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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

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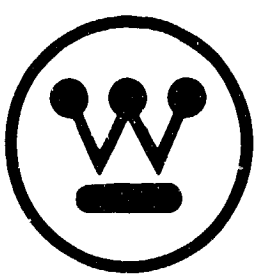

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1. INTRODUCTION

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, 2]

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 quarter, the detailed design effort was completed for the test system preparation which has been divided into two categories: 1) system components and 2) test system-laboratory integration. In addition, procurement has continued while fabrication and assembly/installation of the test equipment were initiated for the experimental verification unit for silicon production. Specifications for all subsystem control and instrumentation (C&I) and the associated control panels were completed. All C&I components were procured and receipt of components began. Assembly of the subsystem control panels was initiated and installation of the panels in the control room is progressing as the panels are completed.

The required components for the plasma reactor (i.e., reactor shells, arc heaters, cyclone housing, etc.) were ordered and fabrication is nearing completion. Likewise, both the graphite liners and refractory liners were ordered and vendor fabrication was initiated. Final arrangements were made for applying the internal nickel plating to the metal reactor shells.

Procurement of system components for both the $SiCl_4$ and the sodium injection systems was completed. Both of these chemical subsystems are currently in the fabrication/assembly activity. Fabrication of the $SiCl_4$ storage tanks was initiated by the vendor following approval of the tank drawings. Main subassemblies of the sodium system are nearing completion and will be installed at the Arc Heater Laboratory during the next quarter.

Order placement was accomplished for the gas scrubber and the waste water treatment tank and controls of the effluent disposal system. Fabrication drawings were reviewed and vendor fabrication was initiated. Likewise orders were placed for the components of the silicon collector (i.e., housing, quartz crucibles, and graphite internals). An expendable silicon collector using a castable

refractory was designed for use during initial shakedown tests. Fabrication of this disposable collector is in progress.

Assembly and installation of the required electrical gear was initiated while procurement continued during the quarter. Such items as the A.C. and D.C. bus work, transfer switches, current transformers, distribution panels and power cable tray installations are in progress. Final design and procurement of both the gas mixing panel and bulk gas supplies were resolved with Air Products Co. Site preparation and specifications for the gas storage tanks are in progress with Air Products. Installation of the components (pumps, demineralizer and piping) for the cooling water system was initiated following component deliveries from the vendors. Various support structures and water manifolds also have been installed. The gas burnoff stack was fabricated, received and erected at the test site.

Preparation of the system operations manual and the safety manual was begun during the period. The operations manual details all aspects of the test system operation (e.g., prestart, start up, testing, shut down, and emergency shut down). In addition, a detailed test schedule was formulated for the shakedown and demonstration tests. A system safety manual was started and will contain pertinent safety information for the test system personnel (both operations and maintenance) as well as serving as a safety training document.

Detailed characterization of the Sonicore sodium injection nozzle using water as the test fluid has been completed. Results indicate that flow rates of 45 gph sodium and 50 scfm argon should produce sufficiently small droplet sizes. Verification of the nozzle operation with liquid sodium is almost complete. The nozzle functioned properly and showed no tendency to plug. Sieving of sprayed sodium particles is in progress. The silicon tetrachloride feed system is being constructed and it is planned to commence testing of the hydraulic spray nozzle within the next few weeks.

The reactant injection systems were incorporated into the test apparatus for the Kinetics studies. Full power tests were run with different hydrogen-argon flowrates and $SiCl_4$ injection. Silicon was produced by the hydrogen reduction of $SiCl_4$. The injection flange was modified in order to extend the range of operating parameters. Experiments incorporating $SiCl_4$ and Na injection are being prepared.

Fabrication and assembly of the test apparatus for the Reaction Demonstration tests has been completed. The Nupro metering valve has been calibrated for liquid sodium flow control. Based on shakedown tests in regard to system leak checking, minor modifications have been made in the test apparatus. A method has been devised for charging an evacuated silicon tetrachloride reservoir directly from the sealed drum supplied by the manufacturer. Partial reactant flow tests have been initiated.

3. PROCESS ECONOMICS

Task Investigators: W. H. Reed, T. N. Meyer

The process economics for the arc heater process for the continuous production (recycle mode) of silicon have been reevaluated in light of the condensation reaction mode.[1] As previously discussed, the economics consist of capital equipment costs, fixed capital estimations, and estimated silicon product cost.[2] For the reevaluation, the various cost elements were estimated based on 1) 3000 MT/yr of silicon, 2) a recycle process mode, and 3) a condensation (homogeneous) reaction mode. The results of this analysis are presented in Table 3.1 (capital equipment costs), Table 3.2 (estimation of fixed capital) and Table 3.3 (estimated silicon product cost). All dollar values were adjusted to 1975 dollars.

As shown in Table 3.3, the estimated product cost of $9.42/kg Si is projected to meet the 1986 DOE/JPL goal of $10/kg Si. In addition, a sensitivity analysis was performed to assess the effect of changes in the various cost items upon resultant silicon product cost. As shown in Figure 3.1, the cost items of labor, raw materials, arc heater utilities, and fixed capital contingency were varied by ± 10% of the nominal value. The resultant curves were plotted for the silicon product cost (1975 dollars) to indicate the variation in product cost resulting from changes in the above cost items. It can be seen that a 10% variation in any one of these factors cause less than a 2% variation in the estimated product cost.

Table 3.1

PURCHASED EQUIPMENT COST -- 3000 MT Si/yr (Recycle)
CONDENSATION REACTION MODE

ITEM	COST, $K
Chlorination System and Piping	626.9
$SiCl_4$ Purification System and Piping	424.1
Arc Heater/Reactor System	1138.0
Reactant Injection Systems	193.3
Gas/Salt Separator and Piping	56.6
Gas Recycle, Make-Up and Burnoff	198.3
Silicon Collection	486.7
Cooling Water System	204.1
Electrical Systems	1013.5
TOTAL	4341.5
ADJUSTED TO 1975	3660.0

Table 3.2

ESTIMATION OF FIXED CAPITAL*		3000 MT Si/yr (Recycle) CONDENSATION REACTION MODE
Purchased Equipment (PE)	100%	$ 3660.0 K
Install Purch. Equip.	43%	1573.8
Instrumentation & Control	13.5%	494.1
Building with Services	23.5%	860.1
Yard Improvements	11.5%	420.9
Service Facilities, Installed	55%	2013.0
Land	6%	219.6
Engr. and Supervision	32.5%	1189.5
Construction Expense	37.5%	1372.5
Contractor's Fee	19%	695.4
	Subtotal	$12498.9 K
Electrolysis Plant (Total Fixed Capital)*		6629.0
		19127.9
	Contingency (30%)	5738.4
*1975 Dollars	FIXED CAPITAL*	$24866.3 K

Table 3.3

ESTIMATION OF PRODUCT COSTS 3000 MT Si/yr (Recycle)
CONDENSATION REACTION MODE
FIXED CAPITAL: $24,866,270 (1975 Dollars)

		$/kg Si	
1.	DIRECT MANUF. COST		
	A. RAW MATERIALS	1.40	
	B. DIRECT OPERAT. LABOR (10 AT $5.90 M-HR)	.22	
	C. UTILITIES ($.03/kW-HR)	2.84	
	D. SUPERVISION & CLERICAL (15% OF l_B)	.03	
	E. MAINT. & REPAIR (10% OF FIXED CAPITAL)	.83	
	F. OPERATING SUPPLIES (20% OF l_E)	.17	
	G. LAB CHARGES (15% OF l_B)	.03	
	H. PATENTS & ROYALTIES (3% OF PRODUCT COST)	.28	
			5.80
2.	INDIRECT MANUF. COSTS		
	A. DEPRECIATION (10% OF FIXED CAPITAL)	.83	
	B. LOCAL TAXES (2% OF FIXED CAPITAL)	.17	
	C. INSURANCE (1% OF FIXED CAPITAL)	.08	
	D. INTEREST (8% OF FIXED CAPITAL)	.66	
			1.74
3.	PLANT OVERHEAD (60% OF $l_B + l_D + l_E$)	.65	
4.	TOTAL MANUF. COST (1 + 2 + 3)	8.19	
5.	GENERAL EXPENSES		
	A. ADMINISTRATION (6% MANUF. COST)	.49	
	B. DISTRIBUTION & SALES (6% MANUF. COST)	.49	
	C. RESEARCH & DEVEL. (3% MANUF. COST)	.25	
			1.23
6.	TOTAL COST OF PRODUCT, 4 + 5 (WITHOUT PROFIT)	$9.42/kg Si	

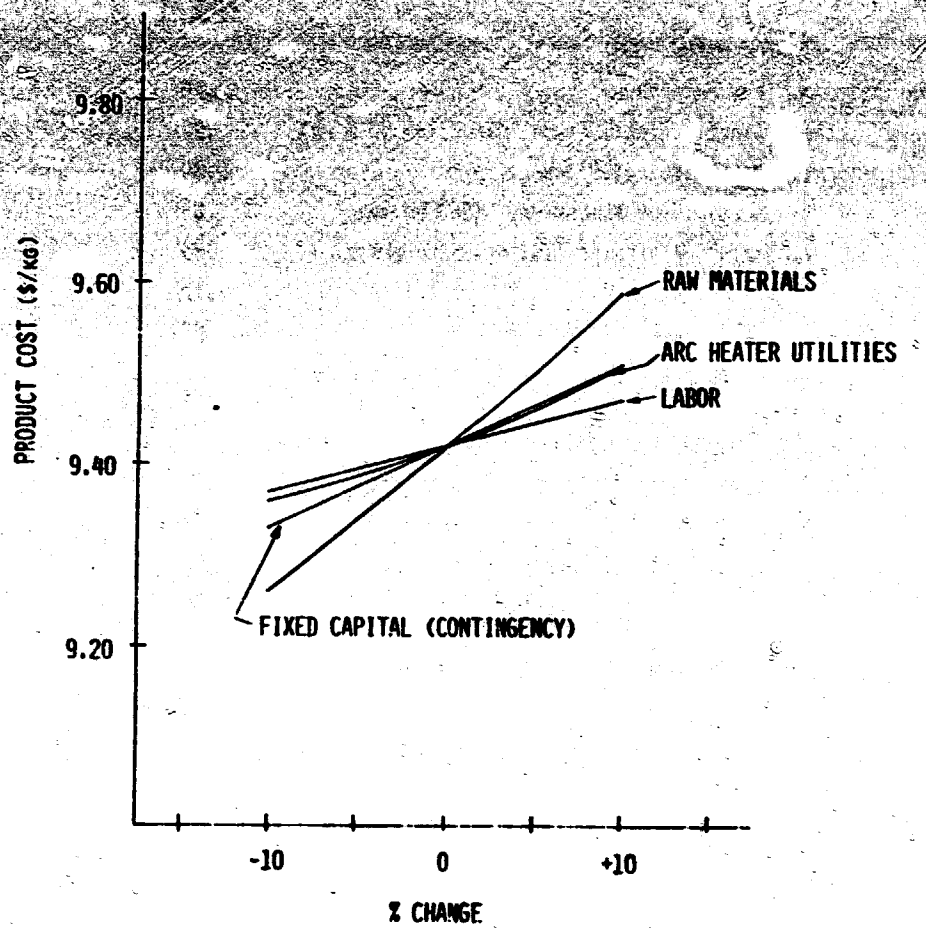

Figure 3.1 - Silicon Product Cost Sensitivity

4. TEST SYSTEM PREPARATION

4.1 Summary

During the quarter, the detailed design effort was completed for the test system preparation. In addition, procurement has continued while fabrication and assembly/installation of the test equipment were initiated for the experimental verification unit for silicon production. As the procurred items are received, these components are either assembled/installed directly at the Arc Heater Laboratory or used for fabrication of the system subassemblies. Installation of the experimental hardware is continuing in preparation for the testing effort scheduled to begin during the next quarter. The status of the subsystem activities is presented in Sections 4.2 and 4.3.

4.2 System Components

4.2.1 Control and Instrumentation (J. W. George, I. N. Bova, W. J. Melilli)

During the quarter, all specifications for C&I components were completed and issued to vendors for quotations. The submitted proposals have been reviewed and all major control and instrumentation components have been ordered. Delivery of the components is scheduled for August through December depending upon the lead time required by the various vendors. Engineering follow was provided for many of the orders in an effort to expedite the deliveries.

