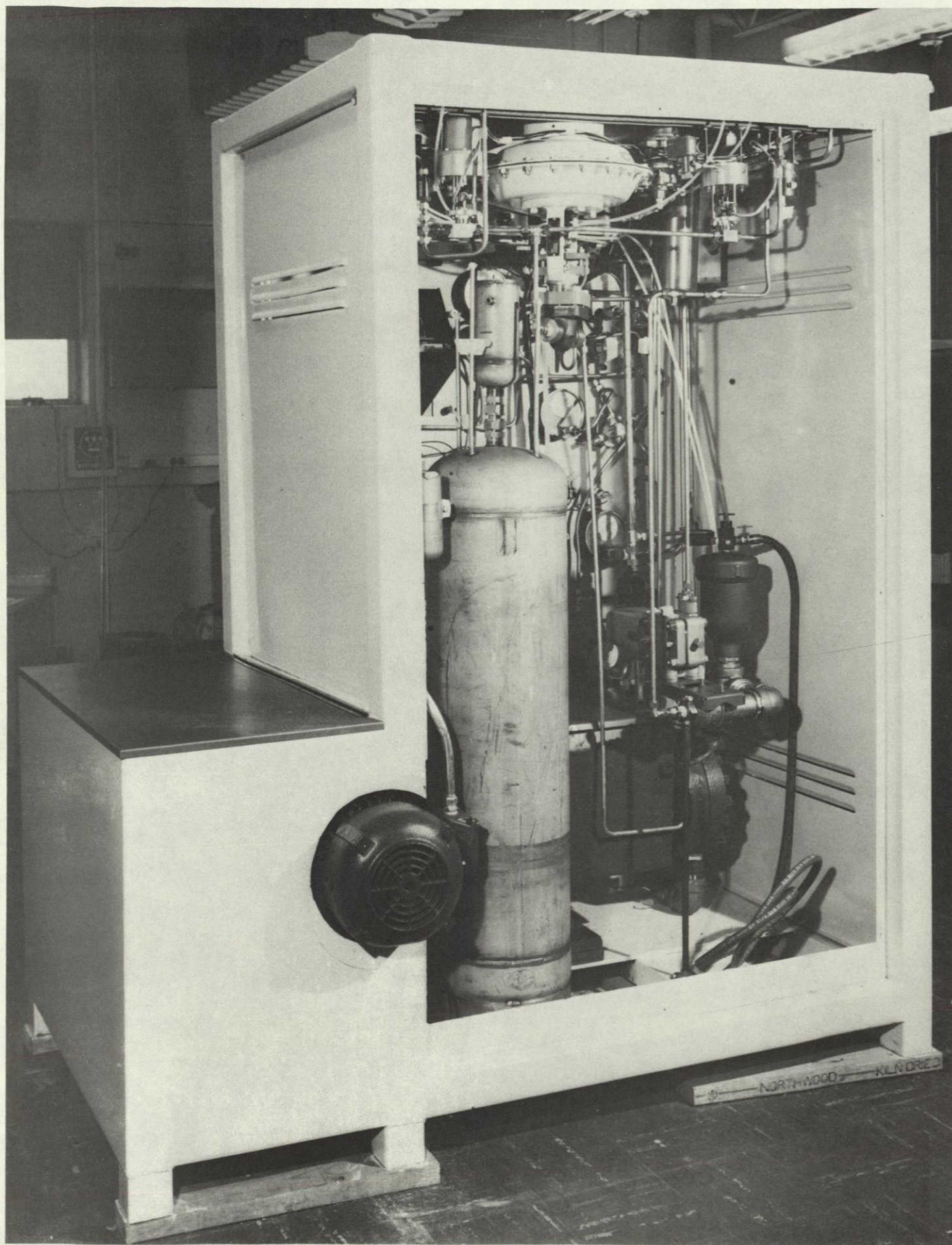


Figure 3.15 - Rear View Of The SiCl_4 Injection Module Showing Loop Components

were finalized with the safety and decontamination personnel. Electrical and piping requirements for the SiCl_4 supply system routing were finalized and installation started.

3.2.4 Sodium System (A. R. Keeton, D. Marschik)

Fabrication of components for the sodium storage and injection system and the NaK thermal control system was completed. All of the components for the sodium and NaK systems have been installed in the sodium room as shown in Figure 3.16. Interconnecting piping has been fitted, cleaned and welded in place. Dye penetrant inspection of welds is complete and a pneumatic pressure test of the two systems is scheduled. Installation of trace heaters and instrumentation has been initiated.

The sodium injection nozzle holder is shown in Figure 3.17 prior to final assembly. The Sonicore nozzle is located at the lower right of the photograph and the NaK flow channels can be seen on the two ribbed cylinders in the center. After final assembly the nozzle holder flange at the top left will mate with a similar flange at the end of the chemical reactor.

The NaK thermal control system has been installed in a modular framework shown in Figure 3.18. The NaK sump tank is located at the bottom while the EM pump is the rectangular component in the center with the perforated metal housing. The NaK to air heat exchanger is located behind the pump and the NaK heater is the vertical section at the right of the photograph.

Detailed operating procedures were prepared for the

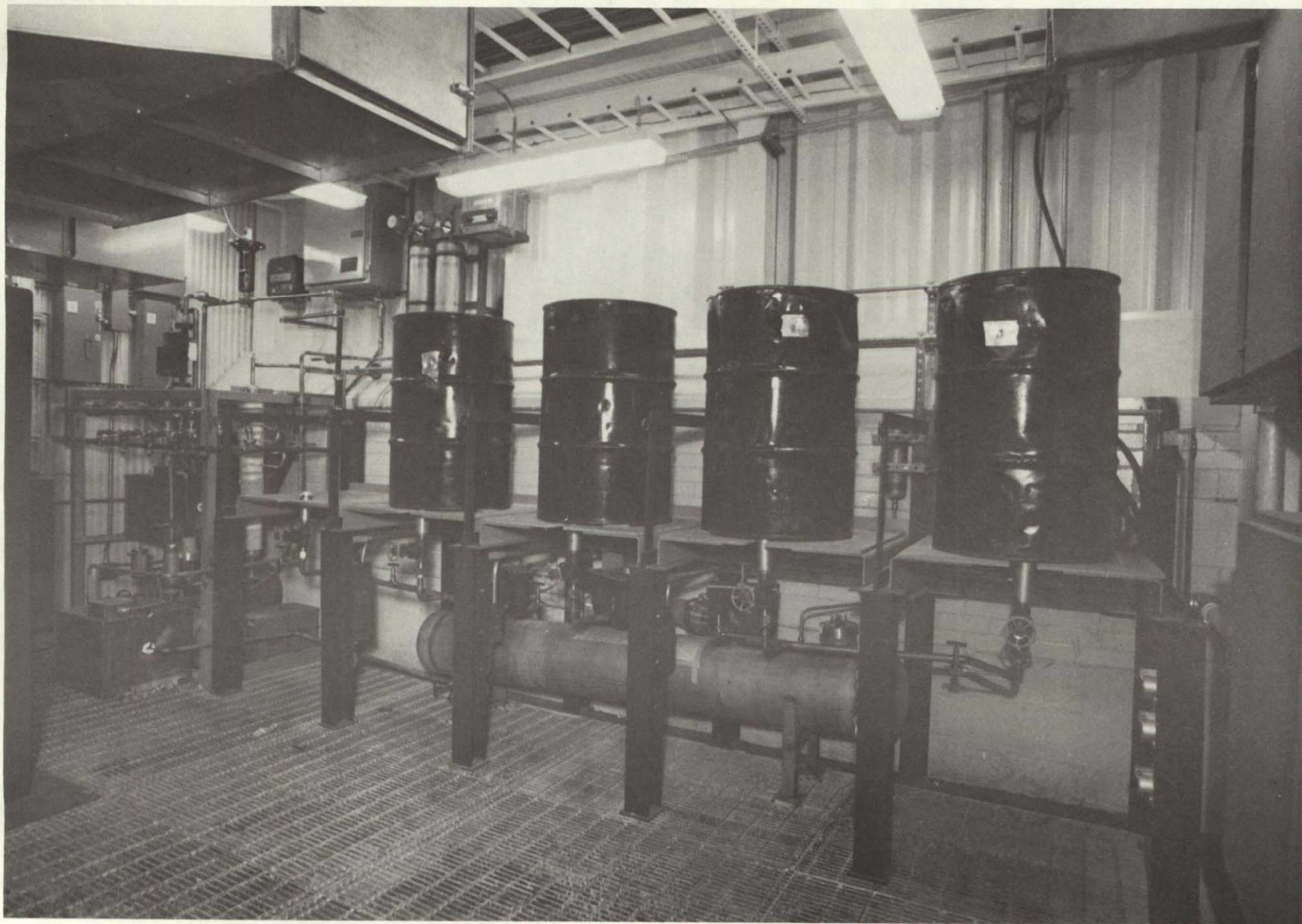
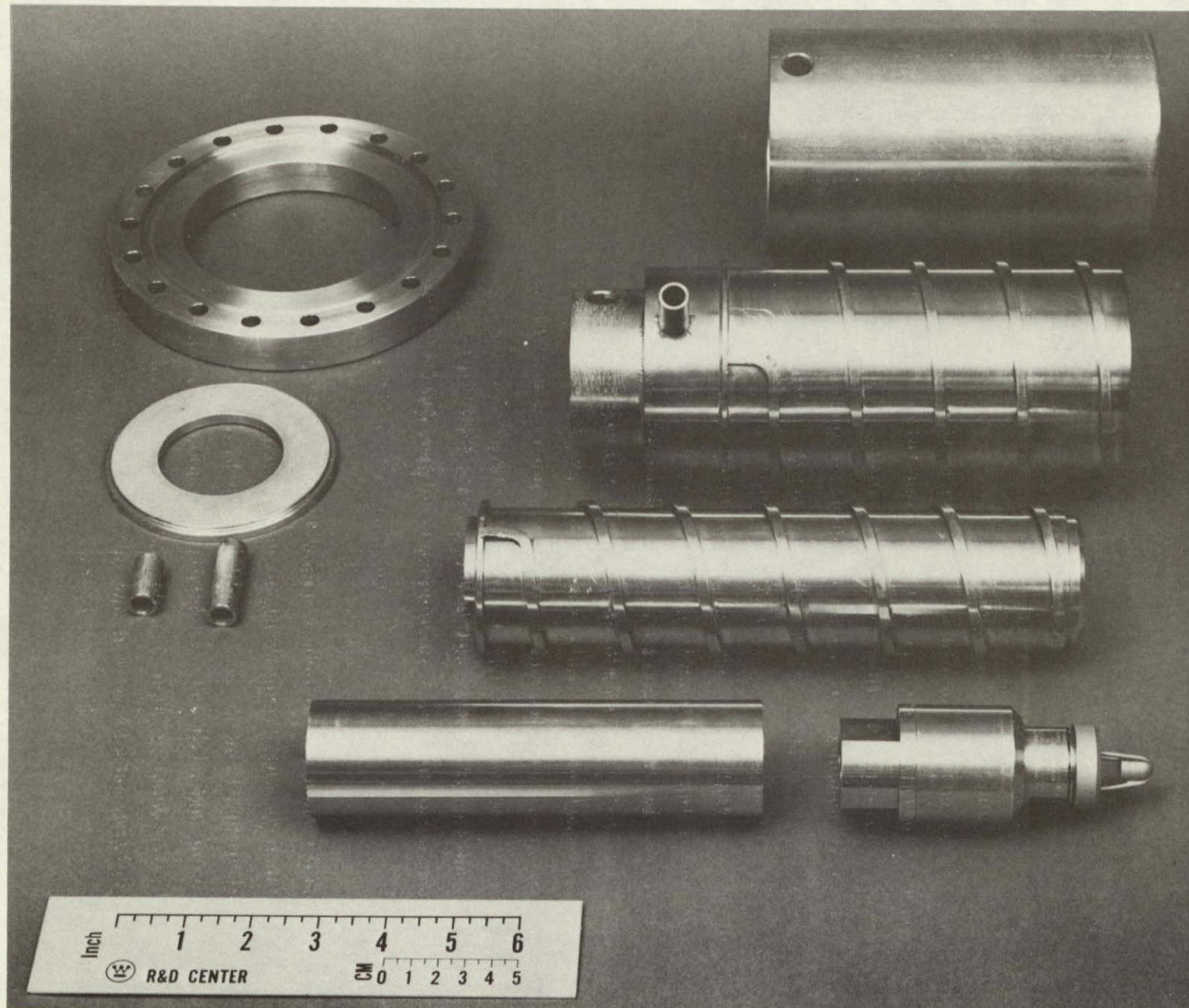


Figure 3.16 - Photograph Of Sodium System Installation



- 3.17 - NaK Heated/Cooled Sodium Injection Nozzle Ready For Final Assembly

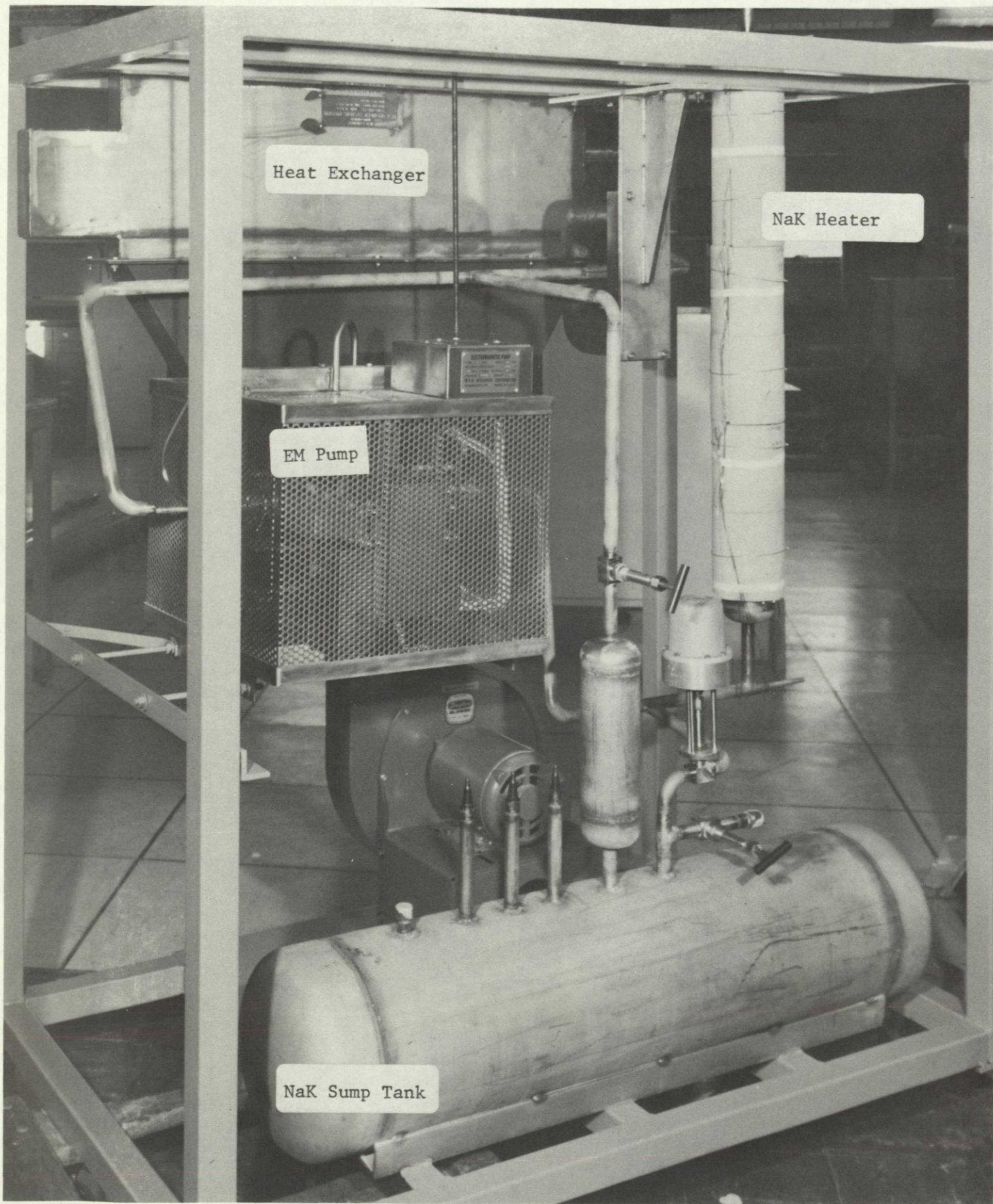


Figure 3.18 - NaK System Module

sodium and NaK systems and are included in Appendix I. These cover all modes of operation including normal and emergency procedures as well as start-up, shutdown and standby operations. Safety precautions and practices as well as cleaning of alkali metal contaminated components were also included in the procedures.