A schematic of the reactant feed-ratio controller is illustrated in Figure 4.1. This control system consists of the instrumentation required to control the ratio of silicon tetrachloride flow (controlled flow) relative to liquid sodium flow (wild flow). The instruments are pneumatic-type with 3-15 psig input/output control signals. The pneumatic signals are converted to electrical signals in the control room for purposes of logging the data. Not

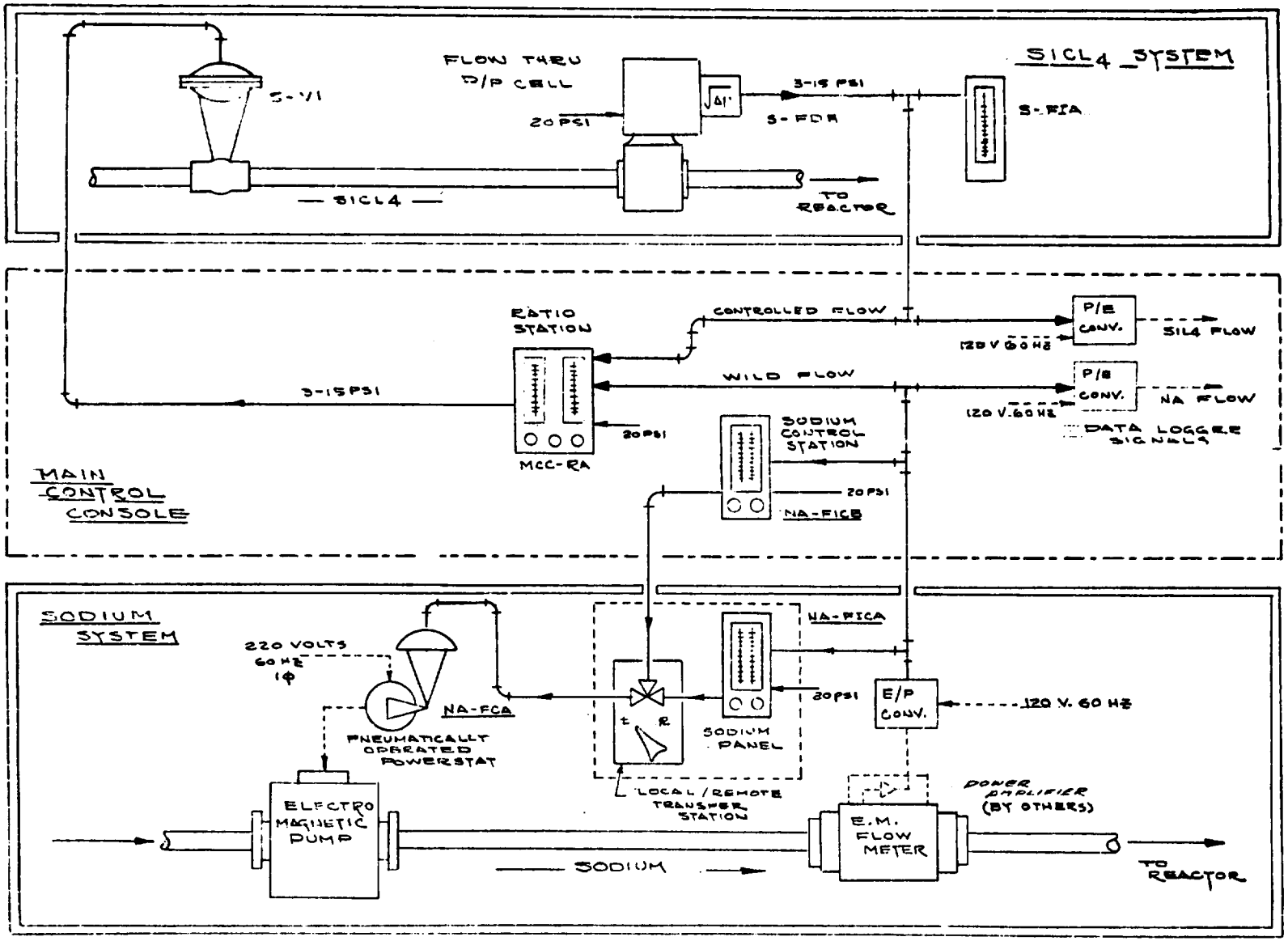

Figure 4.1 - Schematic Of The Na To $SiCl_4$ Feed Rate-Ratio Controller

shown on the schematic, is an alarm system that is designed to produce both an audio and visual alarm when either of the flows, sodium or silicon tetrachloride, change from the respective set points. Two pressure gauges with adjustable dead band, setable from the front of the gauge, are used to sense the flowrate change.

Layout of the sodium system control panel progressed during the quarter and will be completed early in the upcoming quarter. Fabrication of the sodium panel was started and the cutouts for mounting instruments have been made in several panel sections. The instruments are being mounted in the section cutouts as received from vendors.

Several of the panel sections for the electrical control system have been completed and will be installed in the panel racks located in the Arc Heater Laboratory during the upcoming quarter.

4.2.2 Plasma Reactor-Separator (C. B. Wolf, T. N. Meyer, H. T. Thunander)

During this reporting period all components of the plasma reactor-separator system were placed on order except the water and gas connecting hoses. Included in these procurements are the arc heaters, reactor shells, cyclone, cyclone outlet shells, and all graphite and refractory liners. Liner drawings were revised to include modifications of diametral and length tolerances required to suit the properties of readily available graphite materials. Following fabrication of the reactor shell sections, these items will be shipped directly to the vendor to have the internal nickel plating applied. All components of the plasma reactor-separator subsystem are scheduled for delivery next quarter with assembly/installation of the components to follow.

An estimation was made to determine the equilibrium silicon thickness as a function of heat flux rate and pyrolytic graphite thickness. Figure 4.2 shows a typical reactor section with its steel outer shell and graphite liner with a pyrolytic graphite inner layer. The curves predict the equilibrium thickness of solidified silicon which will accumulate depending on heat flux rate and various pyrolytic graphite layer thicknesses. In operation, this silicon layer will vary from less than 1 cm thick in the first reactor section to as much as 5 cm thick near the cyclone entrance. The average heat transfer rate to the cyclone wall is estimated to be approximately 16 watts/cm^2. In the cyclone, equilibrium silicon thickness is more sensitive to variations in heat transfer rates than in the reactor sections because the gas velocity is less sensitive to the silicon layer thickness. The walls are designed to maintain a skull wall for the predicted heat flux rates. In actual operation, where heat flux rates may vary because of reduced or increased reactant feed rates, it is expected that to control the thickness of the skull wall it will be necessary to monitor the heat flow to the cyclone. The power input to the arc heaters may be varied to adjust the heat flow to the cyclone wall in order that the skull thickness is properly controlled. It is expected that a satisfactory skull can be maintained over a heat flux variation of $\pm$ 25% of the nominal design value.

4.2.3 $SiCl_4$ System (R. E. Witkowski, G. C. Burrow, P. A. Ciarelli)

In this quarter, efforts were directed towards storage tank drawing preparation, component procurement, injection module construction and inspection and approval of the site facility. These efforts are described below.

The procurement of 90 percent of all $SiCl_4$ loop components has been completed. Figures 4.3 through 4.9 detail

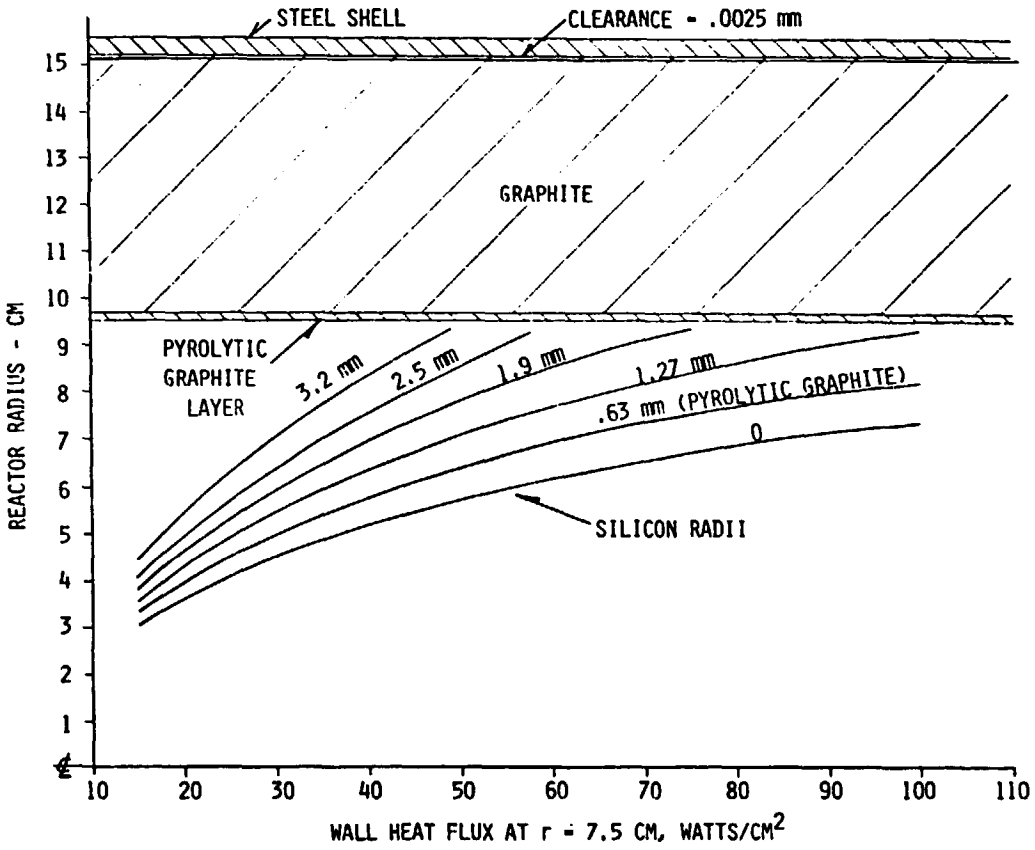

Figure 4.2 - Silicon Equilibrium Thickness As A Function Of Heat Flux For Various Thicknesses Of Pyrolytic Graphite

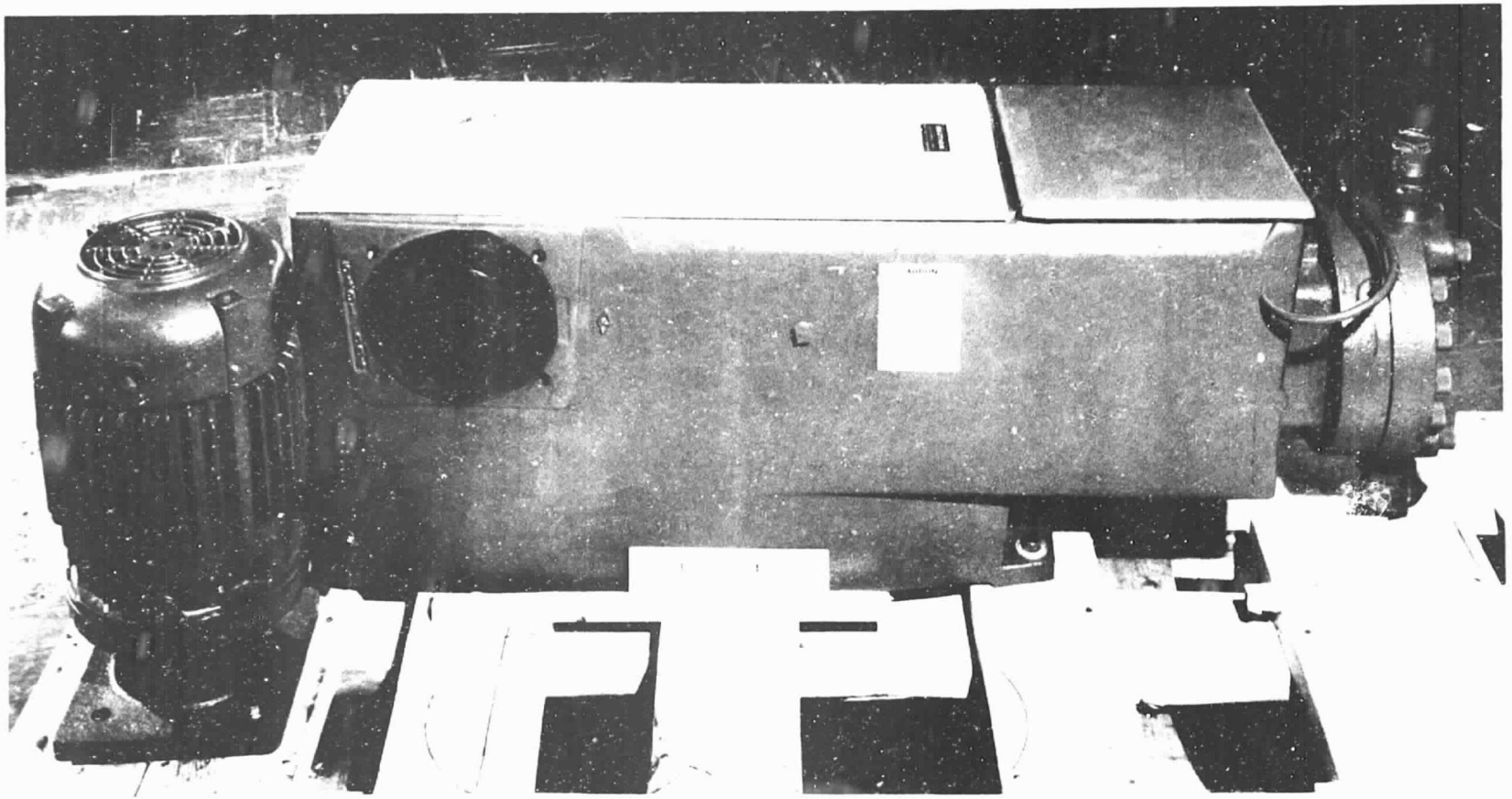

Figure 4.3 - Silicon Tetrachloride Positive Displacement Injection Pump

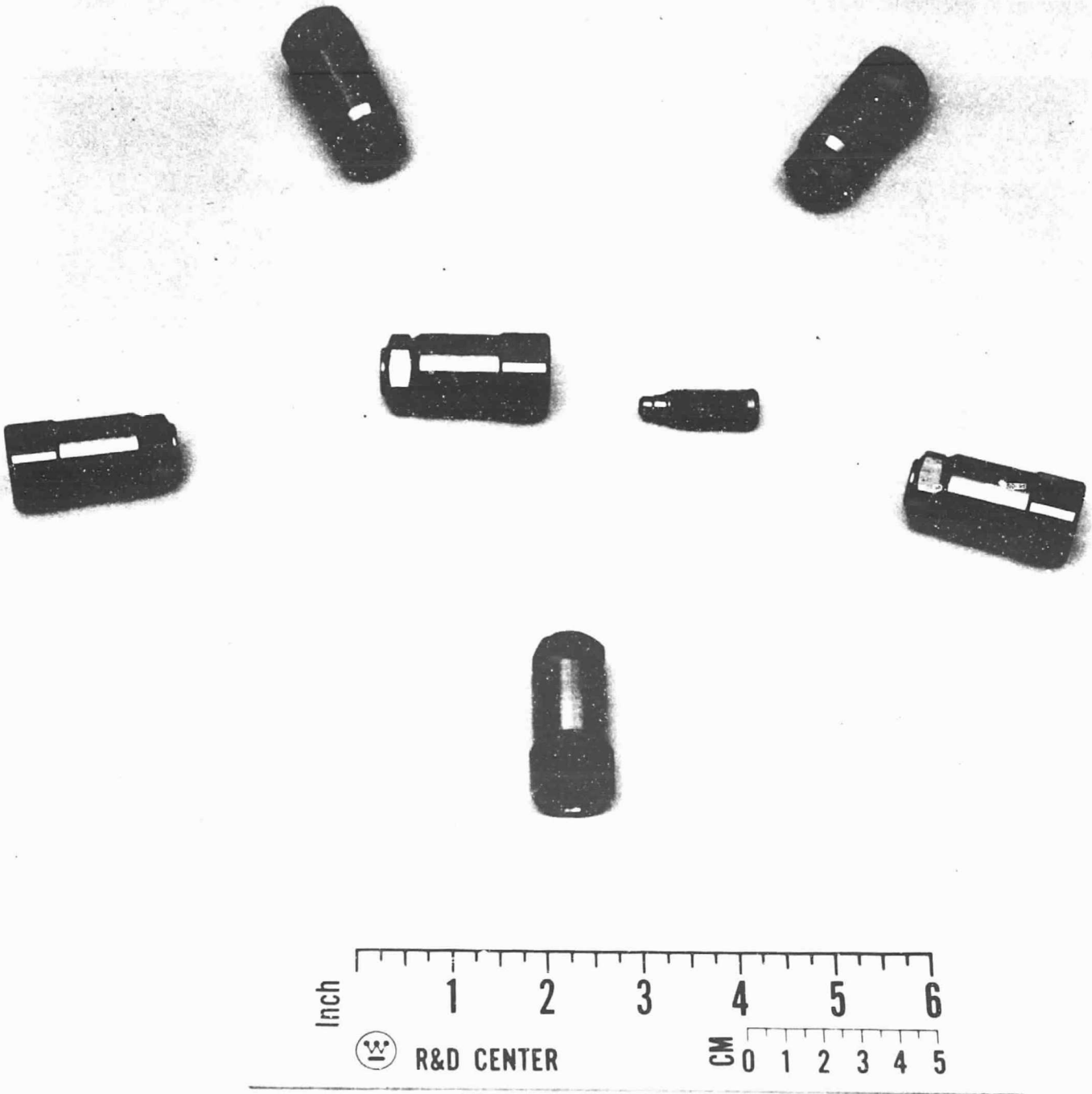

Figure 4.4 - Assembled Silicon Tetrachloride Injection Nozzles And Exploded View Showing Strainer Location

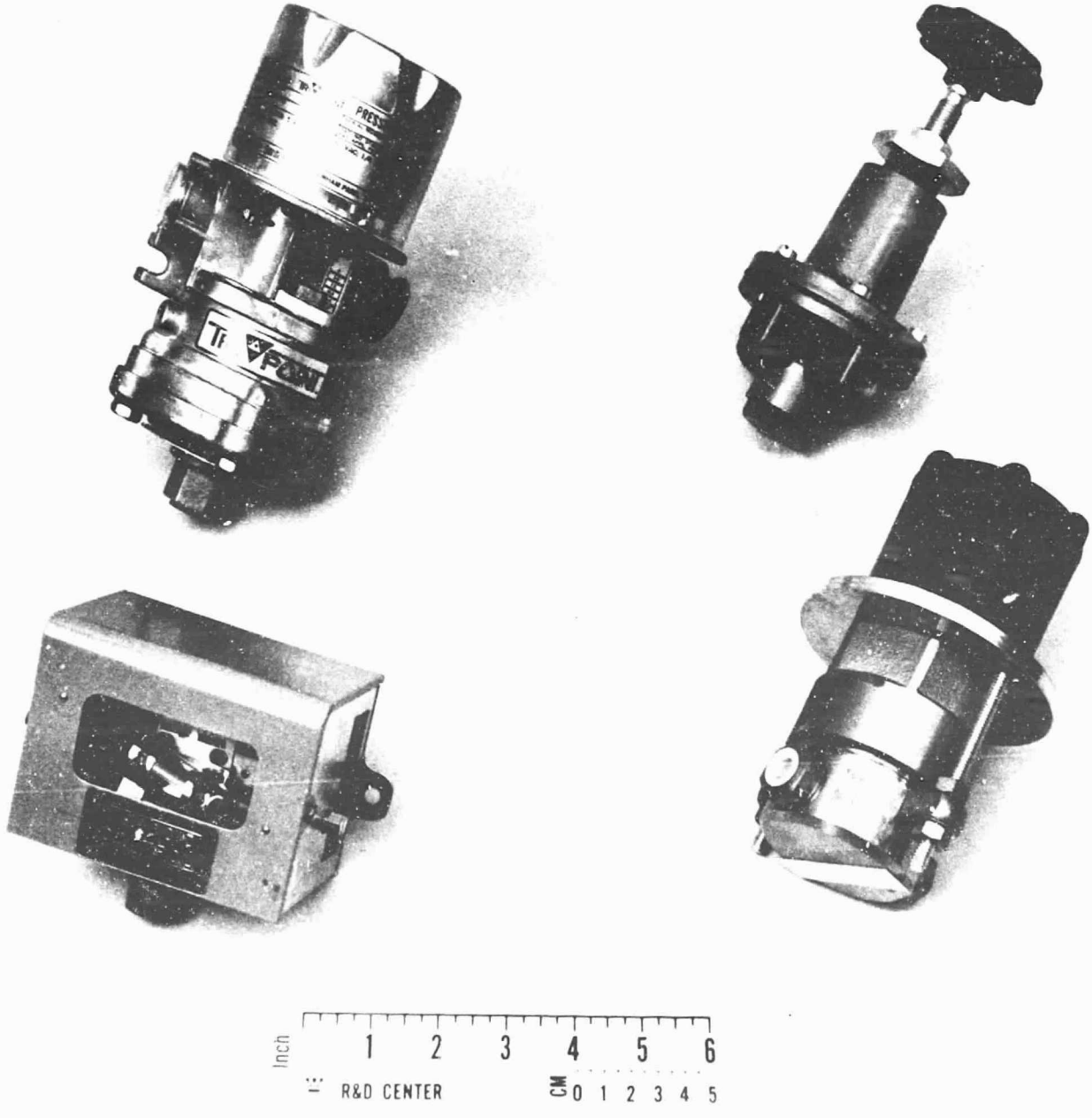

Figure 4.5 - Argon Cover Gas Pressure Switches (left) And Regulators (right)