3.2.5 Silicon Collector (I. E. Kanter)

During this period the collector housing and graphite internals were received. The housing was nickel plated and the interface ring was pyrocarbon coated. This completes hardware procurement activity.

The collector components have been assembled into the housing, the completed unit shipped to the Arc Heater Lab, and installed onto the reactor-separator assembly. This collector contains an expendable cast liner with an approximate 25 liter capacity for use during the shakedown tests.

An expendable collector is under fabrication to replace the collector, fused quartz, and felted components as explained in the previous quarterly.⁴ In order to gain experience with the castable insulation for this collector modification, a 1 gallon and 5 gallon simulation were cast and cured. The 5 gallon casting appears to be acceptable and a full size casting is in progress.

3.2.6 Effluent Disposal System (J. W. George, J. McDonald)

All procurement, fabrication, and assembly activities were completed for the effluent system. Installation of the effluent system components (venturi scrubber, demister, U-bend piping, exhaust piping, and the water

treatment tank) was also accomplished during the period. Interconnecting water piping for the system was installed along with the pH treatment and mixing equipment for the effluent water. A photo of the venturi scrubber and demister unit (during installation) is presented in Figure 3.19.

Shakedown tests of the effluent scrubber system (without pH control) were initiated during the arc heater power tests (Ar-H₂ gas only). System operation was satisfactory. Installation of the pH control system wiring will complete system installation.

3.3 Experiment - Laboratory Integration

3.3.1 Electrical System (W. J. Melilli, P. E. Martin)

The procurement activity for the electrical system was completed during the quarter; while fabrication, assembly, and installation of the system components continued.

During the report period, the dc transfer switch was received and installed. Cable was installed and connected from the dc welders to the switch and from the switch to each set of test cell bus. The dc welders are shown in Figure 3.20. Wiring was completed from the ac auxiliary supply switch to the 2300V/480V transformer. Wiring of the 480 volt ac supply to the motor control center was completed. Likewise, wiring from the control center to the various loads is complete except for connecting the effluent pumps and mixers. A photo of the motor control center is shown in Figure 3.21. All of the sodium drum heater relay panels and drum heater receptacles were installed and wired to the 480 volt ac supply.

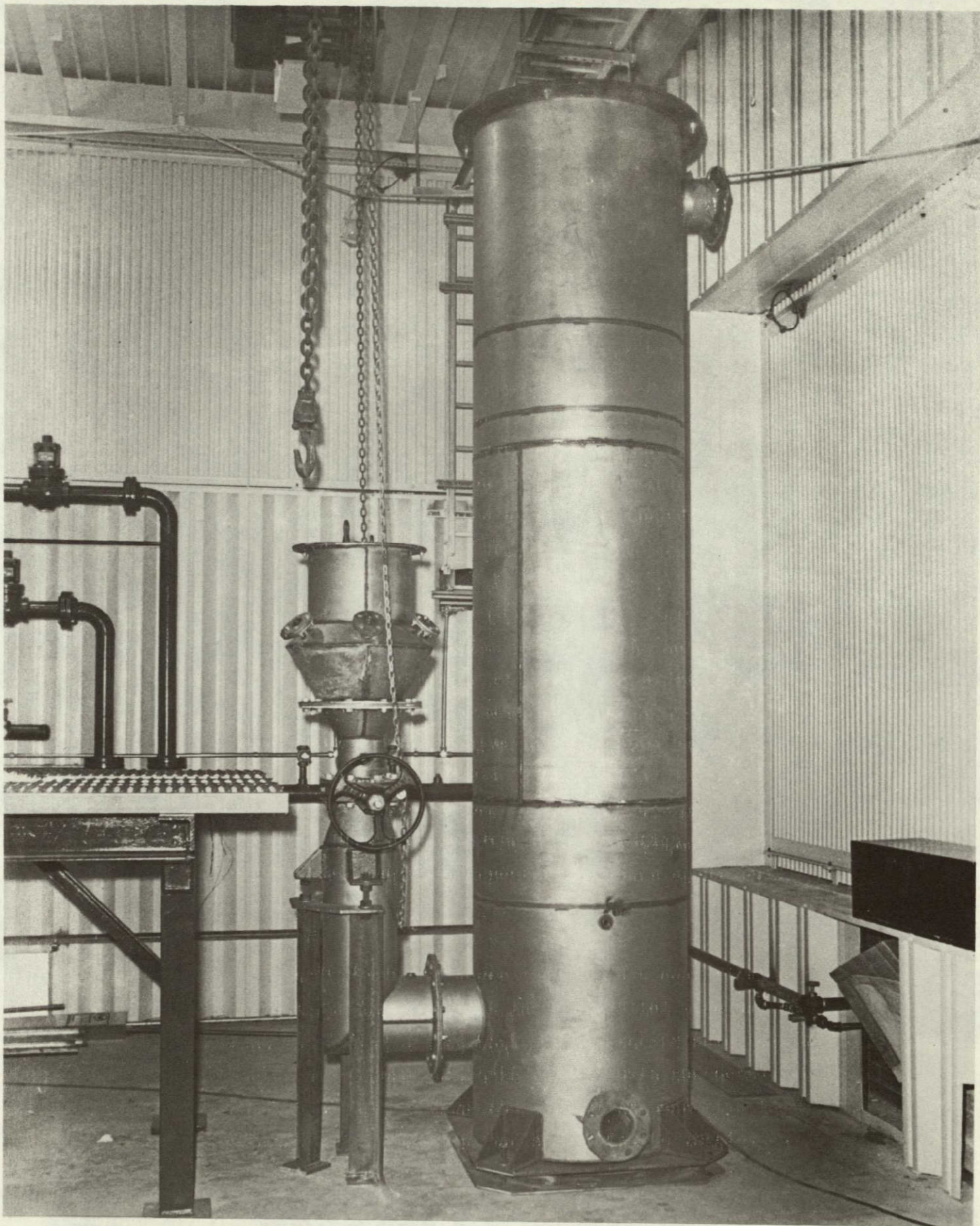


Figure 3.19 - Photograph Of The Effluent Gas Scrubber/Demister Mounted In Place In The Silicon Test Cell



Figure 3.20 - Photograph Of The dc Welder Units Which Supply Arc Heater Field Coil Power

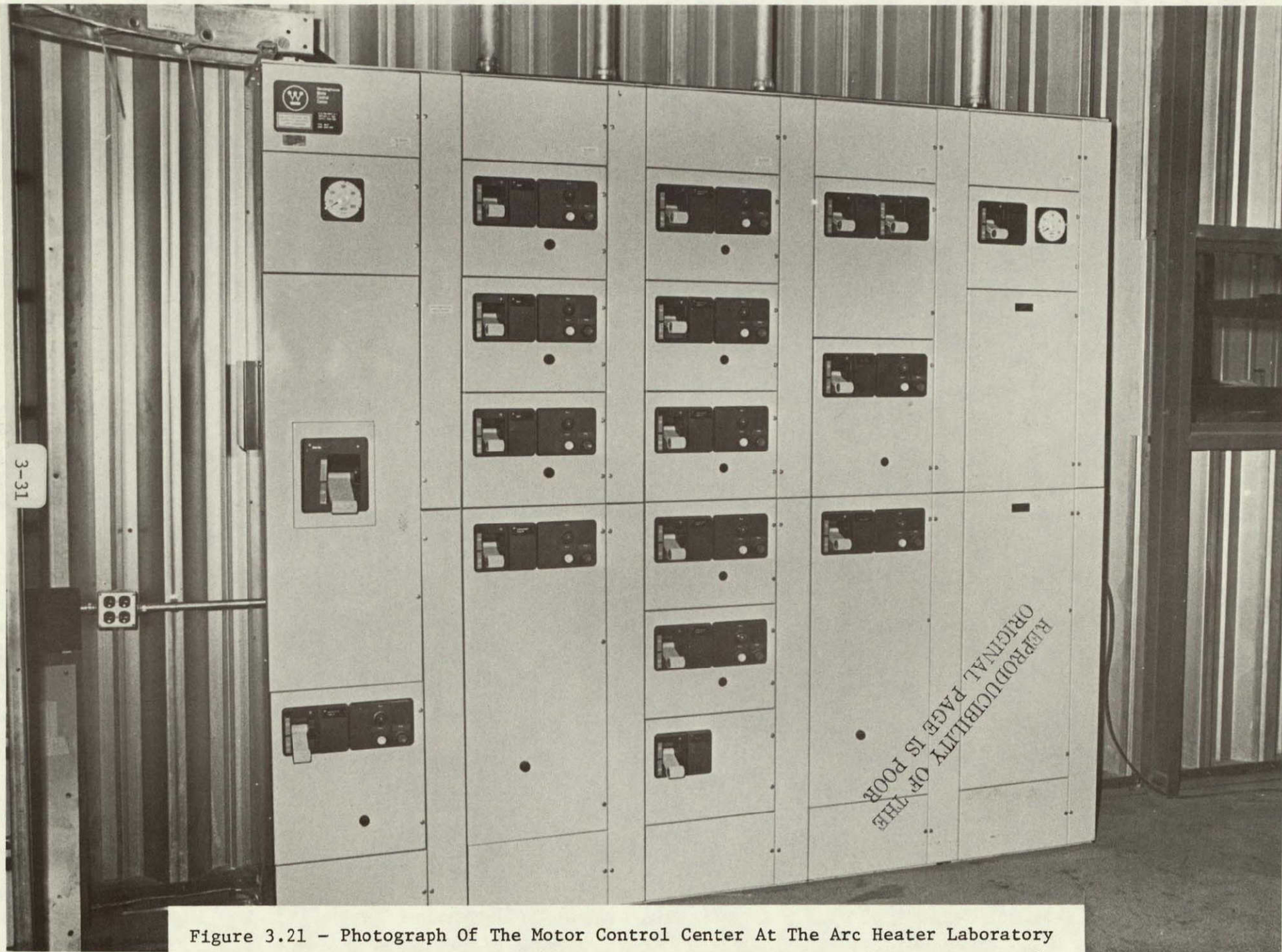


Figure 3.21 - Photograph Of The Motor Control Center At The Arc Heater Laboratory

The trace heater breaker panel and relay panel were wired to the 240 volt ac supply.

All cable tray work was completed. The enclosed cable tray used for supporting the ac power cable in the silicon test cell was installed. During system operation, positive ventilation is provided by a blower at one end of the ac duct work. Air is blown into the test cell to avoid potential accumulation of hydrogen within the duct. Potential transformers and current transformer installation and wiring were completed at the ac transfer switch. Installation of the ac, dc, and electrical grounding cables from the overhead bus to the arc heaters was completed.

Several of the completed electrical systems were operated as part of the installation and shakedown procedures. These included operating the cooling water pumps and verifying correct rotation, operating the SiCl_4 scrubber and the laboratory heating, cooling and ventilation systems. Electrical system readiness was accomplished prior to testing the arc heaters during the gas only-shakedown tests.

3.3.2 Gas System (C. B. Wolf, W. J. Melilli)

Layout drawings for installation of the Ar-H_2 gas system piping were completed. These drawings define the piping arrangement from the gas storage tanks to the various subsystem areas within the test facility. Figure 3.22 presents the general layout drawing for the gas system piping.