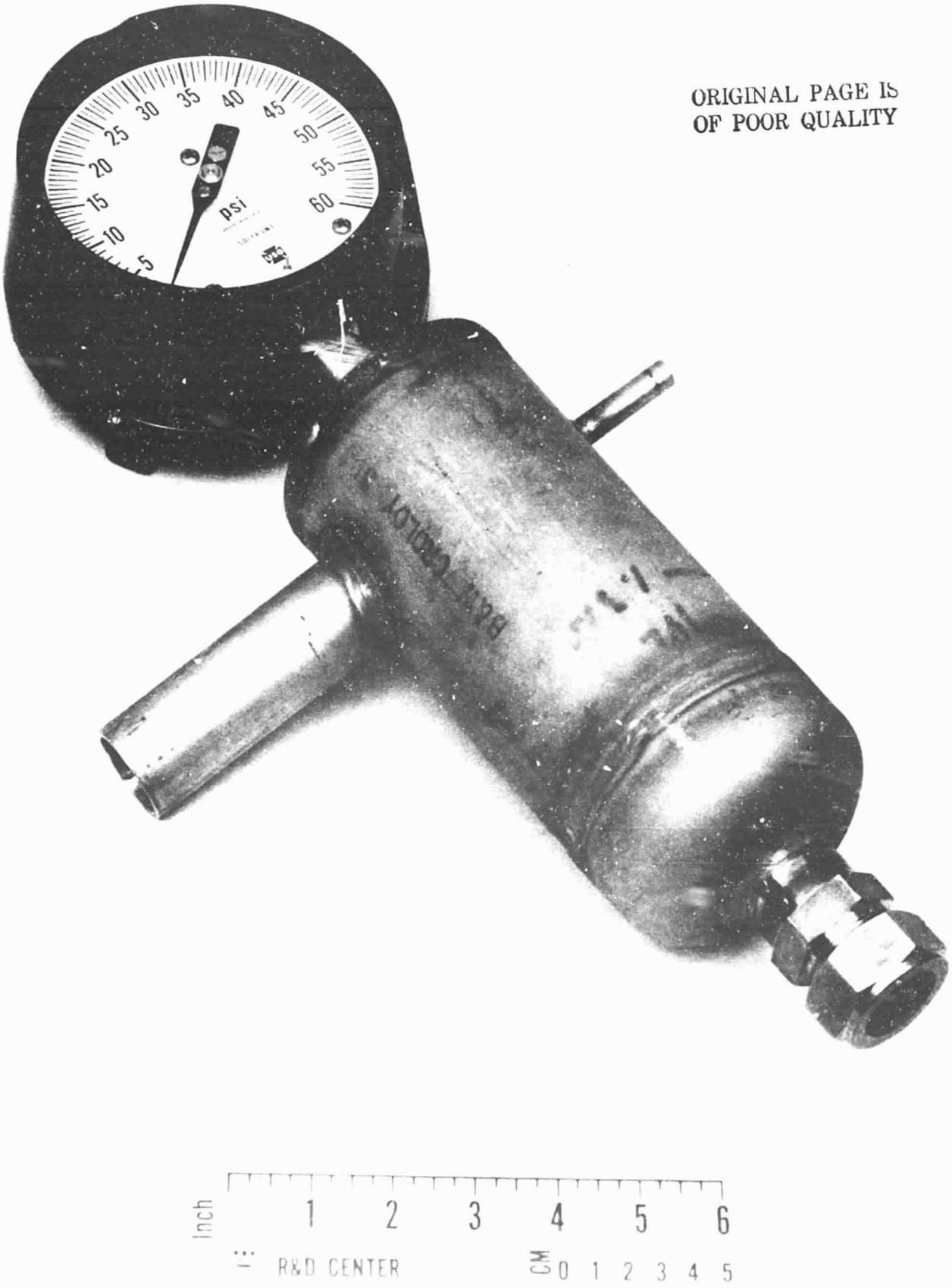

Figure 4.6 - Silicon Tetrachloride Vapor Trap And Associated Pressure Gauge

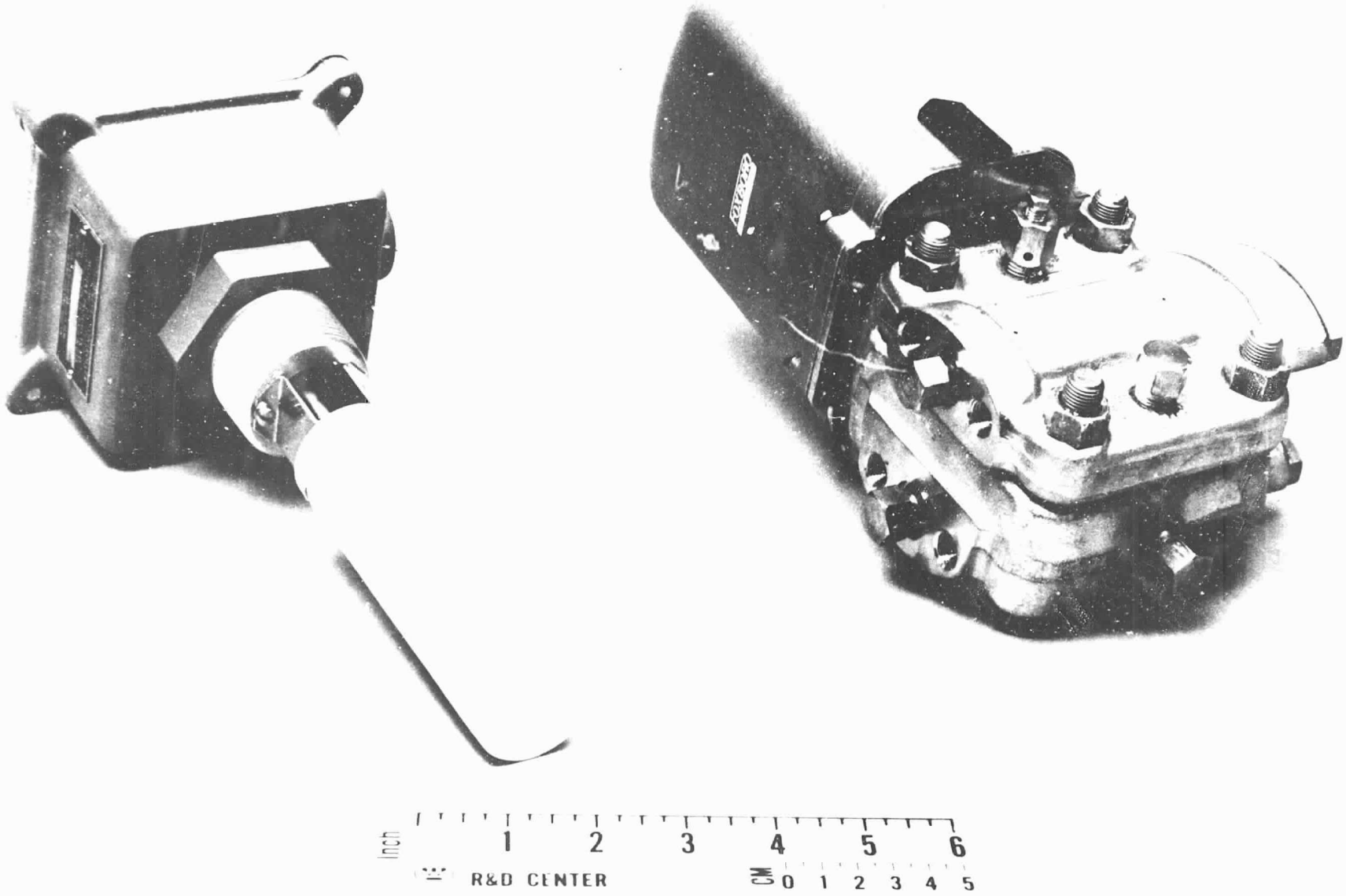

Figure 4.7 - Silicon Tetrachloride Overfill Indicator (left) And Liquid Level Probe (right)

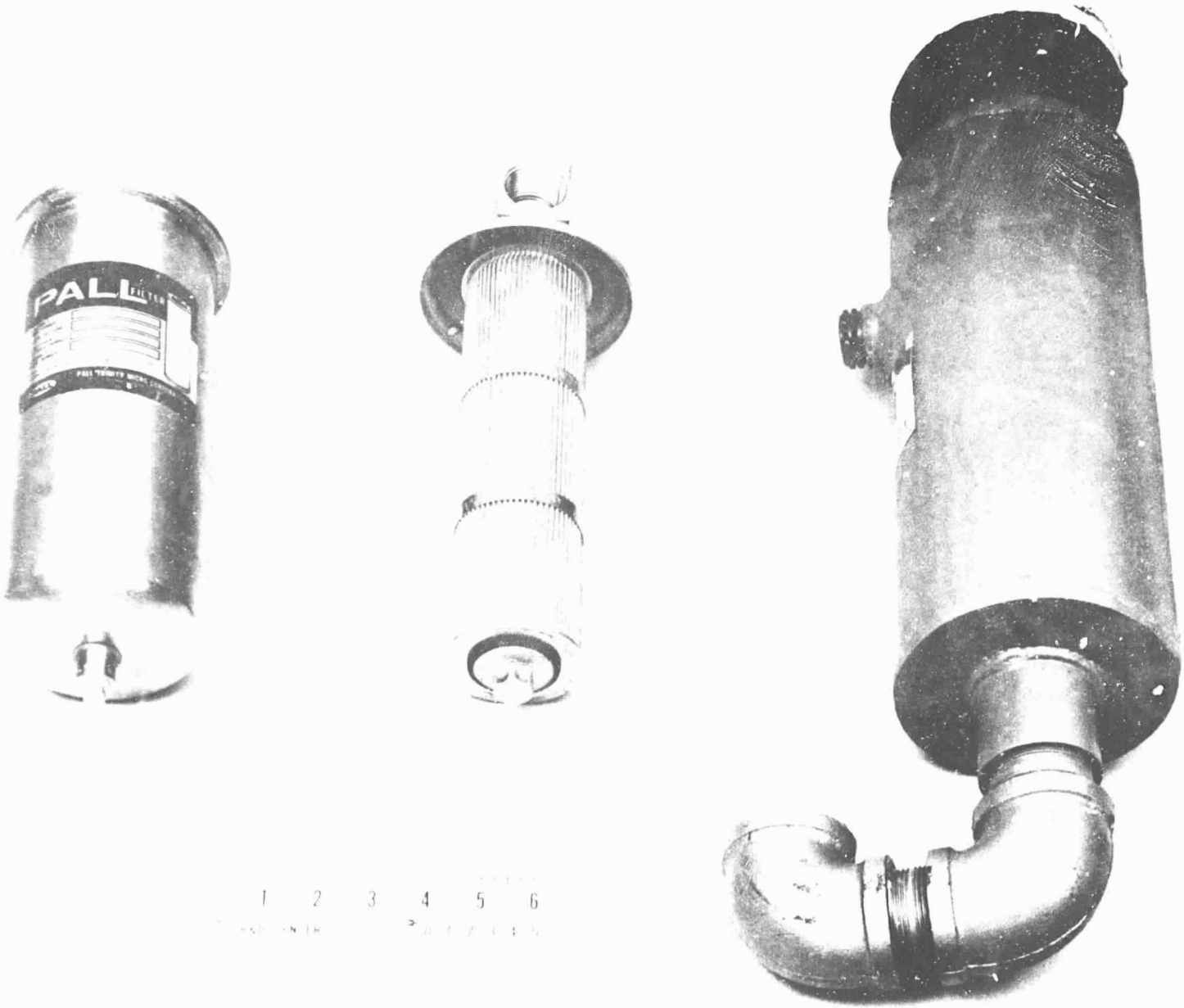

Figure 4.8 - Silicon Tetrachloride Particle Filter (left), 100 μm Mesh Filter (center) And Moisture Protection Cover Gas Column (right)

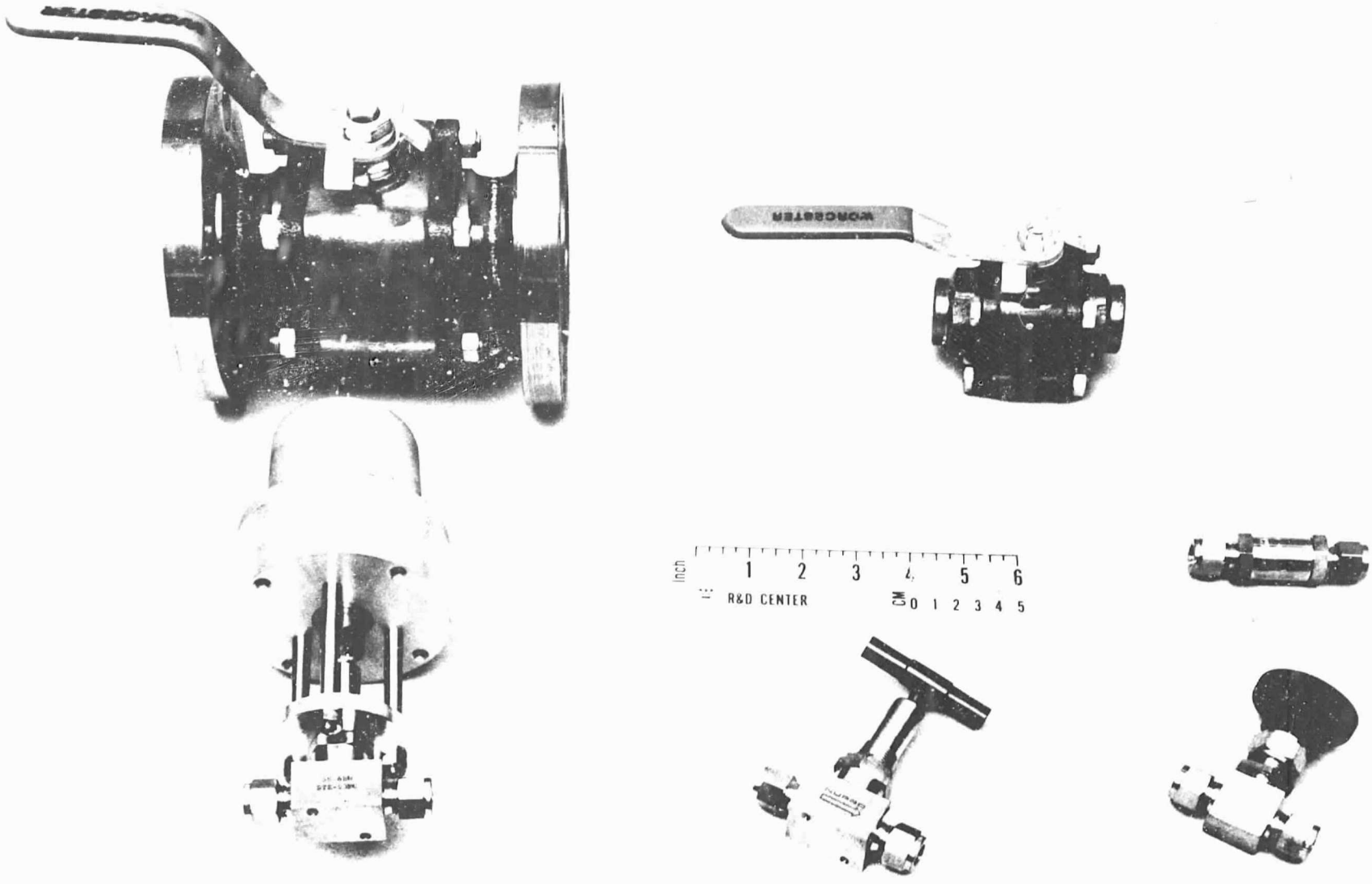

Figure 4.9 - Various Silicon Tetrachloride And Cover Gas Control Valves

examples of the major items which have been received. These include the $SiCl_4$ injection pump, nozzles, pressure switches and regulators, a $SiCl_4$ vapor trap, liquid level indicators, $SiCl_4$ particle filter, and assorted valves.

A purchase order was let for the $SiCl_4$ storage tanks to the Buffalo Tank Division of Bethlehem Steel. This tank manufacturer was recommended by the $SiCl_4$ supplier, Van De Mark Chemical Company. A formal quote was received with a delivery time to meet the project scheduling requirement. Final tank drawings were received and approved. The approved drawings have been received by Buffalo Tank and fabrication has begun.

Kincaid Manufacturing Company was contracted for construction of the injection loop enclosure. Final drawings were received and approved and Kincaid was supplied with a detailed front panel layout to enable component punch-outs to be made. Major fabrication is complete and delivery is scheduled for early October.

The task investigators met with a representative of the Foxboro Analytical Company to finalize details for the preparation of a quote on the Wilks Miran II Infrared process analyzer. This analyzer is intended for use as a means of monitoring $SiCl_4$ for moisture contamination. The Van De Mark R&D Coordinator was contacted for information on their methods for $SiCl_4$ quality control. They have supplied information concerning the details of the methods of chemical analysis that the Van De Mark Chemical Company employs in its quality control of C.P. grade $SiCl_4$.

The site facility for the $SiCl_4$ storage and injection loop was inspected and approved by the $SiCl_4$ task investigators. A meeting with the gas system task investigators was held to

finalize specifications for the argon piping network to the $SiCl_4$ module and storage tanks.

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

Fabrication of components and subassemblies for the sodium storage and injection system and the NaK thermal control system continued throughout the reporting period. Most of the purchased major components for these systems have been received.

Seven 30 kW drum heating jackets have been constructed. Figure 4.10 shows one of these jackets in the open position. The heating elements are arranged in three 10 kW zones with four elements making up each zone. Figure 4.11 shows the jacket being closed around a drum. When the jacket is fully closed, all of the heating elements are in contact with the drum. Figure 4.12 shows the jacket fully closed with the top lid in place. During operation the drum and jacket will set on a pad of thermal insulation.

Each heating zone in each drum heating jacket requires an RTD temperature element (Resistance Temperature Detector) for primary temperature control. The holders for these RTD's are shown in Figure 4.13. These are spring loaded devices that are to be installed in the heating jackets. The RTD is clamped in place by a friction nut shown at the top of each holder. When the heating jacket is closed around a sodium drum the temperature sensing element automatically contacts the drum at a preset spring pressure. Bimetallic thermal switches for over temperature cutout of each zone are designed to be similarly installed.

Preparation of a sodium drum for draining into the base tank entails installation of the heating jacket, inert gas fittings at the top of the drum and a filter and valve assembly at the bottom. Figure 4.14 shows a disassembled

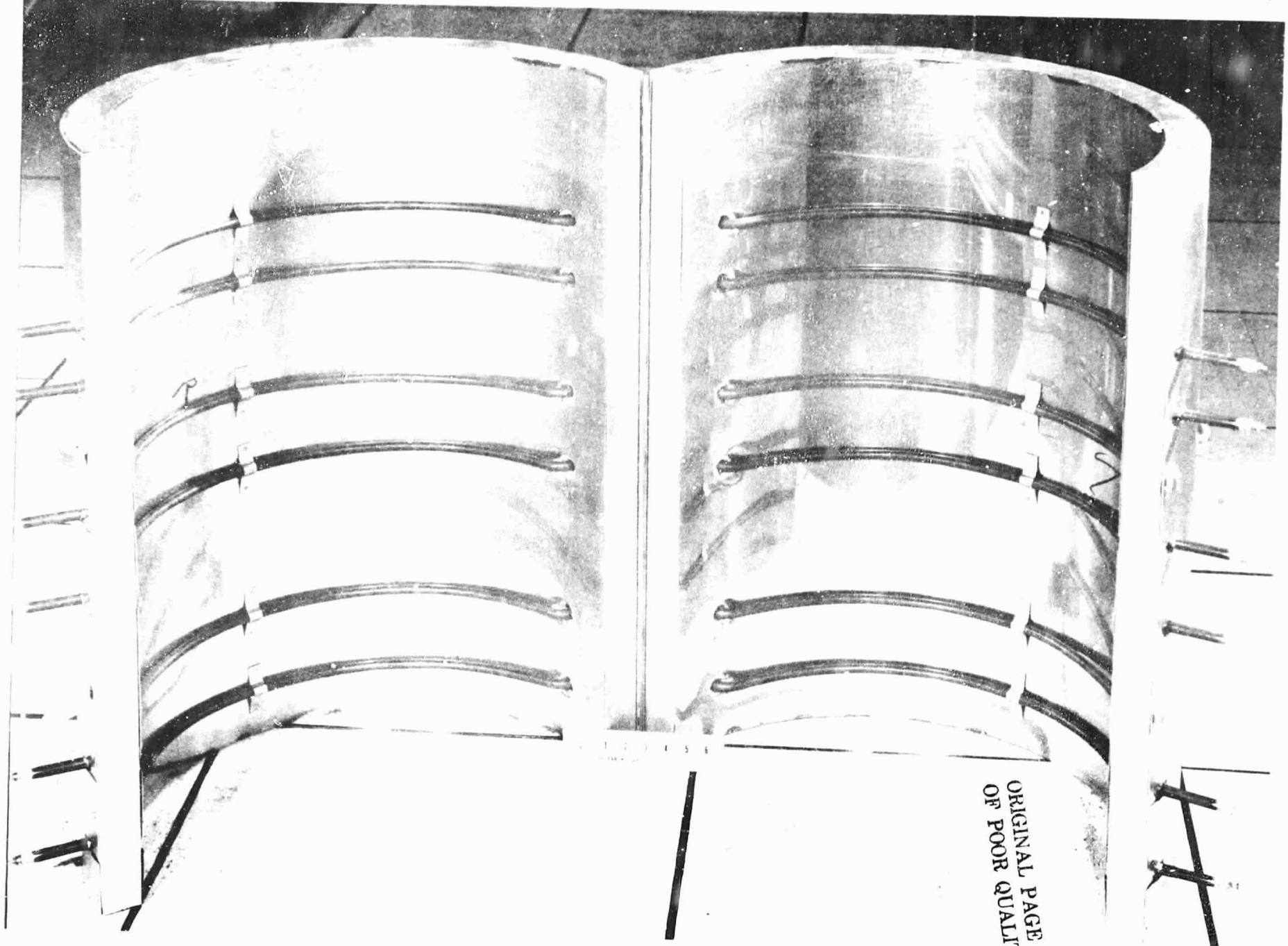

Figure 4.10 - Drum Heating Jacket In The Open Position

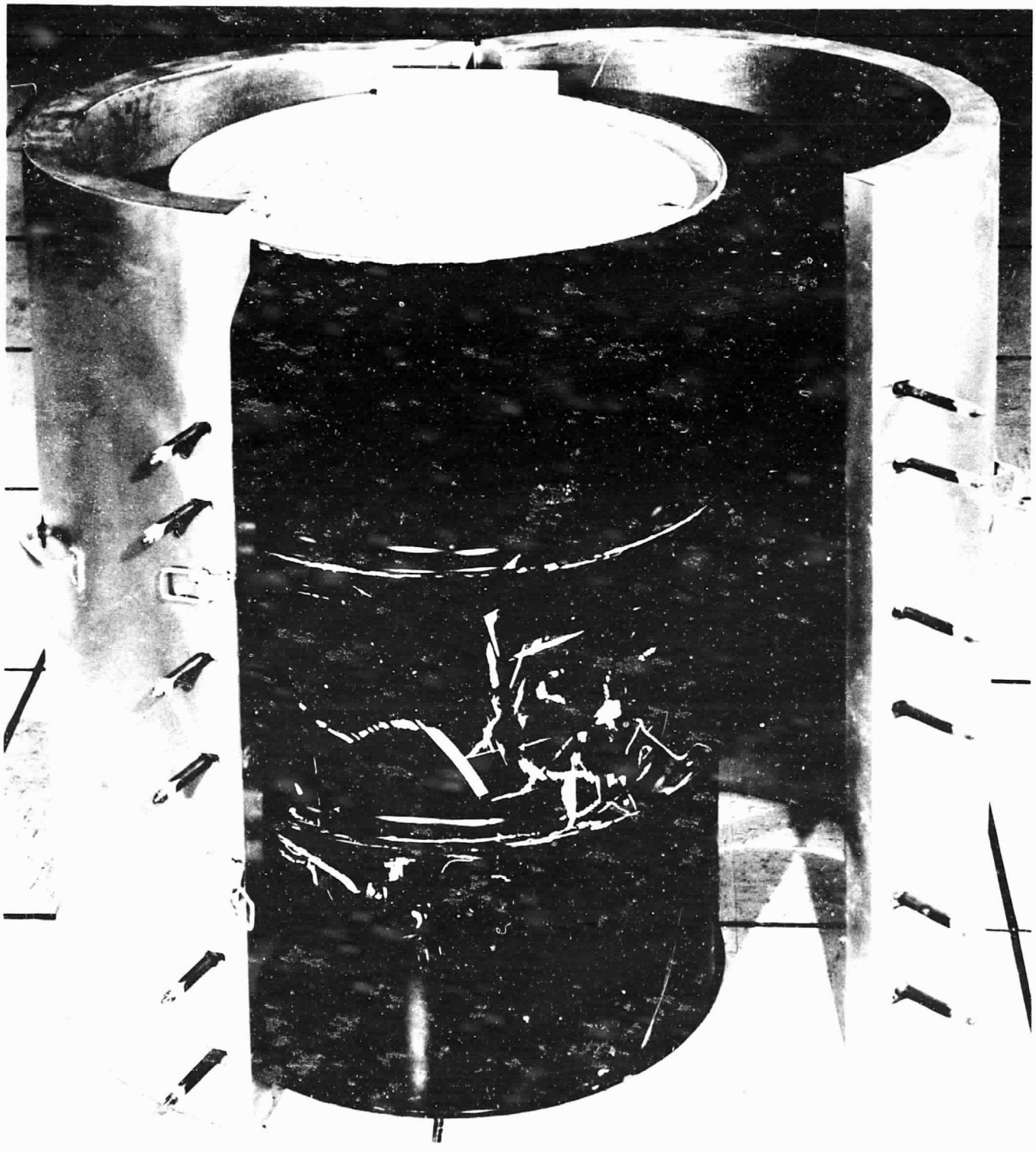

Figure 4.11 - Drum Heating Jacket Partially Around Drum Of Sodium

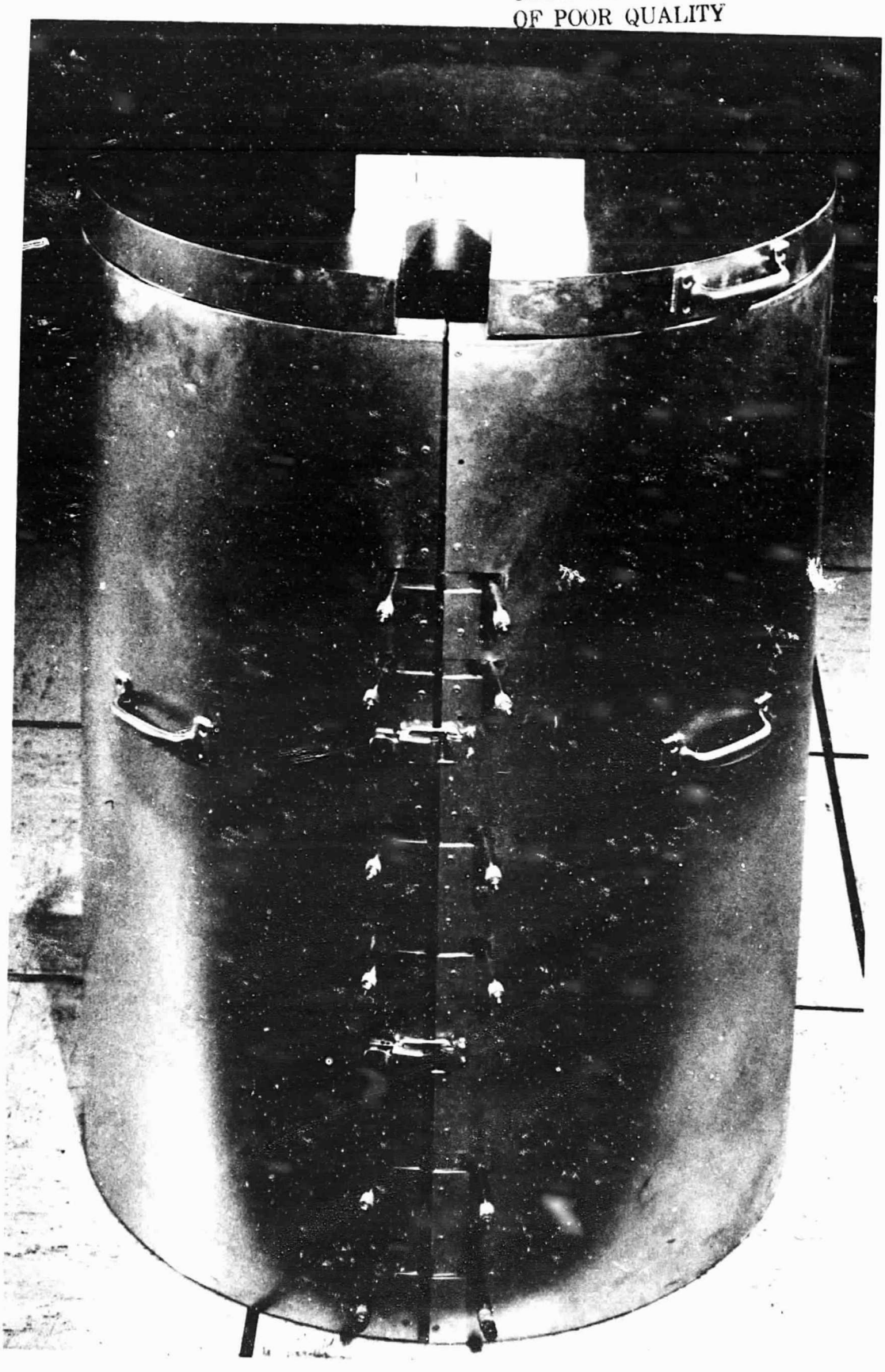

Figure 4.12 - Sodium Drum Heater Jacket With Heating Elements Installed

Figure 4.13 - Spring Loaded RTD Holders For Drum Heating Jacket Temperature Sensors