Procurement and fabrication activities were completed

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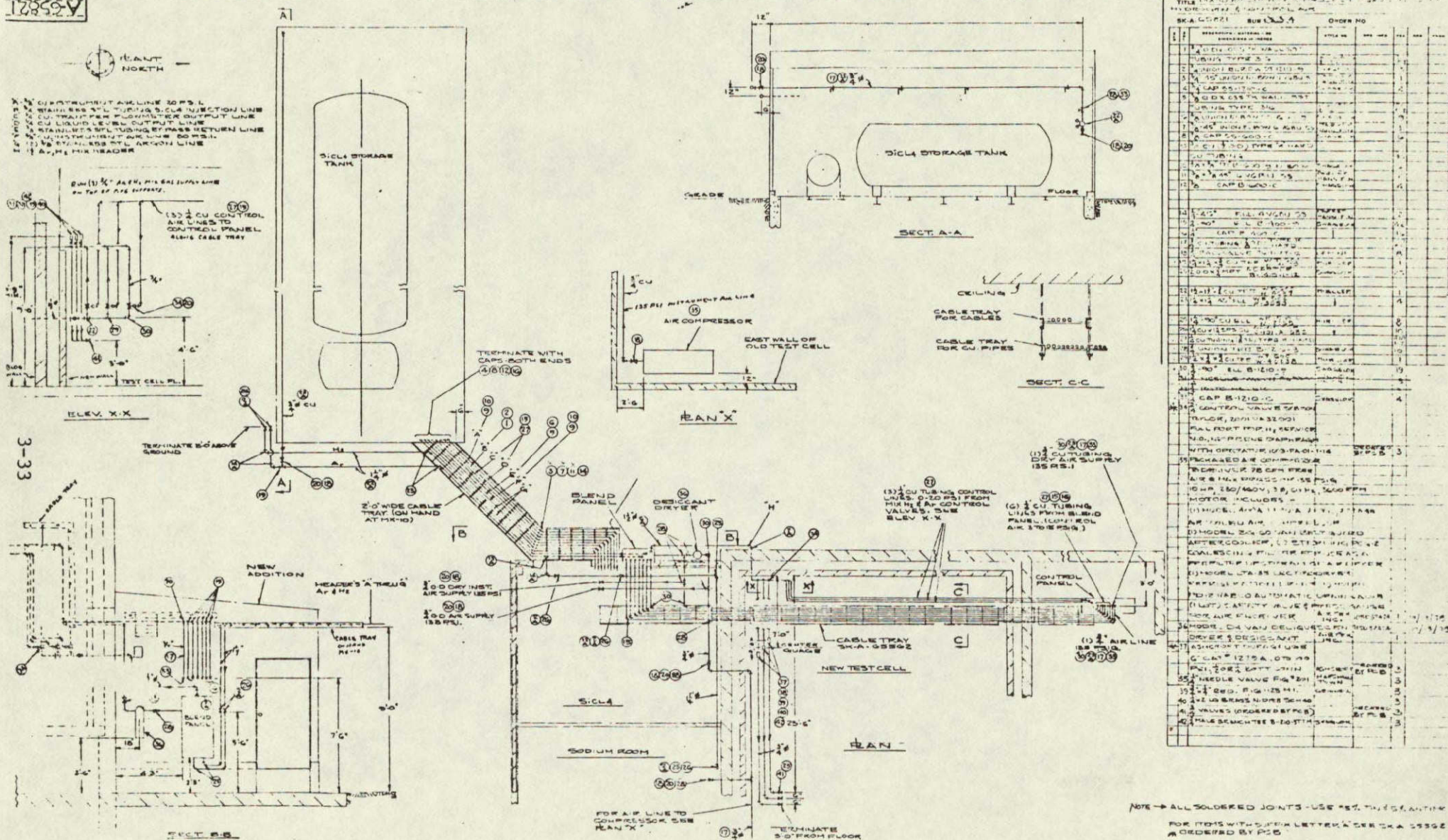


Figure 3.22 - Layout Drawing Of The Gas Piping System

for the gas system. The blend panel which mixes the Ar and H₂ gas for use with the arc heaters was received and installed (see Figure 3.23). Installation of the indoor piping for gas distribution was installed and leak checked. Both the Ar and H₂ storage systems were prepared and installed; Ar is piped to the system from a liquid storage tank, while the H₂ is distributed from a tube trailer unit as shown in Figure 3.24.

Installation of the gas system I&C components is nearing completion along with installation of the pneumatic control tubing and the I&C air compressor.

The gas system was operated and shakedown tests of the system conducted during the initial arc heater power tests (gas only).

3.3.3 Cooling Water System (C. B. Wolf)

The layout drawing for installation of the cooling water piping was completed and is presented in Figure 3.25. The water system contains two cooling loops; a high pressure recycle loop for cooling the arc heater electrodes and a low pressure recycle loop for cooling the reactor sections, cyclone-separator and the silicon collector. The effluent gas cooling water is provided by the cooling water outlet from the heat exchangers.

The component procurement and fabrication efforts for the cooling water system were completed. All of the system components were installed and the water distribution piping completed from the water pumps to the subsystem locations. Figure 3.26 is a photograph of the water pump room components which include the pumps, surge tank, heat exchangers, and demineralizer.

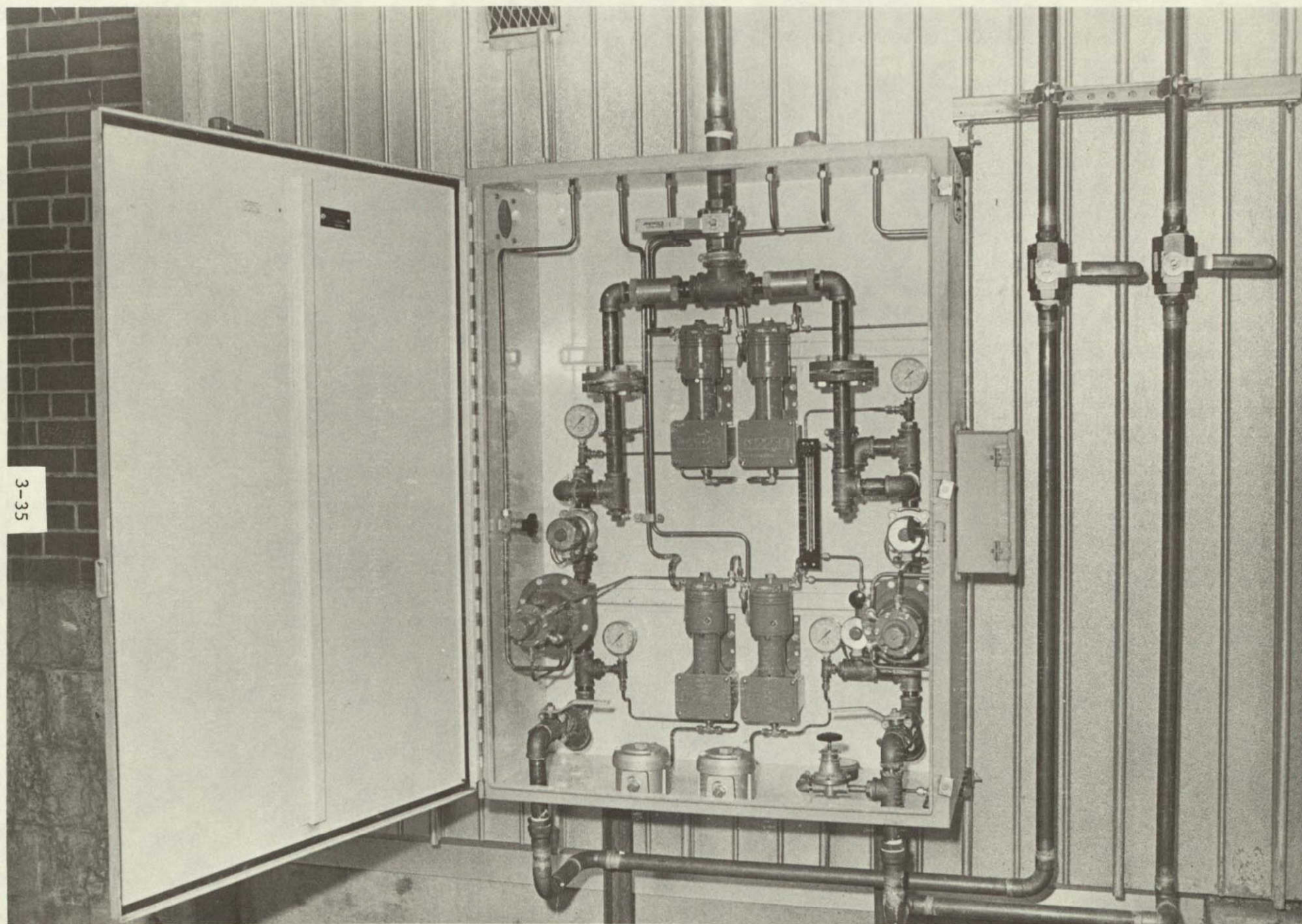


Figure 3.23 - Photograph Of The Ar-H₂ Gas Blend Panel (Mounted Outside At The Arc Heater Laboratory)

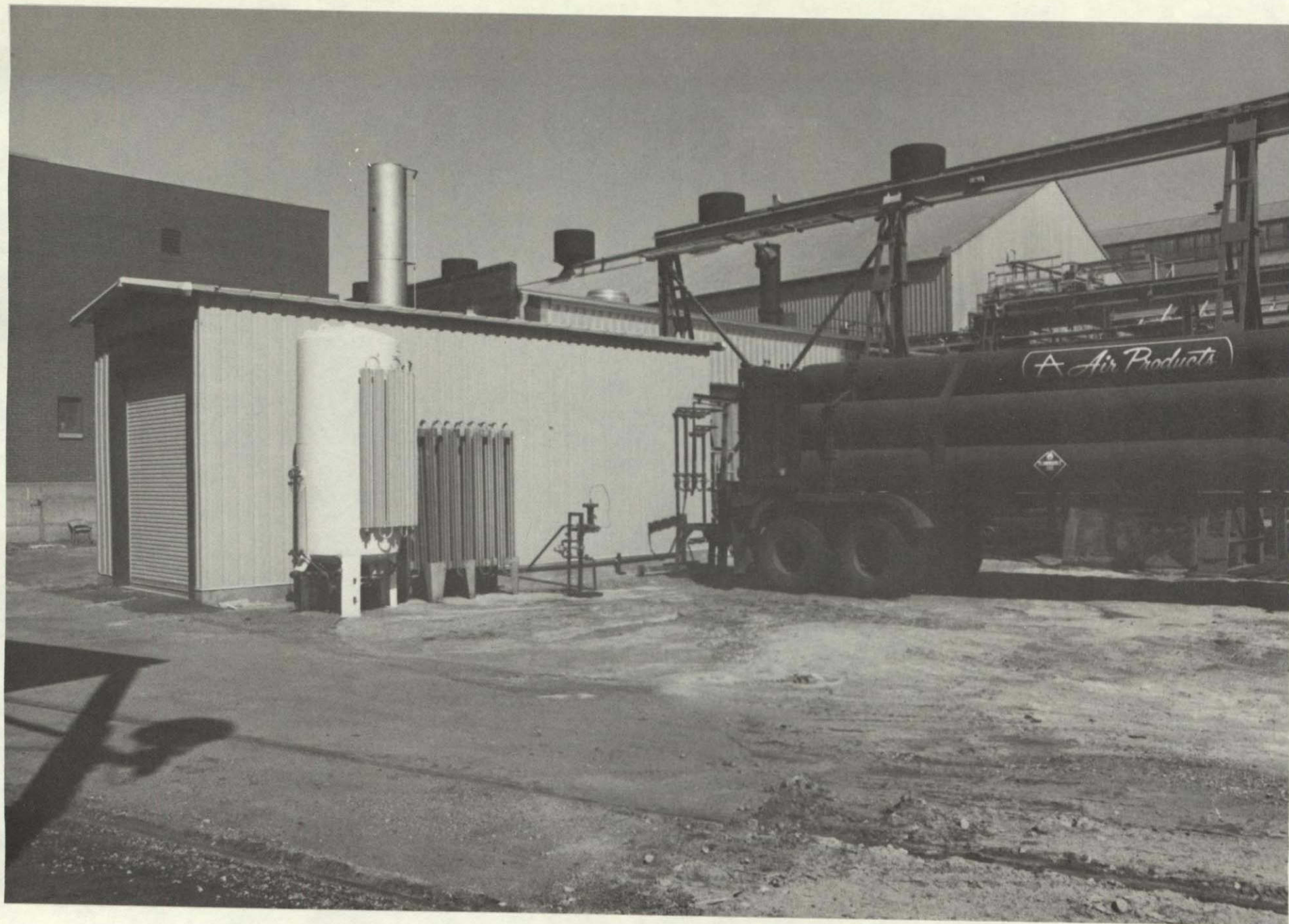


Figure 3.24 - Installation Of The Liquid Ar And The H₂ Tube Trailer Supply Facilities

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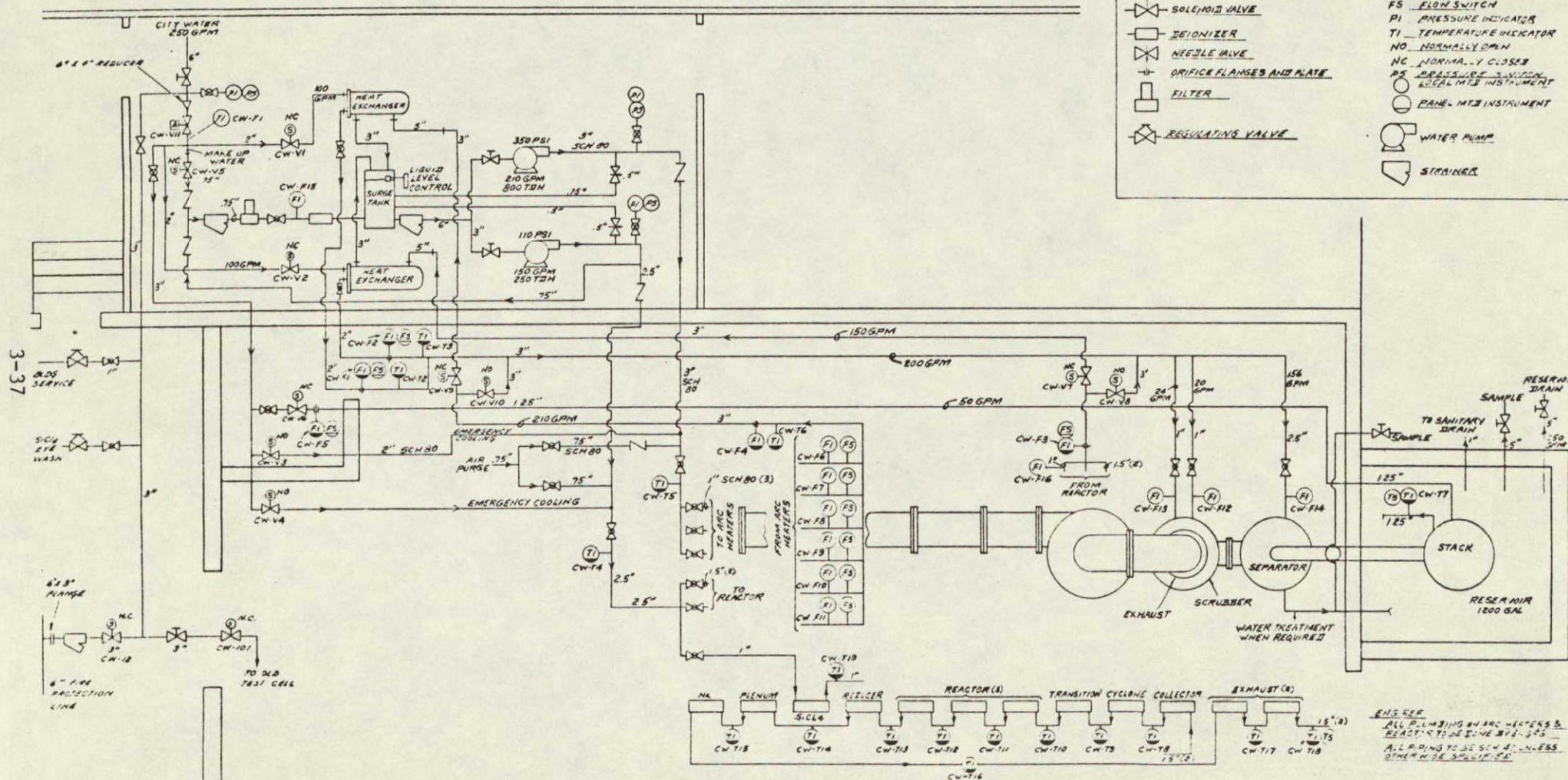


Figure 3.25 - Piping Drawing Of The Cooling Water System

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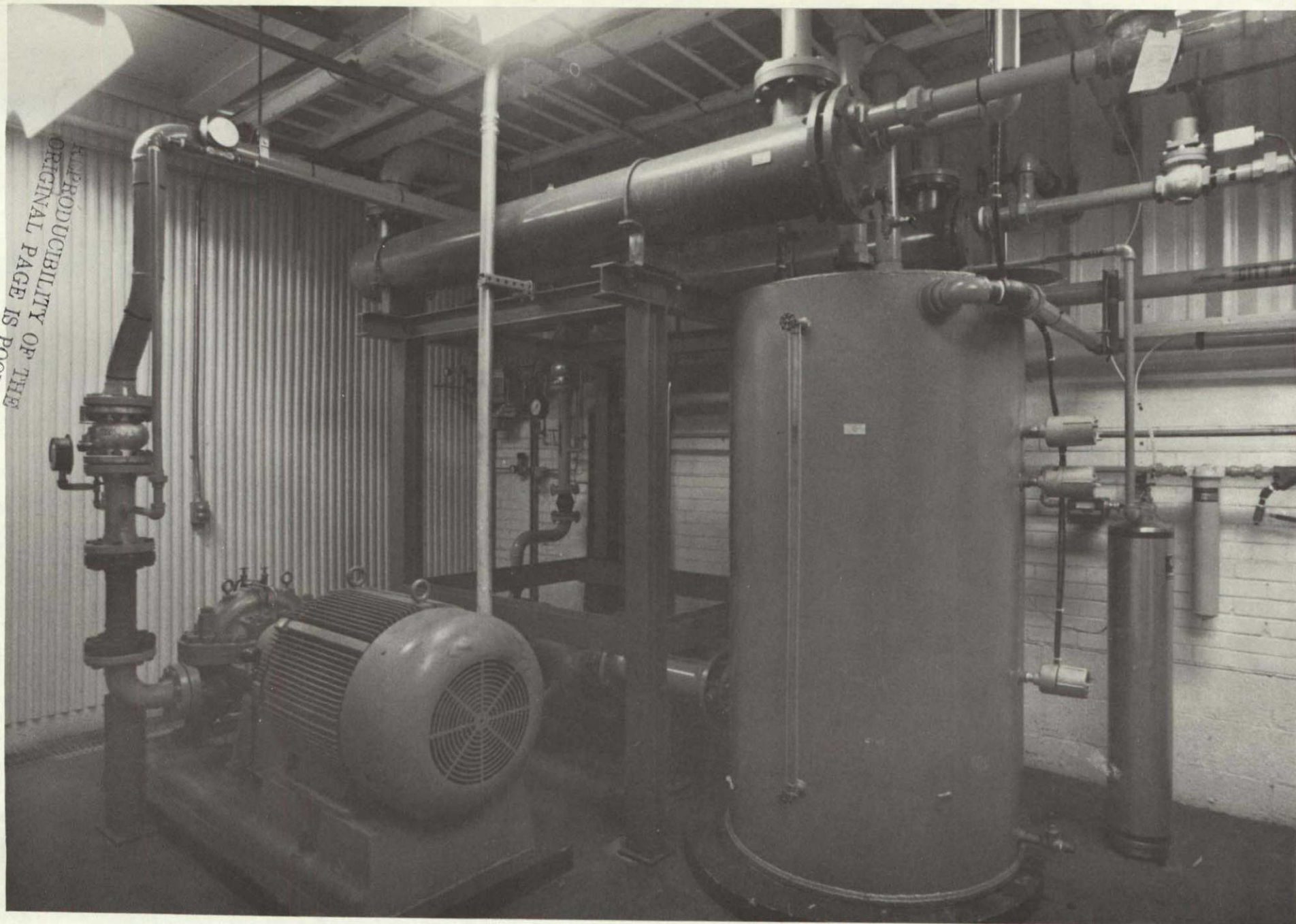


Figure 3.26 - Photograph Of The Cooling Water System Main Components

Power and I&C wiring for the cooling water system were completed and the I&C system monitors installed. Initial shakedown tests were conducted for the system to determine water flows, system operation, and I&C monitors operation. The water system operated successfully during the initial arc heater power tests (gas only).

3.3.4 Gas Burnoff System (J. W. George, C. B. Wolf)

Procurement, fabrication, and installation of the gas burnoff stack (as shown in Figure 3.27) for the silicon test facility were completed. Both the stack cooling water piping and the effluent gas piping to the stack base were installed. A sample port was installed in the effluent piping prior to the stack for system chemistry and environmental sampling. The stack igniter which is interfaced with the I&C system was received and installed. The water treatment tank for effluent neutralization was installed beneath the stack and provides base support for the burnoff stack.

The stack was operated for combustion of H_2 during the initial arc heater power tests using Ar- H_2 gases only.

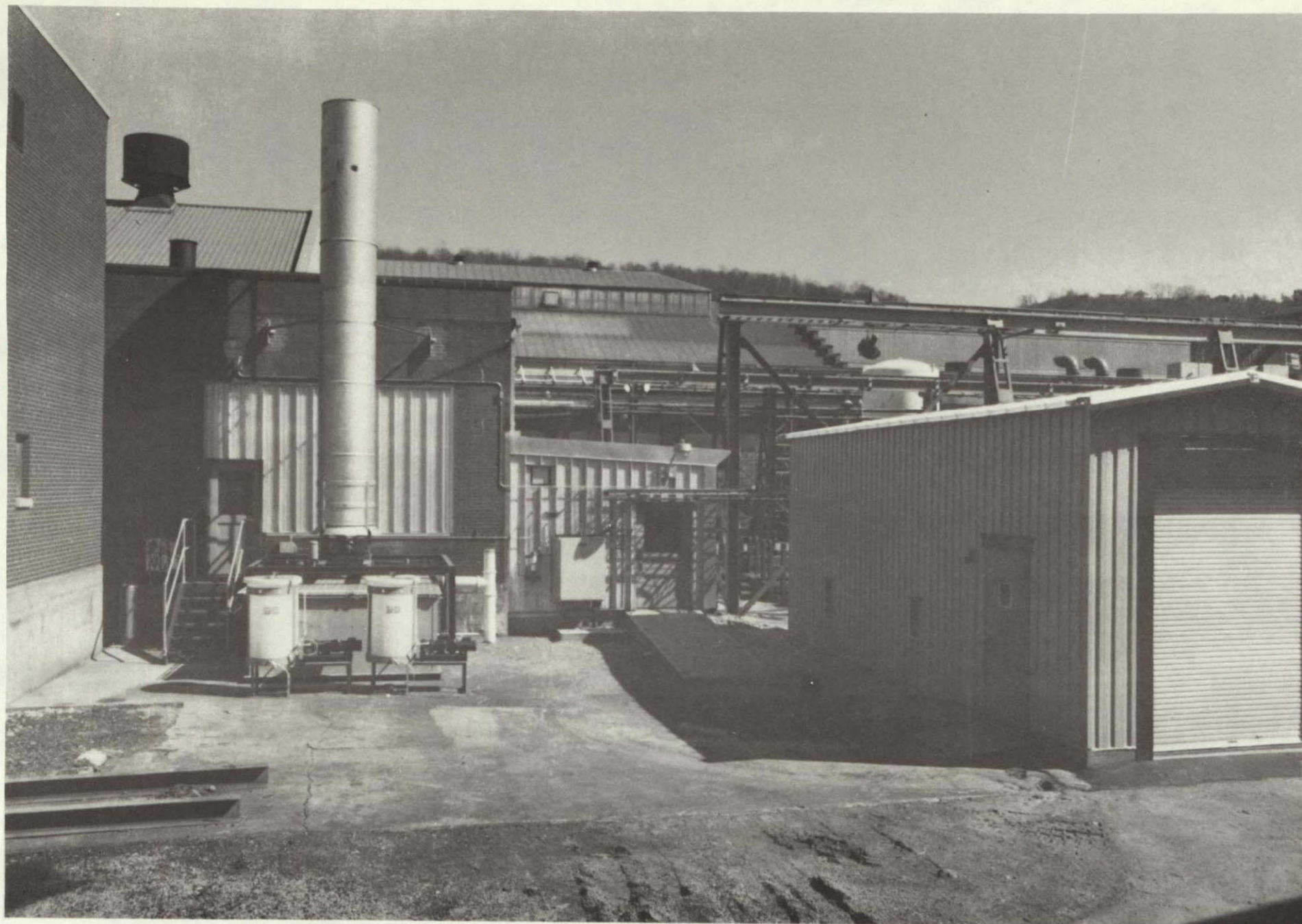


Figure 3.27 - Photograph Of The Gas Burnoff Stack And Effluent Treatment Tank (Installed Beneath Stack)

4. TESTING

Task Investigators: T. N. Meyer, W. H. Reed, P. E. Martin,
J. W. George and J. McDonald

4.1 Procedure Manuals

Preparation of the initial draft of the System Safety Manual was accomplished during the quarter. This draft was distributed to both appropriate Plant Safety and project personnel for comment and review. Revisions to the manual are being incorporated based on the comments received. Following revisions, the manual will be distributed in final form.

During the quarter, two iterations were performed toward finalizing a set of operating procedures. These procedures were written/reviewed by the key project personnel and revised. The second set of procedures contains 63 steps ranging from system start-up to system shutdown. In addition, procedures are being finalized for operation of the sodium and silicon tetrachloride subsystems. These steps exclude those associated with the main chemical control panel operation of the same two subsystems. As a result of the first iteration, it was determined that the system coolant water remain on during emergency shutdown. The control circuitry was modified to permit the coolant water (i.e., either water from pumps or emergency water) to remain on during an emergency shutdown. The initial draft of the complete Operations Manual is in progress and copies will be distributed.

4.2 Shakedown Tests

Shakedown tests were performed on the burnoff stack, the cooling water, the electrical power, and the associated control and instrumentation system. The first test operation of the reactor using arc heated argon gas (90 scfm) was conducted at a power level of 110 kW. A reactance of 3.64 Ω was used in

each phase of the grounded neutral three-phase system. Before operating the reactor on arc heated hydrogen gas, the burnoff stack was operated on hydrogen gas without arc heating. Following this checkout of the burnoff stack, a second power run at 700 kW was conducted using a flow mixture containing both hydrogen and argon.

4.3 Data Acquisition and Analysis

The work defining the transfer of data from the magnetic tape unit of the data logger to the computer and establishing requirements for data reduction are essentially complete. The total effort resulting in the computer programs for complete data processing is in progress.

4.4 Product Characterization

A complete family of analytical techniques will be employed to correlate reactant and gas impurities with impurity levels established for the silicon product. Depending on the material and impurity, these techniques will include spark source mass spectroscopy, vacuum distillation followed by X-ray and emission spectroscopy, gas mass spectroscopy, dew point probe, oxygen analyzer, and spectrophotometry.

5. INJECTION TECHNIQUES

Task Investigators: M. G. Down, A. R. Keeton, J. E. Bauerle
and D. F. Ciliberti

5.1 Summary

The experimental stage of the project is complete. A satisfactory rationalization of the laser imaging results for the particle size produced by the Sonicore sodium nozzle has been determined from further computer analysis. These results are compared with the sodium sieving data and give an overall picture of the nozzle characteristics. The silicon tetrachloride nozzle has been tested and high speed photographs indicate that its operation was successful.

A summary report of the entire program is being prepared and decontamination of the test chamber is in progress. Final recommendations for the sodium and silicon tetrachloride reactant systems have been relayed to the personnel responsible.

5.2 Work Accomplished

5.2.1 Particle Size For Sodium Nozzle - Laser Technique

In the previous Quarterly, a discrepancy was noted between the particle size data obtained with the 1-D and 2-D spectrometers at Particle Measuring Systems (P.M.S.). The 1-D data indicated consistently larger mean particle diameters than the 2-D. This apparent anomaly has been shown to be due to a high degree of coincidence error inherent in the less sophisticated 1-D instrument. This effect is due to the very high concentration of small particles which can cause particle events which are actually two or more small particles passing through the sample area simultaneously. The computer analysis of the data from the 2-D spectrometer

includes certain rejection criteria to eliminate such coincidence errors that can occur in the 1-D. These are:

Bulk Area - A rectangle is drawn around the image such that the sides of the rectangle touch the extremities of the image. If the ratio of the particle area (number of shadowed elements in the image) to the area of the rectangle is $< .5$, the particle is rejected.

Light Slice - Two adjacent small particles can be seen by the OAP-1-D as one particle event and sized accordingly. The Light Slice computer algorithm rejects this event because there is a light slice normal to the optical array.

Aspect Ratio - Particles with dimensions normal to the optical array of > 3 times the dimension parallel to the array are rejected. This helps eliminate streakers which may result from impact with part of the probe such as the tips.

Further computer analysis of the 2-D data was performed with these rejection criteria deliberately omitted from the program. This resulted in an increase in the apparent particle sizes to values similar to those reported by the 1-D instrument. This check thereby confirmed that the large 1-D values were caused by the inability of the instrument to include the more rigorous rejection criteria of the 2-D. Since the 1-D data cannot be modified to accept these computer algorithms, they cannot be included in our assessment of the nozzle performance. The 2-D data, however, is considered to be fully representative of the nozzle spray produced.

A detailed study of this 2-D data for the Sonicore nozzle, Figure 5.1, shows several features of interest. Initially, when the nozzle was operating in its normal mode, the mass median drop (D_{GM} in Figure 5.1) size varied between 18 and 68 μm . As expected, however, when the resonator cup was removed from the nozzle, causing it to operate like a more conventional gas atomizer, the median diameter increased to approximately 240 μm . We have noted before that the mean diameter might be expected to decrease with increasing gas to liquid mass ratios. Within the band of results in Figure 5.1, this was generally found to be true, although the relationship was not sufficiently reproducible to enable a precise empirical dependence to be formulated. From this, we can conclude that since the highest gas to liquid ratio in these tests was 0.28, the higher values permitted in the silicon reactor (up to 1.0) should easily produce a mass median drop size < 25 μm .