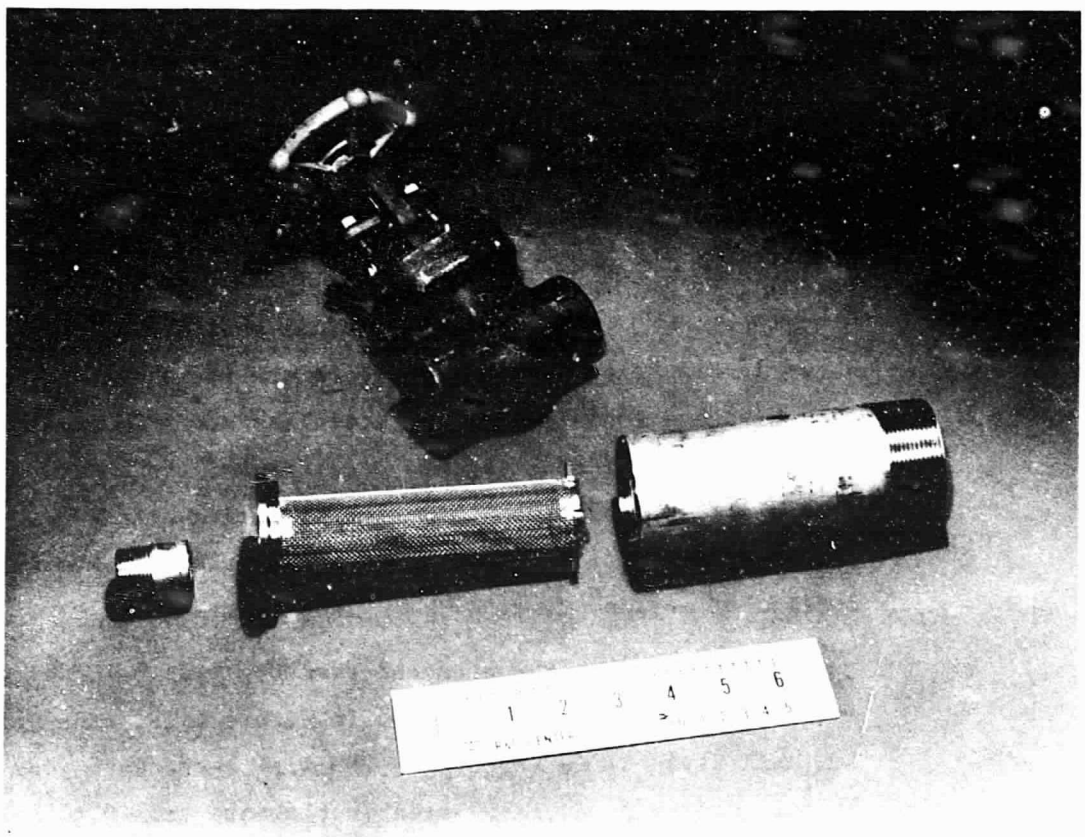

Figure 4.14 - Valve And Filter Subassembly For Sodium Drums

filter and valve assembly for draining the sodium. Eleven completed assemblies are shown in Figure 4.15. Eleven assemblies are required to allow time for sodium cleaning and vacuum bake out after removing the assembly from an empty sodium drum.

Figure 4.16 shows the sodium base tank assembly. The three pneumatically actuated drain valves can be seen at the top of the tank. The five sodium level probes are installed in the flange located at the right side of the tank. The tank is sloped about 5^{o} to permit complete draining of the sodium after use.

The two-20 micron pore size final sodium filters are shown in Figure 4.17. These filters are identical in construction and contain sintered stainless steel filter elements sealed in all welded stainless steel tanks. Each element is star shaped and provides an active filtering area of 2.5 ft^2. It is planned that only one of the elements will be on line at any one time.

Sodium flow to the chemical reactors will be provided by an electromagnetic pump pictured in Figure 4.18. The pump (an MSAR Style I) is rated at 3 GPM with a pressure rise of 85 psi and an infinitely variable flowrate from 0 to full flow. It has no moving parts and can be operated up to a maximum temperature of $1000^{o}F$. The NaK pump for the thermal control loop is identical in design.

The NaK sump tank is shown in Figure 4.19. This all welded stainless steel tank has an integral 15 micron sintered stainless steel filter housed in the pipe section extending vertically from the sump tank. The purpose of this filter is for purity control of the NaK. The three vertical pipes to the right of the filter are for installation of the NaK level probes.

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Figure 4.15 - Drum Drain Filter And Valve Assemblies, Fabrication Complete

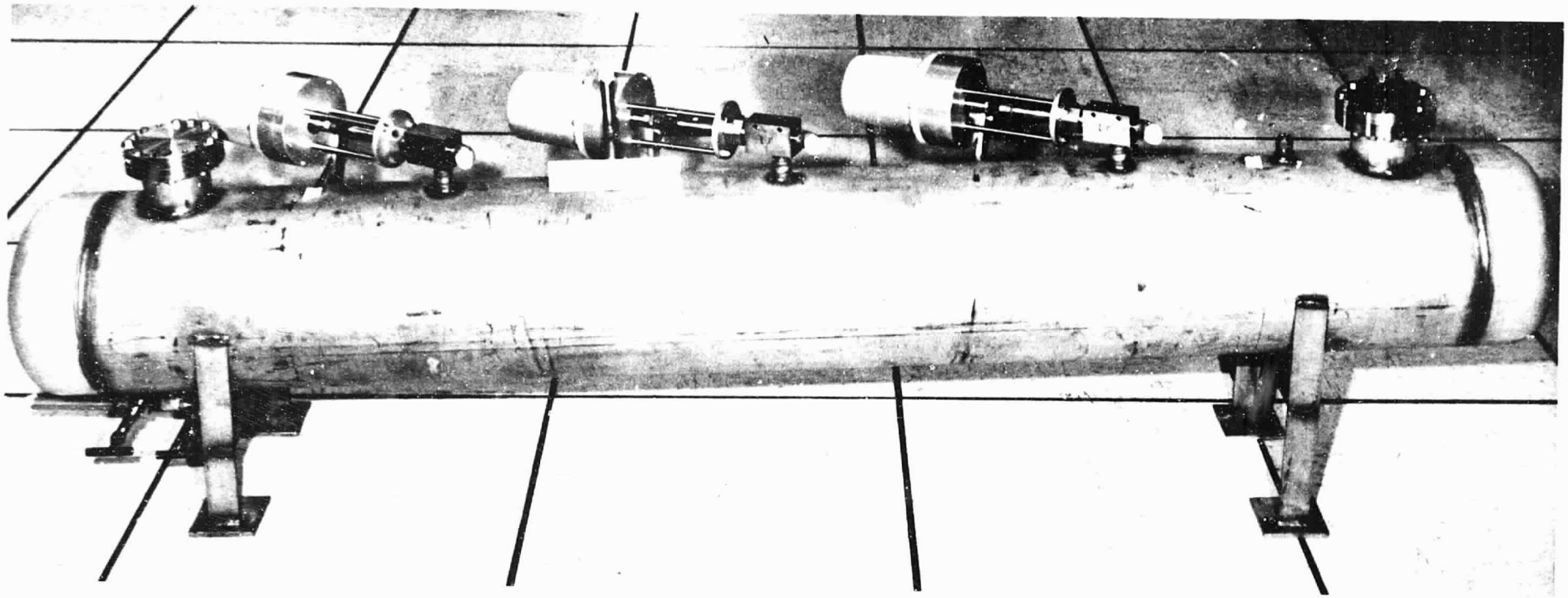


Figure 4.16 - Sodium Base Tank

Figure 4.17 - Final Filters For The Sodium Injection System

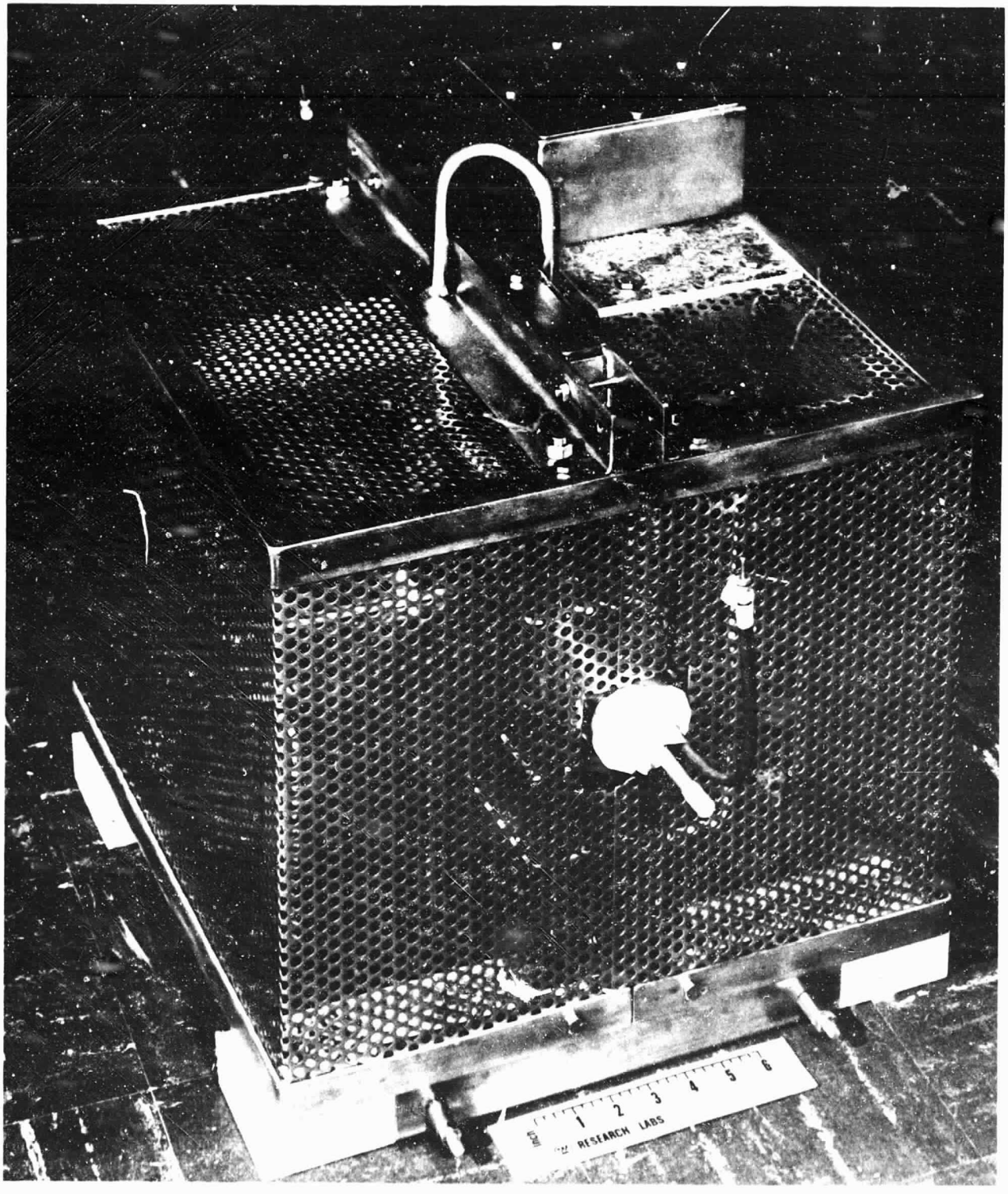


Figure 4.18 - Electromagnetic Sodium Pump

Figure 4.19 - NaK Sump Tank

4.2.5 Silicon Collector (I. E. Kanter)

During this period the insulation brick, ceramic felt and fused quartz crucibles have been received. Orders for the collection housing and the graphite internals have been placed.