5.2.2 Particle Size For Sodium Nozzle - Sieving Technique

The results of the sodium particle sieving experiment for the Sonicore nozzle previously described are presented in Table 5.1. These data are shown to obey a log-normal distribution in Figure 5.1 which also includes the 2-D data for the same nozzle. The mass median diameter is 140 μm and the σ_g value is 1.85. Both of these figures reflect a noticeable difference between these results and those obtained by the laser technique. The larger median diameter indicated by the sieving data is not inconsistent with the inherent limitations of the two techniques. In the sieving experiment, the very smallest particles tended to be swept upwards in the injection chamber and were later found to have been trapped on the filter. These particles, of course, were extremely small and probably have little effect on the overall

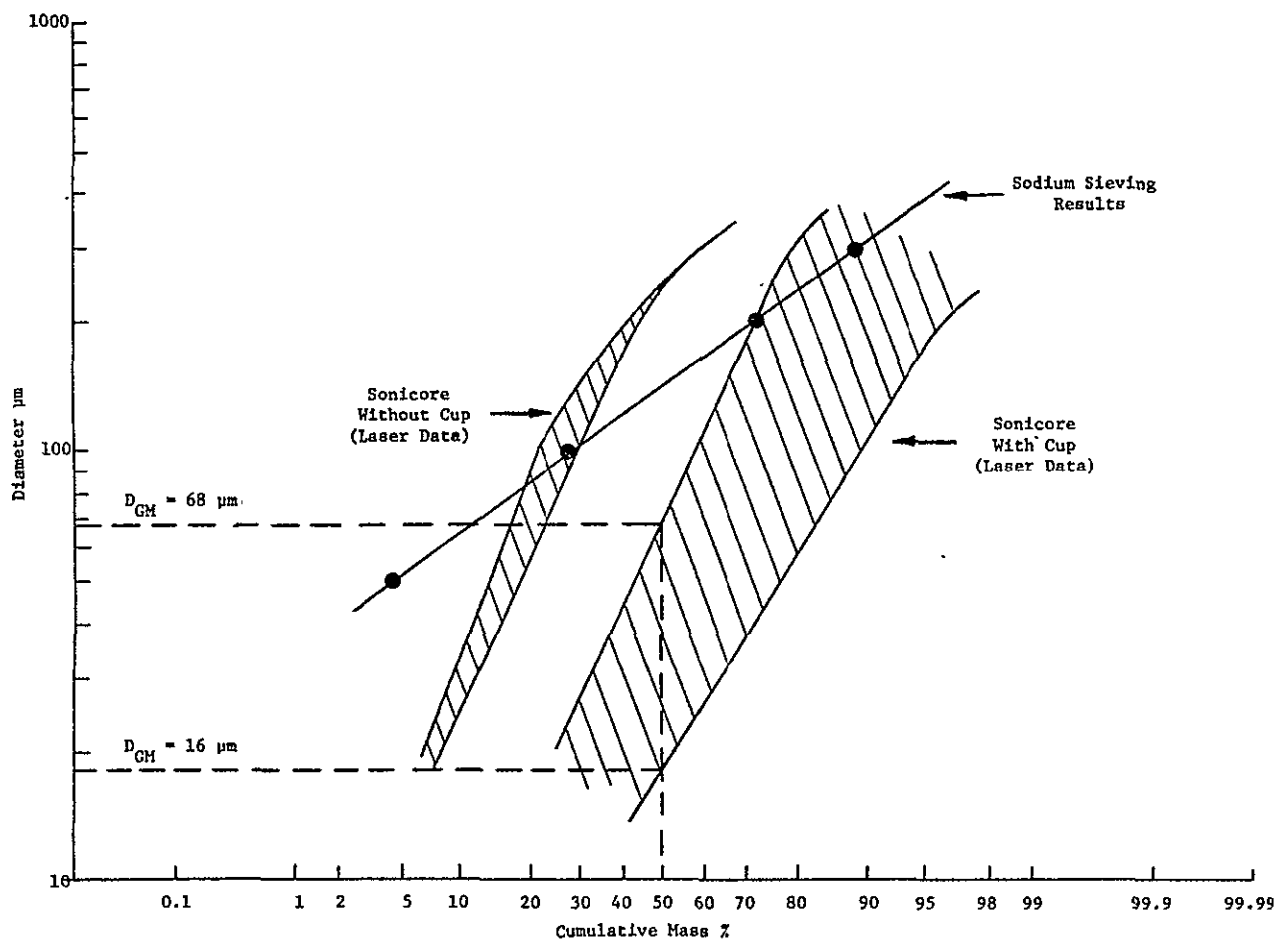


Figure 5.1 - Particle Size Data For Sonicore Nozzle

TABLE 5.1

Particle Size Analysis of Sodium Spray
(Sonicore Nozzle - Model 312T)

<u>Size Range (μ)</u>	<u>Mass of Na (g)</u>	<u>Mass %</u>	<u>Cumulative Mass %</u>
300-400	0.0865	11.24	100.00
200-300	0.1249	16.23	88.76
100-200	0.3434	44.63	72.52
50-100	0.1802	23.42	27.89
<50	0.0344	4.47	4.47

Test Parameters

Sodium Flow 45 GPH
Argon Flow 50 SCFM
Argon Pressure ~40 psi

Gas:Liquid Mass Ratio 0.8

mass distribution. A more serious source of error which might lead to high results, however, is the possibility of larger particles, or even drips, being produced when the spray is first started up or switched off. These larger particles would certainly fall to the bottom of the chamber and be collected. In the laser experiment, however, the spray was only sampled while the nozzle was continually operating in a steady state. Any larger, non-typical particles formed during start-up would not show up in this analysis. Another reason why the laser experiments might miss some larger particles is that the water was sprayed horizontally as opposed to vertically downwards in the sodium freezing and sieving tests. Some heavier particles could, therefore, have been gravitationally separated from the spray before reaching the spectrometer probes.

A photograph of the test system for the sodium nozzle evaluation is presented in Figure 5.2 along with the instrumentation and controls that are depicted in Figure 5.3.

5.2.3 Silicon Tetrachloride Nozzle

The pumped supply system used for testing the silicon tetrachloride injection nozzle is shown schematically in Figure 5.4. The liquid was pumped from a 5 liter steel storage cylinder using a positive displacement pump (Tuthill Pump Co., Chicago, Pump No. OLE) with a maximum capacity of 90 GPH at 100 psi. A flow meter and pressure gauge was incorporated in the line which delivered the silicon tetrachloride into the test chamber nozzle holder.

A series of test injections have been performed in which the liquid was sprayed at approximately 9 GPH at a pressure of 100 psi for periods up to a minute. The spray

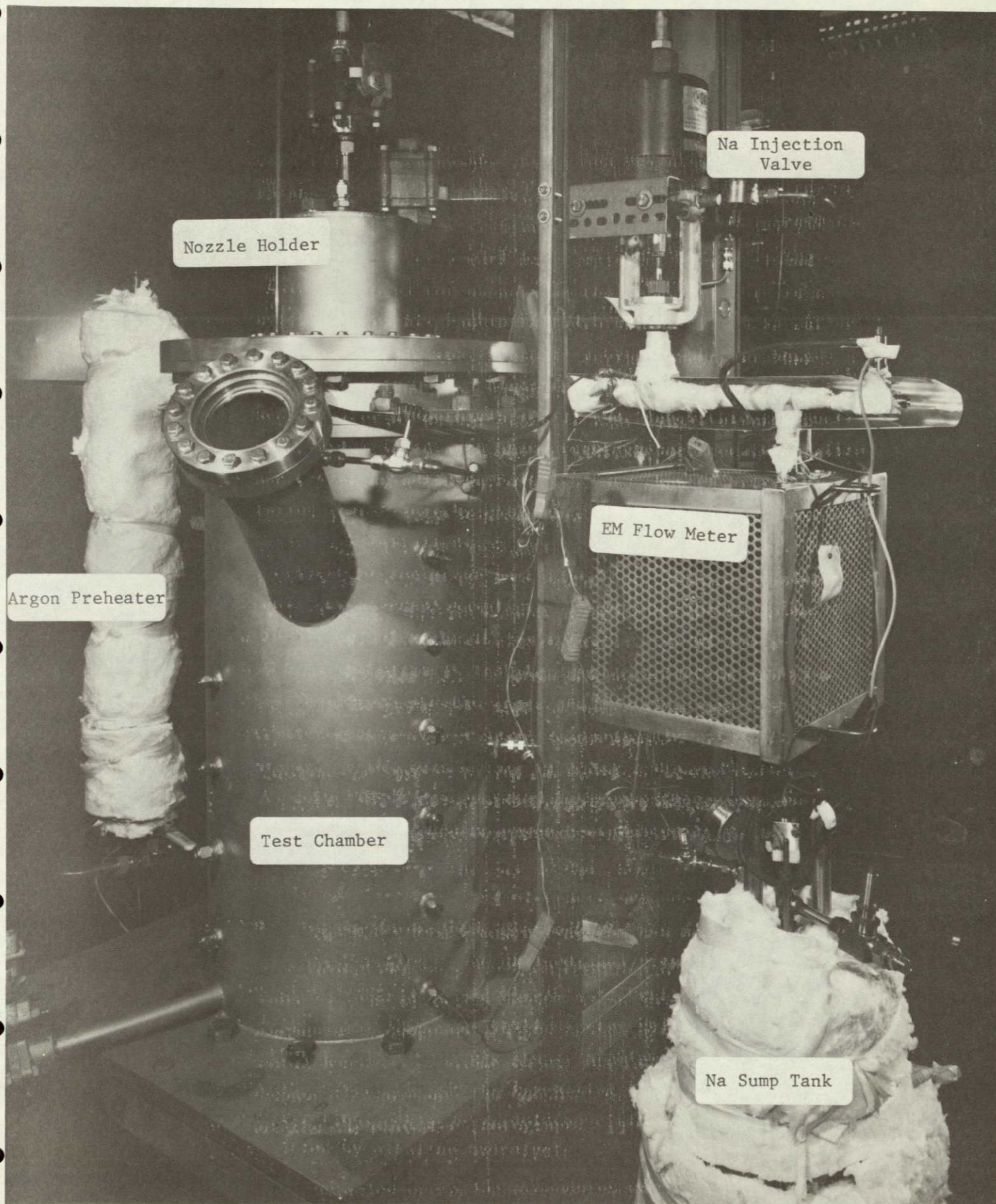


Figure 5.2 - Photograph Of The Test System For Sodium Nozzle Testing

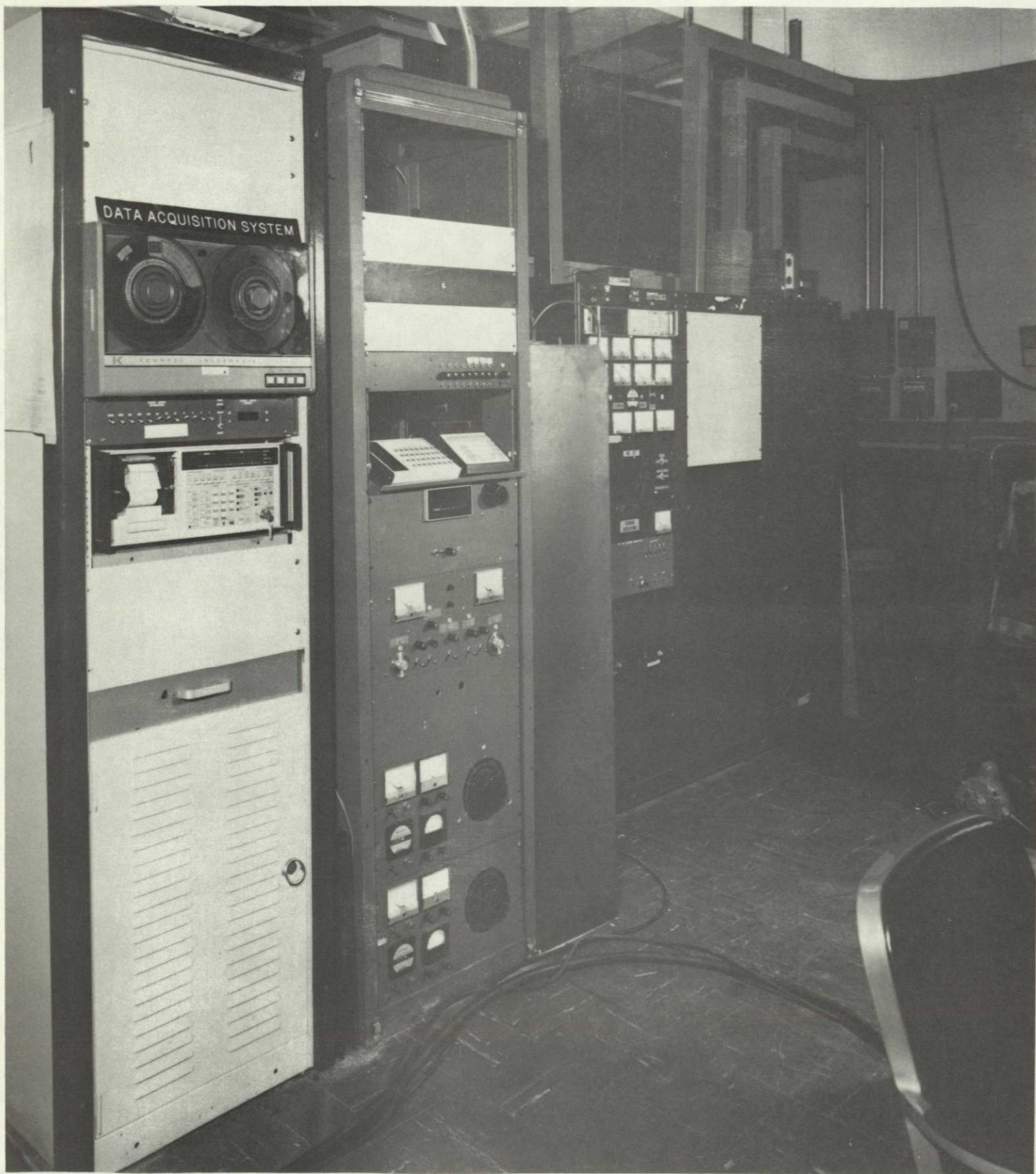


Figure 5.3 - Photograph Of The Instrumentation & Controls For The Injection Techniques Experiments

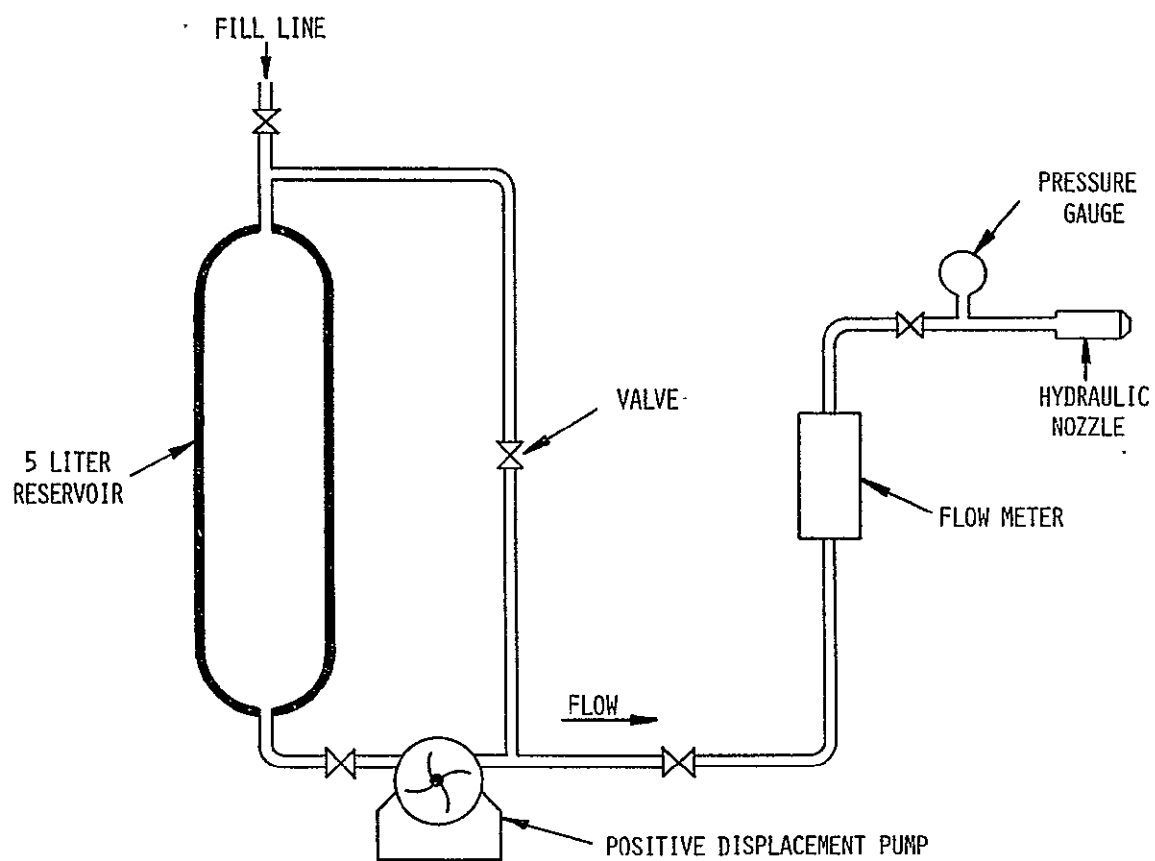


Figure 5.4 - Schematic Of The Silicon Tetrachloride Supply System

was studied visually and appeared to consist of a uniform, fine mist of liquid droplets. High speed movie photographs (1000 frames per second) were taken of the spray and these are being studied in order to determine spray angle, velocity and general uniformity of the atomization process.