Fabrication of the collection system components is in progress and a few courses of brick have been cut using the completed mock-up jig for the annulus, see Figure 4.20. The brick courses for the floor of the housing have also been cut.

In order to provide a less expensive collector during the initial shakedown tests (in place of the quartz curcibles), an expendable collector was developed. The quartz crucibles will be used for the final testing series. The expendable collector, as shown in Figure 4.21, replaces the graphite, ceramic felt and fused quartz components. The collector consists of a stainless steel can filled with a castable refractory similar to the insulation brick. A cavity is molded in the casting to provide for product collection. To date, the fabrication of the can and its associated lifting hardware is complete.

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

During the quarter, t crubber was ordered and design drawings were review returned to the vendo. specifying locations of the nozzles, flanges, and bolt circle sizes. A sketch of the gas scrubber is shown in Figure 4.22. The scrubber vendor has indicated that the delivery of the equipment may be delayed because of difficulty in procuring small quantities of Incoloy 825 which is the material of construction for the venturi and scrubber components. Interface with the vendor is continuing in an effort to resolve the procurement problem with the Incoloy material.

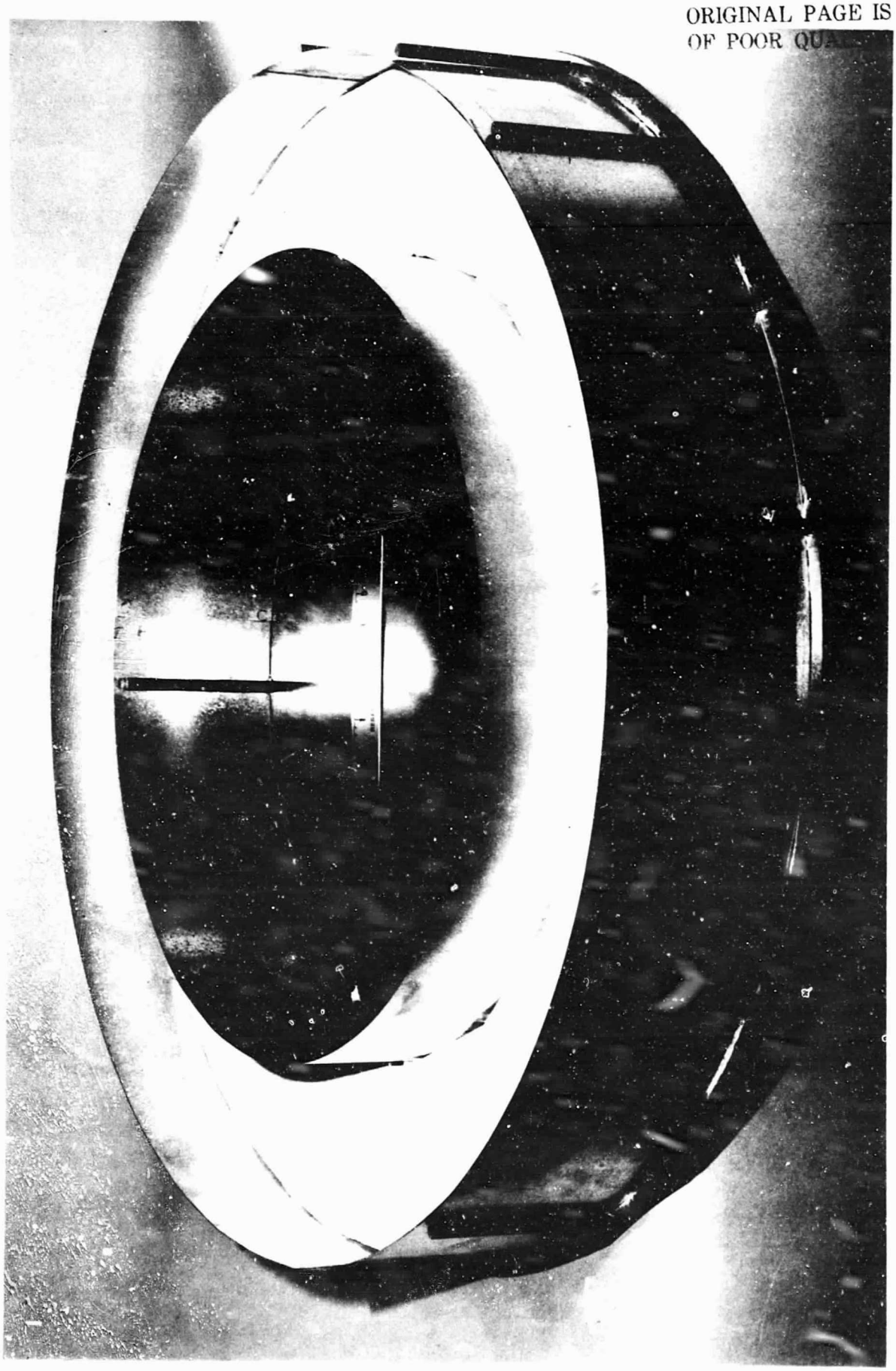


Figure 4.20 - Photograph Of The Mock Internal Assembly For Precutting Insulation Bricks

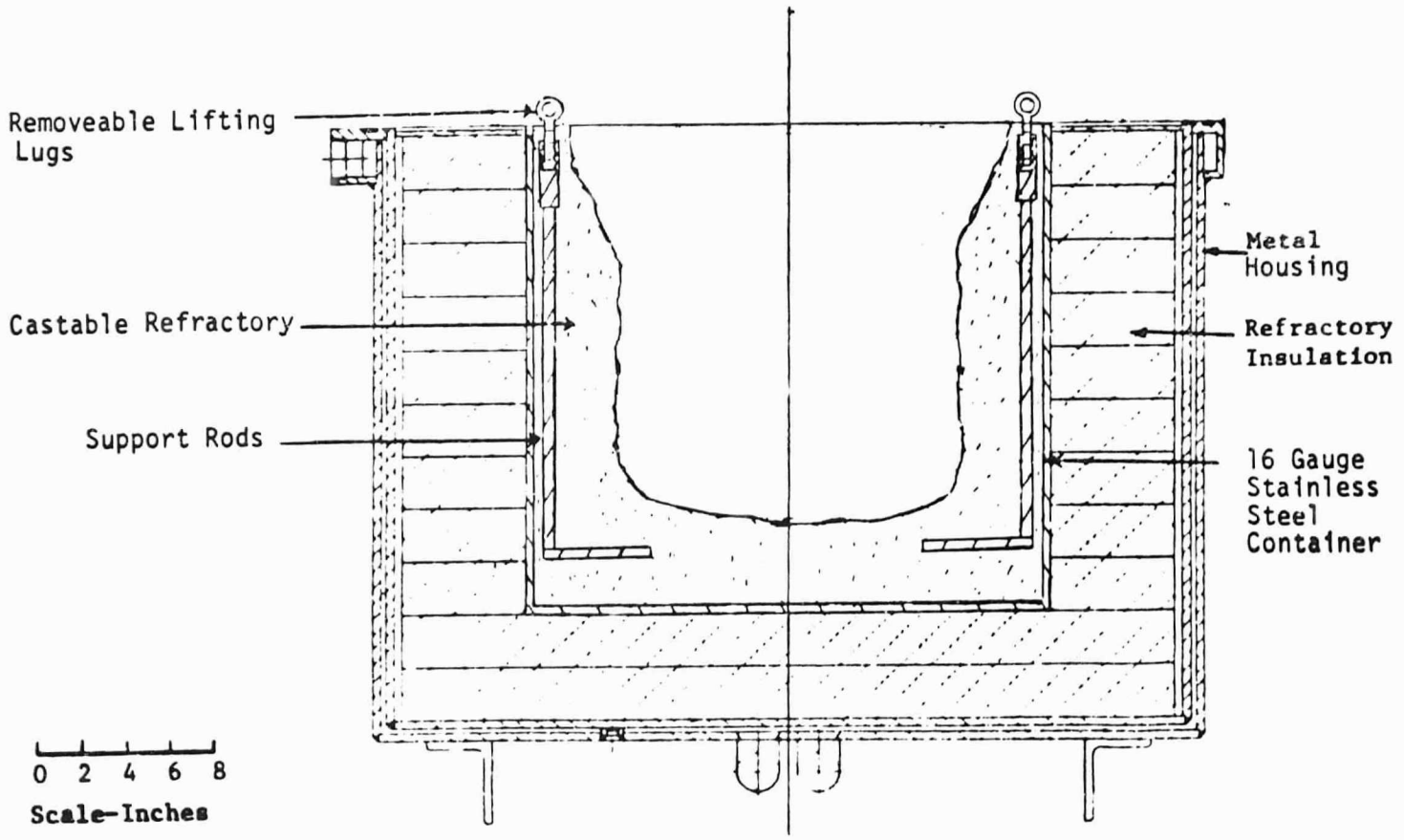


Figure 4.21 - Cross Sectional Drawing Of Expendable Silicon Collector For Shakedown Experiments

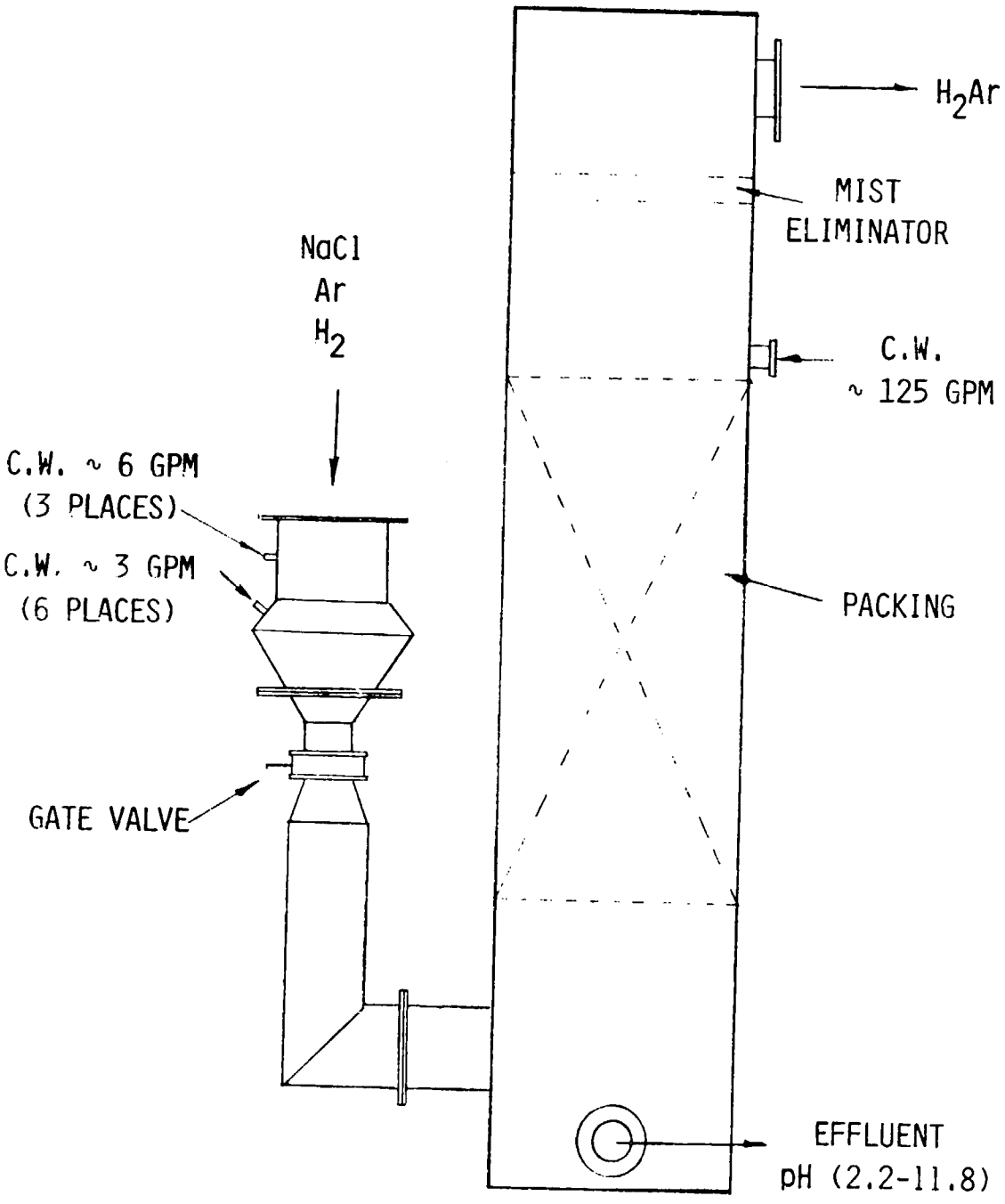


Figure 4.22 - Sketch Of The Gas Scrubber For The Effluent Handling System

The pH control instrumentation and chemical feed tanks with metering pumps have been ordered along with the water treatment tank. The pH of the effluent (water) will be monitored at the entrance of the treatment tank. Any pH adjustment will be made at the second cell of the tank and a final pH measurement will be made at the exit of the tank. A schematic of the water treatment system is shown in Figure 4.23.