6. KINETICS STUDY

Task Investigators: J. V. R. Heberlein, J. F. Lowry and
D. F. Ciliberti

6.1 Summary

The apparatus became fully operational for studying the reduction of SiCl_4 by Na and the collection of the resulting products. Several minor modifications allowed the operating time at nominal power and design flow rates to increase continuous operation to more than 15 minutes. The product was collected on the walls of a SiC tube of 1" I.D., and experiments were completed to determine the amount of silicon collected as a function of axial location in the flow duct. The results show good agreement between experiment and theoretical predictions.

6.2 Work Description

6.2.1 Apparatus Modifications

Several problems with the system operation were solved:

(a) SiCl_4 flow control was impeded because SiCl_4 dissolved a significant quantity of Ar when kept under Ar pressures over 5 psig for more than a day. The solution to this problem was achieved by minimizing the pressurization time of the SiCl_4 supply tank.

(b) Leakage through the pressure relief valves in the SiCl_4 supply system and oxidation of the leaked SiCl_4 caused scrubber clogging. Installation of a trap between the outlet of the relief valves and the vent to atmosphere solved this problem.

(c) The scrubber was modified to remove more of the small particles from the gas stream. These particles were part of the reaction product and caused clogging at the scrubber exit. The size of the flow duct from the scrubber to the burnoff stack was increased, and installation of a different

array of spray nozzles increased the efficiency with which the particles are washed out of the gas stream.

(d) Initially, clogging of the SiCl_4 injection holes limited running time to a few minutes. Operating time was increased to more than 15 minutes by modifying the graphite liner in the injection section, such that the SiCl_4 flow passages leading into the reaction tube in a downstream direction, at an angle of 45° with the tube axis. Thus, the SiCl_4 vapor was injected into the flow downstream of the Na. Additionally, the hydrogen flow was started only after the SiCl_4 flow was established, thus reducing the possibility of hydrogen diffusion into the SiCl_4 flow ducts where it could lead to Si formation and clogging of the ducts.

6.2.2 Experimental Results

Experiments were performed at torch power levels between 16 kW and 30 kW with a hydrogen-argon ratio of 2 and 4. In a few cases, only argon was heated in the torch, and the resulting power level was around 8 kW. Calorimetric measurements of the heat transferred to the torch, the injection section, and the different parts of the test chamber, and pyrometer measurements of the SiC tube wall temperature comprise the diagnostics employed in every run. For the product separation analysis, two different arrangements were used for the vapor flow duct: a SiC tube with 2" I.D., and a SiC tube with 1" I.D. inside a 2" I.D. SiC tube. In the second case, heat losses from the wall were significantly reduced and a long portion of the tube had a wall temperature above the melting point of silicon. Typical results are given in Table 6.1. The wall temperature results can be considered to represent a lower limit, since the measured radiation from the tube wall was not corrected for the attenuation by clouds of particle dust which formed part of the reaction products. Based upon the calorimetric

measurements and knowledge of the mass flow and energy inputs, the bulk stream enthalpy is determined. For the relative elemental inputs used, an equilibrium calculation determines the dependence of temperature on enthalpy. Table 6.1 lists the bulk temperatures resulting from these measurements and calculations.

Table 6.1: Typical tube wall temperatures and bulk gas temperatures for runs at 26 kW torch power with a 1" I.D. SiC tube

<u>Axial Location</u>	<u>Distance from injection nozzle</u>	<u>T_{bulk} °K</u>	<u>T_{wall} °K</u>
torch exit		3670	
injection nozzle exit	0	3100	
1st window	5 cm		~1900
2nd window	16 cm		~1650
end of test section I	25 cm	2820	

The increasing temperature difference between bulk gas temperature and tube wall temperature results from the low thermal transport in the tube due to the existence of laminar flow conditions.

During one experiment, the particle probe was inserted into the flow stream and gas was drawn through the filter for one minute. After this time, the filter was clogged.

The reaction products are routinely analyzed by Electron Dispersive Analysis of X-rays (EDAX) which gives the elemental composition, and occasionally by x-ray diffraction, to find the crystalline components. The SiC tube was cut into different sections, while weighing these sections before and

after each run determined the total amount of product collected in each. This product was then separated into water soluble and insoluble substances.

The products were collected in the following forms:

(a) In the regions where the SiC tube wall temperature was above the melting point of silicon and the dew point of NaCl, the wall was covered with silicon. During cooling after the experiment, this silicon formed mounds around the circumference and frozen pools at the bottom of the SiC tube.

(b) In the regions where the tube wall temperature was slightly below the melting point of silicon, the inside wall was covered with a gray scale which could be easily scraped off in the form of large flakes. These flakes consisted of crystalline silicon and varying amounts of NaCl, ranging from trace values (1 to 2%) in the flakes from tube sections with a wall temperature only slightly below the dew point of NaCl to more than 50% farther downstream.

(c) The end sections of the SiC tube were covered with a glassy layer of NaCl, containing a trace of silicon. This glassy substance fused tube sections together and formed crystals in the form of icicles at the tube end.

(d) The rest of the product in the gas stream formed brown particles upon mixing with cold gas. These particles ranged in size from 3 μm to 70 μm and consisted of a mixture of amorphous silicon and NaCl. These were collected in the test chamber, in the trap between test chamber and scrubber, in the scrubbing water and at the exit of the burnoff stack.

(e) In the experiments without Na injection, amorphous silicon was found on the SiC tube walls in the form of a dark brown powder, consisting of submicron size particles, in addition to crystalline silicon condensed on the walls of

the injection flange.

In the experiments where a substantial portion of the SiC tube had a wall temperature above the melting point of silicon, more than 25% of the amount of silicon injected in the form of SiCl_4 was collected on the tube wall as polycrystalline silicon, distributed over a length of 30 cm, corresponding to an X/D ratio of 12. The experimental results agree with theoretical predictions obtained from a heat and mass transfer analysis, which was originally formulated for turbulent flow and has been adapted for the laminar flow conditions prevailing in this experiment.

7. REACTION DEMONSTRATION

Task Investigators: A. Y. Ranadive and D. F. Ciliberti

7.1 Summary

Full scale testing has been initiated. Repeated clogging of the liquid sodium metering valve orifice has led to the abandonment of this method for monitoring the sodium flow rate. Another method which makes use of the vapor pressure-temperature characteristics of liquid sodium has been pursued, and testing at lower tube wall temperatures (1300°C) was initiated. Test products have been analyzed as being elemental silicon and sodium chloride.

7.2 Work Description

Full scale testing was initiated in early October. Problems with sodium injection repeatedly arose during the initial tests. The first test carried out for a duration of fifteen minutes did not result in the expected deposit of reaction products onto the cold finger condenser. Post test inspection of the apparatus revealed that the sodium flow has stopped just short of entering the vaporizer. This long time was due to the relatively large volume in the two valve bellows of the liquid sodium line. In the next test, therefore, a device was incorporated in the sodium line upstream of the vaporizer and downstream of both valves to monitor the presence of sodium. Briefly, the device consisted of two coils each comprised of one primary and one secondary winding with both primaries connected in series and likewise both secondaries also connected in series; a nulling circuit, a signal generator and an oscilloscope completed the circuit. The presence of sodium at the coils modifies the wave form displayed on the oscilloscope. However, a very large time lag occurred between inversions of the wave form which indicates

a declining sodium flow. This flow problem was confirmed in the post test analysis which did not indicate the presence of sodium in either the reaction tube, the exhaust gas scrubber or on the cold finger condenser. Evidently, the extremely small orifice of the sodium metering valve was being plugged due to the plating out of sodium impurities (oxides).

This method of sodium injection was modified, and in subsequent tests sodium was injected via a precharged sodium vaporizer with an Ar/H₂ sweep gas. Vapor pressure versus temperature data was used to control the concentration of the sodium vapor with the flow rate of Ar/H₂ gas effecting the Na flow rate.

Six full scale tests based on the modified design of sodium injection were attempted. A persistent problem that arose at this time was the sporadic breakage of the alumina reaction tubes. During these tests, the tube wall temperature was in the range of 1450-1550°C, and the failure of the alumina tubes was attributed to their poor thermal shock resistance. Due to its superior resistance to thermal shock, a mullite reaction tube was used for the fourth test. A most dramatic failure of this tube occurred. The breakage of the inner sodium conveying tube splattered hot (300°C) sodium aerosol onto the walls of the mullite tube, and the subsequent destructive reaction between the sodium and the glass in the mullite caused virtual disintegration of the mullite tube. Despite the inadequate thermal shock resistance, high quality alumina tubes (e.g., McDanel's 998 and Coors AD 998) are more suitable candidates and a decision was made to continue using these tubes, but employing lower tube wall temperatures to prevent tube breakage.

In the sixth test, a lower tube wall temperature was used (1300°C). While tube failure was prevented, 10 minutes after the run was commenced a greatly increased bubbling and turbulence

was detected in the effluent gas scrubber and this was due to failure of the cold finger condenser. On reducing the nitrogen flow, for cooling the cold finger, the intense bubbling in the scrubber ceased.

Figure 7.1 shows photographs of the broken cold finger condenser. Three characteristic condenser deposits were identified. Closest to the end plate was a 2.5 cms long region composed of a fine yellow-brown powder. Adjoining this region was a 3 cms long more flaky deposit. Closest to the reaction zone was a silver-grey metallic coating approximately 6 cms in length. Samples of these deposits have been analyzed by EDAX and have shown the expected peaks for silicon, sodium, and chlorine. The EDAX method has indicated mass ratios of these elements in all the deposits is approximately stoichiometric. The agreement is particularly good for the silver-grey metallic coating. Since the excess silicon tetrachloride could not have condensed out at the prevalent high reaction tube temperature and was, therefore, swept out with the Ar purge, the formation of elemental silicon and sodium chloride is clearly suggested by the quantitative results.



Figure 7.1 - Photographs Of The Cold Finger Condenser Showing
Deposition Of The Reaction Products

8. CONCLUSIONS

- Based upon the data generated during the sodium nozzle testing, the Sonicore nozzle will produce an acceptable size of sodium droplets for reactor injection using a gas to liquid mass ratio of 0.8.
- Preliminary results of the SiCl_4 nozzle characterization tests indicate satisfactory performance consisting of a uniform, fine mist of liquid droplets.
- Results of the laboratory scale Kinetics experiments have shown
1) the high temperature reaction produces silicon and NaCl which are separable by the condensation mechanism and 2) good agreement was achieved between experimental data and theoretical predictions of a heat and mass transfer model.

9. PROJECT STATUS

9.1 Present Status

During the report period, the procurement activity was completed for the experimental verification test unit. In addition, the fabrication effort for the test equipment was essentially completed. The installation of the test equipment was initiated and continued during the reporting period. Initial shakedown tests of the subsystems were initiated with testing of the arc heater/reactor performed. The Safety Manual for the project was completed and is planned for distribution and comment. Also, activity continued on the preparation of the System Operation Manual.

Test activity was completed on the three research support programs (i.e., Kinetics, Reaction Demonstration, and Injection Techniques). Reaction tests for both the Kinetics and Reaction Demonstration Tasks were performed, and both activities produced silicon from the high temperature, SiCl_4 -Na reaction. The Injection Techniques study completed the sodium injection tests and the corresponding nozzle-particle analysis. In addition, the SiCl_4 injection-spray tests were completed. A summary report on the injection tests and recommendations for the test unit is currently in progress.

Two technical papers were prepared for the 4th International Symposium on Plasma Chemistry which will be held in Zurich, Switzerland in August, 1979. One paper presents the work on the Kinetics Task, while the other paper describes the work on the experimental verification of the silicon production process.

9.2 Future Activity

- Complete installation of the test equipment for the experimental verification unit.

- Continue shakedown tests of the experimental verification unit and subsystems.
- Initiate reactant flow tests for the production of silicon.
- Complete the summary report on the Injection Techniques study.

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APPENDIX I

OPERATING PROCEDURES FOR THE SODIUM SUPPLY SYSTEM FOR SILICON PRODUCTION

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OPERATING PROCEDURES FOR THE SODIUM SUPPLY SYSTEM
FOR SILICON PRODUCTION

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1.0 SAFETY PRECAUTIONS AND PRACTICES

1.1 General Safety Practices

All personnel that will work around, operate, maintain or disassemble the sodium system should read the Manufacturing Chemists Association (MCA) "Chemical Safety Data Sheet SD-47" and the Westinghouse "Safe Practice Data Sheet S-13."

The most important thing to remember about sodium or NaK is that a violent reaction occurs when it comes in contact with water. Therefore, keep water away from any exposed sodium or sodium containers. Do not pick up or handle sodium or sodium contaminated equipment. The sodium will react with the moisture on your skin and cause chemical and thermal burns.

The minimum safety equipment required around the sodium and NaK systems is eye protection. When these systems are at elevated temperatures, under pressure, or being worked on, flame-proof coveralls, leather gloves and face shield must also be worn.

If sodium or NaK is involved in a fire, only one type of fire extinguisher can be used. This is the "Metal-X" (Ansul) Type Fire Extinguisher for class "D" fires. These are yellow in color. DO NOT USE WATER, CO₂, OR OTHER CHEMICAL AGENTS ON A SODIUM OR NaK FIRE. Do not attempt to clean up the residue from these fires. Contact a member of the Liquid Metal Laboratory at the R&D Center if there is any question or problem with these systems.

1.2 Opening Sodium or NaK (Alkali Metal) System for Maintenance or Normal Operation

When a sodium drum is replaced on the sodium system, when the sodium injection nozzle is changed or when there is a problem such as a sodium leak, sodium contaminated equipment will be opened to the atmosphere. The following safety procedures must be followed:

1.2.1 There must always be at least two people involved in the operation (one could be an observer).

1.2.2 Safety clothing including flame-proof coveralls or leather coat, leather gloves and face shield must be worn.

1.2.3 The work area must be dry with no flammable material in the immediate area.

1.2.4 "Metal-X" powder in a suitable container and a Class "D" fire extinguisher must be located nearby for easy access.

1.2.5 The alkali metal component or piping section to be opened should be drained of all metal if possible and purged with inert gas.

1.2.6 A metal catch pan should be placed under the area where the system is to be opened.

1.2.7 Inert gas pressure should be reduced to 0.5 to 1 psig before opening system and a slight purge flow maintained when system is opened.

1.2.8 A pipe cap, temporary flange or plug should be ready to insert into the system opening to minimize exposure to the air.

1.3 Procedure in Case of a Small Sodium Leak

If a small sodium leak occurs during a silicon production test, proceed as follows:

1.3.1 Dress in full safety clothing.

1.3.2 Contain the leak by placing a metal drip pan underneath. If there is danger of the leaking sodium contacting a live electrical circuit, de-energize the circuit.

1.3.3 If the leak is at a mechanical fitting, try tightening the fitting to stop the leak.

1.3.4 If the leaking sodium starts burning, cover it with Metal-X powder.

1.3.5 When the test run is completed, or if a leak develops any other time than during a silicon production run, shut down the system and repair the leak.

1.4 Procedure in Case of a Large Sodium Leak

If a large liquid metal leak (described as a leak of one liter or more of liquid metal in a few minutes) occurs, proceed as follows:

1.4.1 Energize the emergency shutdown circuit.

1.4.2 Dress in full safety clothing.

1.4.3 Try to stop the leak by isolating the leaking section by closing valves.

1.4.4 If the leak cannot be isolated, such as in the bottom of a sodium drum, reduce the gas overpressure as low as possible.

1.4.5 Stand by with Metal-X fire extinguisher to put out any fire started by the sodium.

1.4.6 Do not attempt to clean up the leaking sodium until it has stopped leaking and solidified.

1.5 Sodium or NaK Fire Fighting Procedure

DO NOT USE water, CO₂, or any other fire extinguisher except Metal-X (Ansul) on alkali metal fires. Metal-X fire extinguishers are painted yellow and are rated for Class "D" fires.

1.5.1 Obtain a face shield and wear at all times when around spilled or burning alkali metal. NOTE -- This is the minimum safety equipment to be worn. If time permits or if hazard appears too great, wear full

safety equipment to cover entire body. A self-contained breathing unit is located in the sodium room area and should be worn if smoke is dense.

1.5.2 Turn on emergency air ventilation fan.

1.5.3 Put out fire by directing stream of Metal-X over burning alkali metal, covering with powder until fire is smothered.

1.5.4 Allow extinguished fire to cool before attempting clean-up and decontamination.

1.5.5 If fire is not quickly controlled, call Plant Fire Department for assistance, informing them that alkali metals are involved and not to use water.

1.6 First-Aid for Personnel that have been Contacted by Sodium or NaK Metal

1.6.1 Scrape or brush off excess alkali metal from body. (Remove clothing if covered with alkali metal.)

1.6.2 Flood area with water (use safety shower if necessary).

1.6.3 Call Medical (X7700) and state that medical assistance is required for an alkali metal burn.

2.0 PREHEAT

2.1 Preparing for Drum Heat-Up

Before heat is applied to any sodium drum, the following must be accomplished:

2.1.1 Install argon fitting and pressure relief valve (R-1.....R-5, Fig. 1) in the top of the drum and the drain valve/filter assembly (Na-1.....Na-5, Fig. 1) in the bottom.

2.1.2 Install the heating jacket around the drum and position the drum on the drain stand above the base tank.

2.1.3 Connect the argon fitting at the top of the drum to the argon supply header and apply 3.0 ± 0.5 in. H_2O by opening valve A-1.....A-5, Fig. 1.

2.1.4 Connect the drain valve assembly (Na-1.....Na-5, Fig. 1) to the base tank line while purging with argon. NOTE -- Soap check connection to insure leak tightness.

2.1.5 Plug in the instrumentation and power plugs into the stand receptacles.

2.2 Liquefaction of Sodium

The heating jacket for each sodium drum contains three zones of 10 KW each. Each of these zones must be energized sequentially so that heat is applied at the top first where the sodium/gas interface is located. After a time delay, the middle zone will be energized then. After another time delay, the bottom zone will be turned on. Once the sodium is liquid, the top two zones will be de-energized and the bottom zone will cycle on and off to maintain the temperature. This sequence is necessary in order to liquefy the sodium as quickly as possible without bursting or overheating the drum.

Each heating zone contains an RTD element for primary temperature control and a bimetallic thermal switch for back up overtemperature control. The temperature indicated by the RTD can be read at the local sodium panel, but the bimetallic thermal switch is preset and operates only if an overtemperature occurs. The sequencing temperature control and timing logic for the drum heaters is under software control of the programmable controller.

2.2.1 Check the temperature indicated by the RTD in each heater zone to assure proper indication.

2.2.2 Start heating sequence by turning the heater control to automatic and activating start circuit on programmable controller.

2.2.3 Check drum heating progress to assure normal operation.

2.3 Preheating Sodium System

All piping sections and components of the sodium system must be maintained above 100°C so that the sodium will remain liquid. On the other hand, excessive temperatures could cause undesirable materials to go into solution in the sodium and end up in the silicon product. Therefore, the entire sodium system will be operated between 120°C and 200°C with a nominal temperature of 150°C.

2.3.1 Energize sodium system trace heaters by placing the selector switch in automatic and energizing the programmable controller automatic start circuit. Temperature is controlled by the programmable controller turning each circuit on and off at a preselected rate. Monitor the temperature of each zone and adjust the time on so that the temperature stabilizes about 150°C. Once each trace heater circuit is set, it should not be necessary to adjust it again.

2.3.2 Energize base tank temperature controller and set for 150°C. Base tank temperature is indicated by the base tank temperature indicator controller (Na-TI1) and by two channels on local panel instrument (Na-TI3). Check that base tank temperature stabilizes at 150°C.

2.3.3 Before sodium is initially admitted to the base tank or sodium piping system, the air must be removed and the inner surfaces desorbed of moisture. This can be accomplished as follows:

1. Close the following valves (Fig. 1): Na-6, -7, -8, -15 and A-11, -8 and -9.
2. Open the following valves (Fig. 1): Na-9, -10, -11, -12, -13 and -16.
3. Attach a vacuum pump to valve A-12 and open A-12 and -13.
4. Evacuate sodium system to < 100 microns while preheating entire system.

5. Close valves A-12 and -13 and open valve A-11 (Fig. 1) and back fill system with argon through valve A-8 to a pressure of 3.0 ± 0.5 in. H_2O . Place base tank pressure controller (photohelic) on automatic and set at 3.0 ± 0.5 in. H_2O .

2.4 NaK System Operation

The purpose of the NaK system is to provide thermal control of the sodium injection nozzle. When the arc furnace is operating without sodium injection or under high power conditions, the NaK system must remove heat from the nozzle to prevent excessive temperature. Before flowing sodium through the injection nozzle, the temperature of all of the sodium flow passages must be above the melting point of sodium. In this case, the NaK system supplies the necessary heat. The NaK system is also used to preheat atomizing gas for the sodium injection nozzle. This is accomplished by flowing the argon through the NaK line heat exchanger before entering the nozzle. The NaK system is shown schematically in Fig. 2.

2.4.1 Filling NaK System from Sump Tank

1. Close valves Na KV-1, -2, -3 and -6, and open valves Na KV-4 and -5 (Fig. 2).
2. Evacuate NaK system through the expansion tank to < 100 microns.
3. Pressurize NaK sump tank to 2 psig argon.
4. Open Na KV-1 and fill system with NaK to the mid-level probe in the expansion tank, then close Na KV-1.
5. Pressurize the expansion tank to 5 psig.

2.4.2 NaK Loop Operation

After the NaK loop is filled, begin recirculation by energizing the NaK pump and increasing the pump powerstat until the desired flow rate is reached. Energize the NaK heater power and set automatic temperature control at 150-200°C. NOTE -- This temperature controller should be set to provide the desired temperature at the sodium injection nozzle. Also, increased NaK flow rate will tend to isothermalize the NaK system and transfer more heat to or from the sodium nozzle.

2.4.3 NaK Loop Shutdown

Recirculation of the NaK can be stopped at any time by de-energizing the EM pump. If the system is going to be shut down for an extended period (longer than one day), the NaK should be drained back into the sump tank. This is accomplished as follows:

1. Set the expansion tank pressure slightly higher than the sump tank pressure.
2. De-energize EM pump.
3. Open dump valve (Na KV-1).
4. Vent argon pressure from sump tank.
5. After 10-15 min, close dump valve (Na KV-1).
6. Maintain 1-3 psig argon pressure on sump tank and 3-5 psig on expansion tank.

2.5 Preheating Sodium Injection Nozzle

Before sodium is introduced into the sodium injection nozzle, the temperature of all of the sodium flow passages must be above the melting point of sodium. This is accomplished by flowing hot NaK around the nozzle as indicated in Section 2.4.

2.5.1 Check that the nozzle holder vacuum chamber is evacuated before starting NaK flow.

2.5.2 Start NaK flow in accordance with Section 2.4 and heat nozzle to $150 \pm 5^{\circ}\text{C}$.

2.6 Recirculating Sodium

The purpose of the recirculating sodium system is for checking out and debugging the system prior to injecting sodium into the chemical reactor.

2.6.1 Preheat the sodium system in accordance with Section 2.3 and liquefy the sodium in drum #1 in accordance with Section 2.2. Open sodium valves Na-1 and Na-6. Sodium will drain from drum #1 into the base tank and a flow signal will be indicated from FM-1. As the sodium fills the base tank, level indicators L5, L4, L3 and L2 will make contact sequentially. When L2 indicates contact, close sodium valve Na-6.

2.6.2 Close the following valves: Na-15, -14, -10 and -12 and open the following valves: Na-16, -9, -11 and -13. Energize the EM pump and establish sodium flow through the recirculating system. If the base tank sodium level falls below level indicator L3, open Na-6 and raise the level back to L2. Continue operation as long as necessary to functionally check all parts of the system.

2.7 Precautions Before Energizing Arc Heater

Before the chemical reactor arc heaters are energized, the sodium injection nozzle must be protected from excessive temperatures.

2.7.1 Initiate NaK flow through the nozzle jacket and maintain between 0.5-1.5 gpm.

2.7.2 Initiate nozzle atomizing argon flow and maintain between 25 and 50 scfm.

2.7.3 Initiate nozzle purge gas flow and maintain between 20 and 40 scfh.

3.0 STARTUP (INJECTING SODIUM INTO CHEMICAL REACTOR)

The prestart procedures (Section 2) should be completed before initiation of sodium injection into the chemical reactor.

3.1 Preparing to Inject Sodium into Chemical Reactor

With the sodium injection nozzle preheated to $\sim 150^{\circ}\text{C}$ with NaK flow at ~ 1 gpm and sodium flowing through the recirculating system, increase argon flow to the injection nozzle to 50 scfm. Monitor injection nozzle temperature and maintain at 150°C . De-energize sodium EM pump, close valves A-13, Na-15 and Na-13. Open valve Na-14.

3.2 Establishing Automatic Sodium Level Control in Base Tank

Check that the sodium in all five drums have been liquefied. Open manual sodium drain valves Na-1, Na-3 and Na-4. Activate automatic sodium level control by placing the control switch for valves Na-6, -7 and -8 in automatic and activating the programmable controller automatic level control.

3.3 Transfer of Sodium Flow Control to Main Control Room

The sodium supply system is now fully operational and is ready to inject sodium into the chemical reactor.

3.3.1 The sodium system operator will make voice contact with the main control room operator and inform him to set the automatic sodium flow control at zero in preparation for the control transfer. When the main control room operator is ready, sodium flow control will be switched from the local panel to the main control room.

3.3.2 The main control room operator can control sodium flow at will by opening valve Na-15 energizing sodium EM pump and adjusting the automatic controller to the desired flowrate.

4.0 OPERATION

4.1 Monitoring Sodium System Performance

The sodium system operator will continually monitor the performance of the sodium and NaK systems during operation. Level control of the base tank will be periodically checked to insure that the sodium level is maintained between level probe L2 and L3. Temperatures around the system and cover gas pressures will also be periodically scanned for abnormal conditions. Argon flow to the sodium injection nozzle will require careful monitoring to insure adequate atomizing gas flow.

4.2 Replacing Empty Sodium Drums

The five sodium drums are connected to the base tank so that they can be emptied one at a time and then replaced and have time for the sodium in the replacement drum to liquefy before it is needed for continuous operation. The drum draining sequence is #1, 4, 2, 5, 3, and then back to #1. Then the remote activated automatic valve sequence is Na-6, Na-8, Na-6, Na-8, Na-7, and then back to Na-6 to start sequence over. Manual valves Na-1 through Na-5 must be set by the sodium system operator at the change of each drum to coincide with the drum draining sequence.

When a sodium drum becomes empty as indicated by the loss of drain flow, the programmable controller will switch to and start draining the next drum in the sequence. An audible and visual alarm will also activate at the local sodium panel. The sodium operator will proceed as follows:

1. Dress in safety clothing as indicated in Section 1.2
2. Turn off and unplug all heaters and instrumentation to the empty drum.

3. Close the drum drain valve (Na-1, -5) at the bottom of the empty drum.
4. Initiate the drain line purge gas and disconnect the drain line from the empty drum. Cap the disconnected drain line side.
5. Close cover gas valve (A-1, -5) to the empty drum and disconnect the cover gas line. Cap the disconnected line to the drum.
6. Remove the empty drum, including heating jacket and transport pallet from the drain stand and place it on the cooldown stand.
7. Install a full sodium drum (previously prepared with heating jacket, sodium and gas fittings installed) on the drain stand in place of the empty drum.
8. Remove cap from disconnected drain line and with purge gas flowing connect to the replacement drum drain valve (Na-1, -5).
9. Slightly open cover gas valve A-1, -5 so that gas is flowing and connect cover gas line to replacement drum, then open valve fully.
10. Plug in all heater and instrumentative circuits to the replacement drum.
11. Open the companion drum drain valve (i.e., if drum #1 has just been replaced, open drain valve to drum #2, etc.).
12. Liquefy sodium in replacement drum in accordance with Section 2.2.
13. Prepare another full drum of sodium by installing heater jacket, sodium drain fitting and cover gas fitting, for replacement of the next empty drum.

14. When empty drum on the cooldown stand has cooled down below 100°C, remove heater jacket, cover gas and sodium drain fittings and install plugs in the drum bungs.

5.0 SHUTDOWN (NORMAL)

A normal shutdown is a preplanned or unhurried orderly sequence that is carried out to reduce temperatures, flows, pressures, etc. to a safe standby mode with the minimum damage to the equipment.

5.1 Stopping Sodium Flow

Stop sodium flow to the chemical reactor by reducing the automatic flow control to zero, then returning flow control back to the sodium system local control panel. De-energize EM pump.

5.2 Sodium Piping Blowdown (Refer to Fig. 1)

Close sodium valves Na-11 and Na-12 and open Na-13. Open gas valves A-11 and A-13 and blow sodium in recirculating line back into base tank. A sudden rise in the blowdown gas flowrate will indicate that the line has been emptied of sodium. Close sodium valve Na-13 and open Na-11 and -12. A reverse flow should be indicated by flowmeter FM-4. When this reverse flow signal stops or when the base tank's higher level probe L1 indicates, immediately close Na-11 and -12. Close sodium valve Na-14 and open Na-15. (NOTE -- Valve Na-15 must be unlocked by main control room before it will open.) When the blowdown flowmeter indicates a sudden increase in flow, reduce the gas flow to 5-10 scfh.

5.3 Post Shutdown Standby Conditions (Refer to Fig. 1)

After the arc heaters have been turned off for several minutes, stop the nozzle atomizing gas by closing A-10 and -14. Close sodium drain valves Na-1 through Na-8. De-energize all sodium system heaters, including drum heaters, trace heaters and base tank heaters. Shut down the NaK system in accordance with Section 2.4.3. Set and maintain the following conditions:

1. Close the following valves: Na-1, -2, -3, -4, -5, -6, -7, -8, -11, -12, -13, -14 and -16, and A-10, -12 and -14.
2. Open the following valves: Na-9, -10 and -15, and A-1, -2, -3, -4, -5, -6, -7, -8, -9, -11 and -13.
3. Maintain a purge flow of 3-5 scfh through the sodium injection nozzle.
4. Maintain cover gas pressure (1-3 in. H₂O) on base tank and sodium drums.
5. Turn off main electrical disconnect switches to the sodium drum heaters, trace heaters, EM pump and NaK main heater.
6. Turn all control switches from automatic to manual.

6.0 SHUTDOWN (EMERGENCY)

An emergency shutdown is one that is designed to quickly lessen the real or potential danger to personnel and/or equipment caused by malfunctioning equipment or an uncontrolled condition.

6.1 Conditions Requiring Emergency Shutdown

The following conditions will require an emergency shutdown of the entire silicon production facility.

1. Fire, explosion, or a major leak of SiCl_4 , sodium, hydrogen or water.
2. A major rupture of the chemical reactor.
3. Uncontrolled conditions of electrical discharge, reactant flow or hydrogen flow.
4. Plugging of the chemical reactor gas discharge line.

6.2 Emergency Shutdown Procedure

PUSH EMERGENCY SHUTDOWN BUTTON. The following will occur:

1. Arc heater power will de-energize.
2. Argon and hydrogen flow to the arc heater will stop.
3. DC field supply will be de-energized.
4. Sodium drum and trace heater power will de-energize.
5. Silicon test cell emergency fan will be energized.
6. Sodium room emergency fan will be energized.____
7. Sodium and SiCl_4 pumps will de-energize.
8. Audible and visual alarm will activate in the main control room at the sodium panel, at the electrical panel, and at the SiCl_4 room.

6.3 Post-Emergency Standby Conditions

After an emergency shutdown has been initiated, perform the following as soon as it is safe to be in the area:

1. Analyze the reason for the shutdown and perform the sodium piping blowdown procedure (Section 5.2) if this operation does not compromise safety of personnel or equipment.

2. If there is any doubt about the safety of the blowdown operation, do not perform the blowdown operation, but close all sodium valves (Na-1 through Na-16) and perform the operation indicated in Section 5.3 except for items 1, 2 and 3.
3. If the blowdown procedure in No. 1 above has been performed, set the standby conditions in accordance with the normal shutdown standby conditions in Section 5.3.

7.0 DECONTAMINATION (SODIUM AND NaK)

Decontamination in this procedure refers to the process of removing alkali metal and its residue from parts, components and equipment.

NOTE -- Observe safety precautions and practices stated in Section 1 when decontaminating.

7.1 Cleaning Parts Contaminated with Sodium or NaK

The first step in cleaning parts contaminated with the alkali metals (Na and NaK) is to remove all of the bulk material (practical) into a separate container for controlled disposal (see Section 7.2). The next step is determined by the size, complexity and amount of the remaining alkali metal in the part to be decontaminated.

7.1.1 If the part to be decontaminated is large or with complex passages or with an unknown quantity of alkali metal remaining, proceed as follows:

1. Open all ports possible to prevent excessive pressure build-up.
2. Start a flow of pure argon or nitrogen into the component and gradually add steam to the inert gas flow.

3. Continue to increase steam ratio, while monitoring the reaction, until pure steam is flowing through component.
4. Continue steam flow for several minutes after all signs of reaction has stopped.
5. Carefully replace steam flow with water and flush water for several minutes.
6. Drain water from component and dry by purging, heating, and/or evacuating.

7.1.2 If the part to be decontaminated is small and delicate, proceed as follows:

1. Prepare a beaker of methanol so that the part to be decontaminated can be fully submerged.
2. Prepare a metal cover to go over the methanol beaker to smother a flame if it should catch fire.
3. Carefully insert the part to be decontaminated into the beaker and allow it to remain until all bubbling has stopped.
4. Remove part from methanol and flush with water.
5. Dry by vacuum baking for several hours.

7.1.3 If the part is only contaminated on the external surfaces or simple open internal surfaces and the quantity of alkali metal is small, proceed as follows:

1. Place the part in an open tray in a well-ventilated area and carefully flush with water from a safe distance.
2. Dry by vacuum baking for several hours.

7.2 Disposing of Waste Sodium

The waste sodium remaining in the sodium drums will be returned to DuPont for disposal. Other waste alkali metal will be handled as follows:

1. Place the waste alkali metal in a dry open tray in a well-ventilated area. The quantity at any one time will be limited to 100 grams.
2. Use water to react away the alkali metal by spraying with a hose from a distance. Gradually allow water to come in contact with the alkali metal. Do not hit it with a large stream all at once.
3. Flush all residue away with large quantities of water.

7.3 Clean-Up After a Sodium Spill

The following procedure should be used to clean up a sodium spill:

1. If sodium is burning, follow fire fighting procedure in Section 1.5.
2. Allow sodium to solidify and cool to room temperature before disturbing.
3. Dress in safety clothing in accordance with Section 1.
4. Scrape, shovel and sweep up spilled sodium and place in a metal pan.
5. Dispose of waste sodium in accordance with Section 7.2.

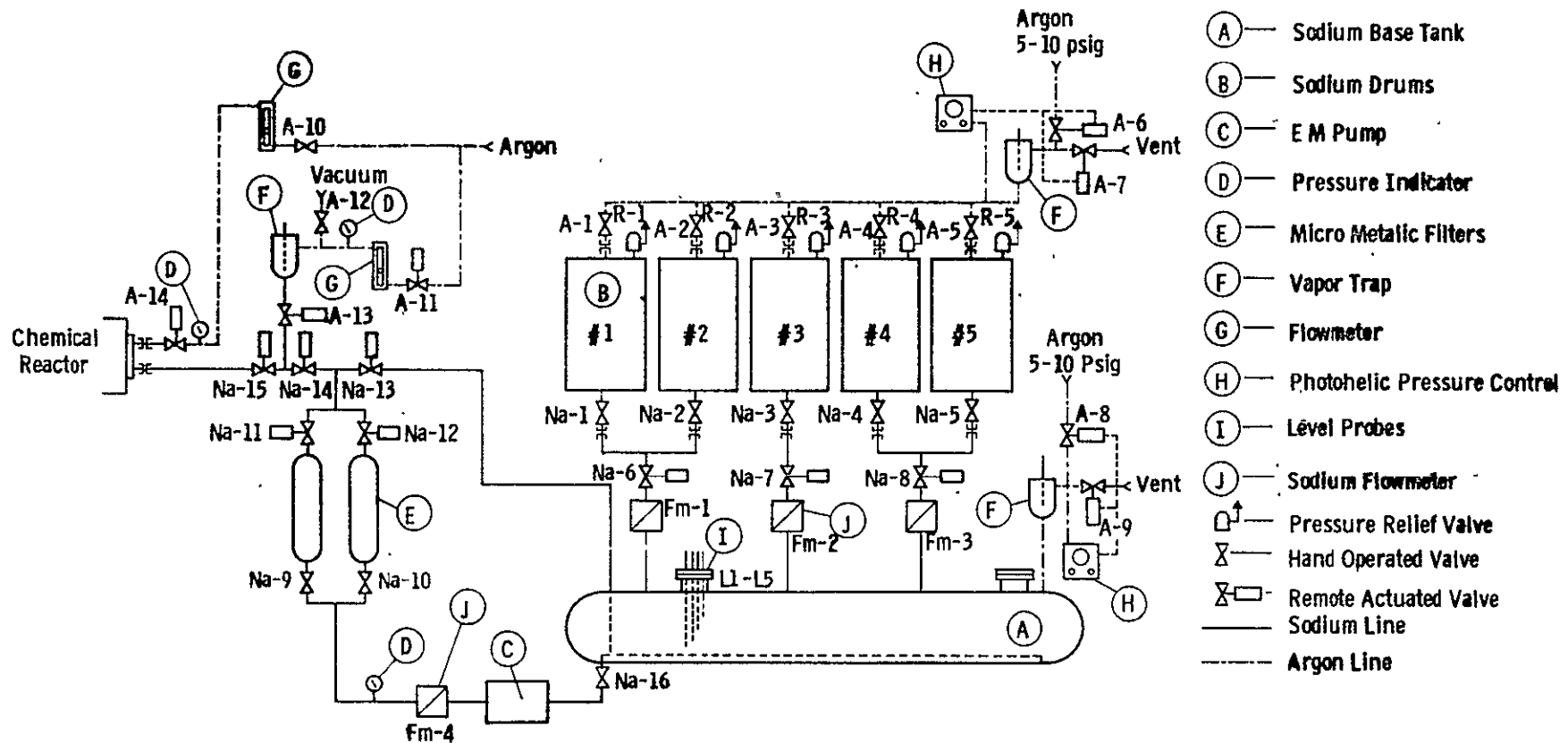


Fig. 1 — Sodium supply system

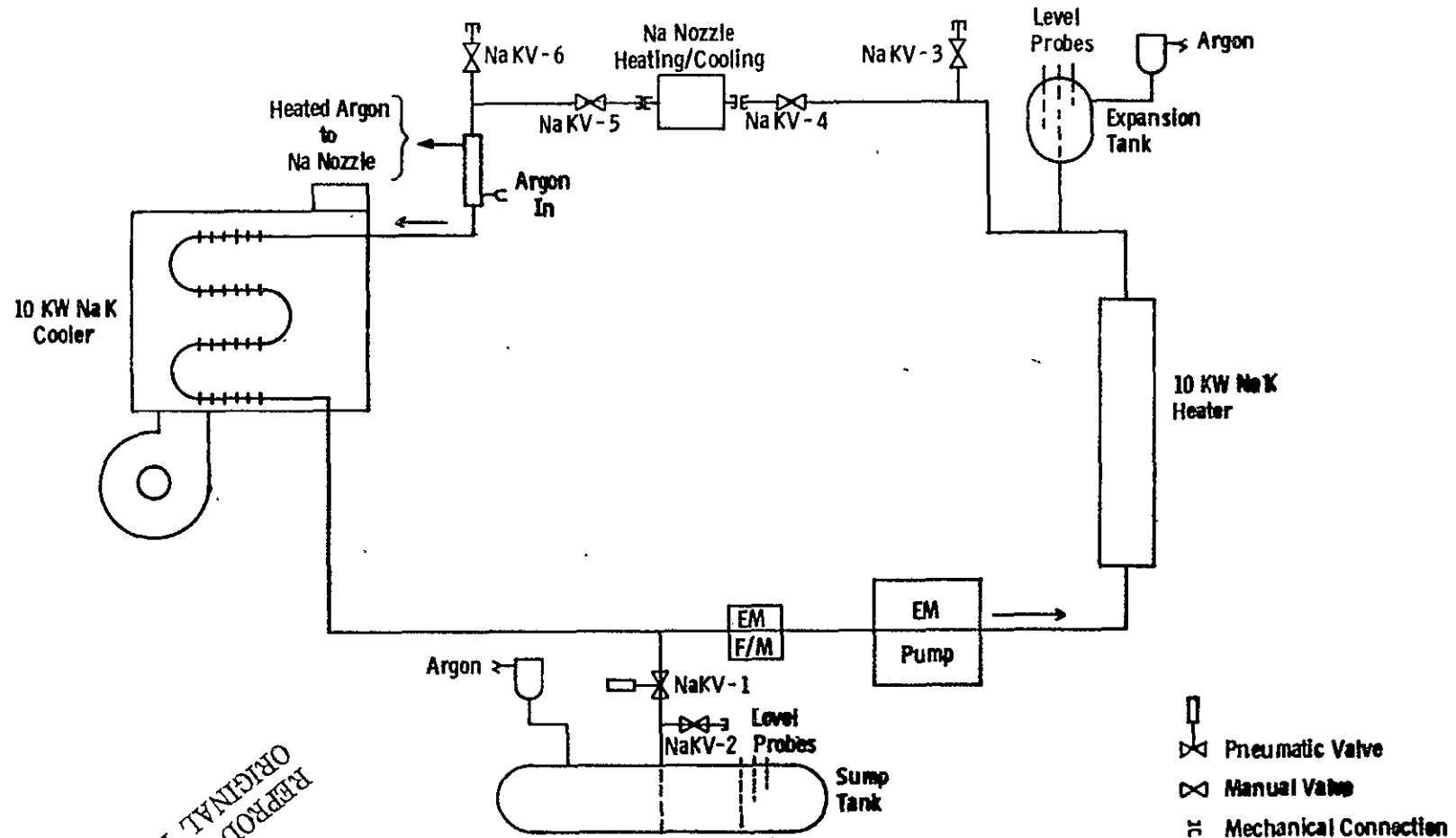


Fig. 2 - Na K loop for Na nozzle temperature control