During the next quarter, interface with the scrubber vendor will continue until the material procurement problem is resolved and the scrubber is received and installed. Also, the water treatment equipment is scheduled for delivery and installation.

4.3 Experiment - Laboratory Integration

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

With the completion of the electrical system drawings during the previous quarter, ordering of all equipment and materials for the system has been accomplished. To date, approximately 85% of these orders have been received. Expediting of the procurement effort was implemented in order to provide close order follow with the vendors.

For the electrical system, fabrication and installation of both the A.C. and D.C. bus and supports, the A.C. transfer switch supports, and also the potential and current transformer supports was accomplished. In addition, assembly and installation were completed for the cable trays, A.C. transfer switches, PT's and CT's, and the A.C. power cables. Effort is currently in progress for mounting and wiring the six electrical distribution panels, installing and connecting the D.C. power cables, and wiring of the heating and ventilation system.

It was also necessary to review the routing and usage of

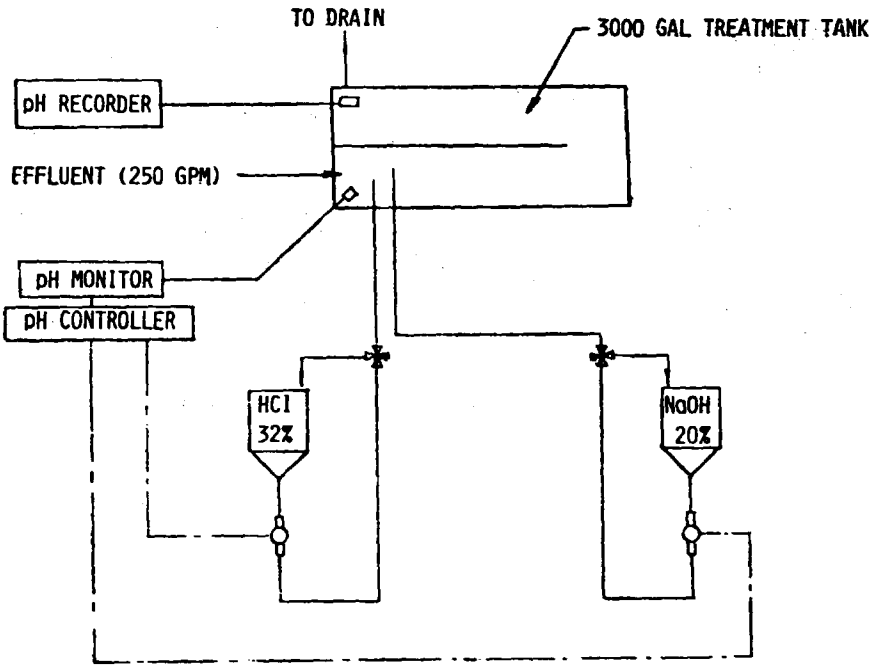


Figure 4.23 - Schematic Of The Water Treatment System

existing cables located in the overhead trays to determine which were needed and which could be rerouted to provide space for the new higher voltage power cables.

Installation and assembly will continue through the next report period in preparation for the testing program.

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

During this reporting period separate quotations were received for both the hydrogen and argon gas mixing system and the gas storage system. Based on price and delivery, Air Products was issued orders for both the mixing and storage equipment. Subsequently a meeting was held with Air Products to review and finalize the equipment specifications for the gas blend system. The basic schematic shown in the April-June quarterly[3] was followed with the addition of a second solenoid valve included at the hydrogen trailer and control room pressure indication via pneumatic transmitters.

A meeting was also held at the Arc Heater facility with an Air Product's field engineer to review the gas storage site. Based on the site and expected gas usage during various test sequences, tentative recommendations were made with final locations and site preparation requirements to follow after Air Products reviews available equipment. It was suggested that the argon be provided as a liquid in a tank approximately 6 feet in diameter by 14 feet high containing about 60,000 cubic feet of argon. The hydrogen will be supplied in tube trailers varying in size depending on the expected usage during a given time period. Each test run is expected to use about 15,000 cubic feet of hydrogen per hour with the heaviest monthly usage occurring in January for a total during that month of 315,000 cubic feet. The suggested trailer size for that period is 120,000 cubic feet with other months having a lower rate and a trailer size of 75,000 cubic feet.

After obtaining the latest gas system information, the gas equipment layout dra·ing was revised to incorporate this revised information and changes. An additional change to this drawing was the elimination of the nitrogen system. The nitrogen was to be used as a dry gas supply for the pneumatic instruments. However, due to the addition of more instrumentation and, therefore, increased gas consumption an air compressor with drier will be used. The compressor will supply a flowrate of 25 scfm, a maximum pressure of 150 psig and have a dew point of 35°F. An additional drier will be located outside of the building for lowering the dew point of the air used for pneumatic equipment exposed to ambients below 32°F.

4.3.3 Cooling Water System (C. B. Wolf)

Cooling Water System procurement has continued through this reporting period. Installation of procurred items was initiated and is presently nearing completion. Installation is being coordinated with the building modification effort. The high and low pressure pumps, surge tank, and demineralizer have been received and are installed. A schematic of the cooling water system is shown in Figure 4.24. The heat exchangers have not been received, as yet, but are scheduled for installation early in the next quarter. The heat exchanger support structure has been installed also. All water system manifolds are installed, and nearly all other connecting piping and valves are installed with the exception of the heat exchanger connecting piping. Included in the water system are piping and valves for the stack cooling water.

The water system includes provision for emergency cooling in the event of a loss of power to the pumps or a pressure loss in the main cooling water line supplying the heat exchangers. Emergency cooling water is provided by the plant

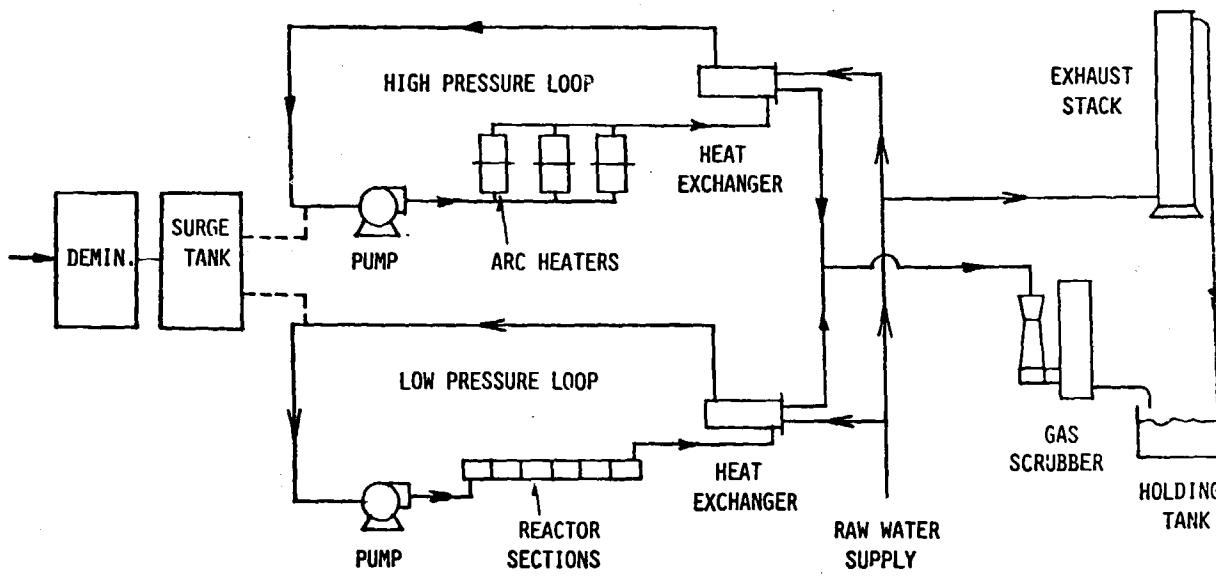


Figure 4.24 - Schematic Of The Cooling Water System

fire protection lines, which are separate from the service lines.

Water system installation will be finalized as soon as possible after receipt of the heat exchangers. Electrical wiring and instrument connections will proceed according to the installation schedules.

4.3.4 Gas Burnoff Stack (C. B. Wolf, J. W. George)

During this quarter the burnoff stack was ordered and has been received. Detailed engineering drawings of the stack support structure were completed. The structure includes provision for the replaceable waste water treatment tank located directly beneath the stack. The stack igniter and associated controls were ordered and received. Piping required for stack cooling water is included on the water system drawings.

The support structure has been installed and is complete except for the decking required at the base of the stack. The stack has been erected and braced by ties to the building framework.

Preliminary testing of the igniter and its controls are in progress. As described earlier, the gas burnoff stack igniter is a propane-fueled torch that is ignited by a spark type igniter (spark plug). The flame of the torch is monitored to insure ignition of the hydrogen. If a flame is not detected, then the spark igniter attempts to relight the torch. Should the torch fail to reignite, the propane gas is automatically shut off and an alarm is activated indicating a flame-out condition in the gas burnoff stack. The flame detection system will be a commercially available unit as will the torch ignition system. A schematic of this system is illustrated in Figure 4.25.

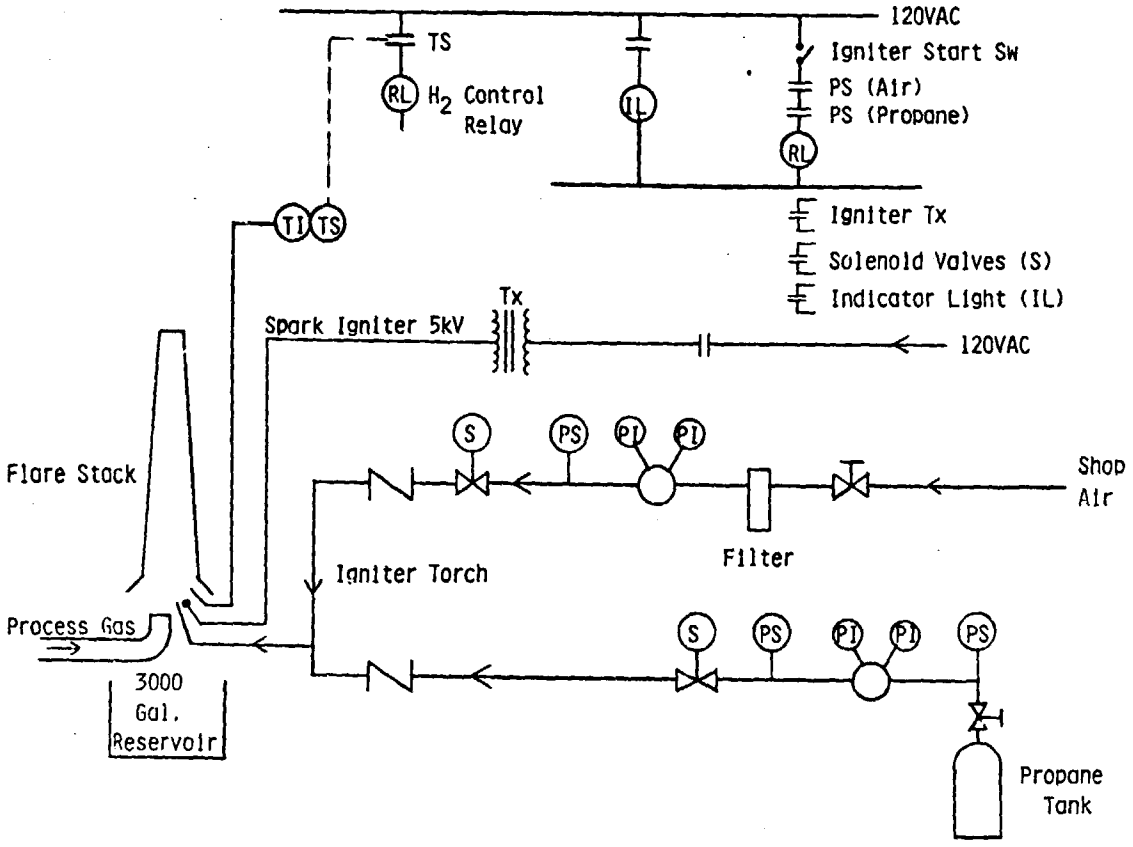


Figure 4.25 - Schematic Of The I&C Components For The Gas Stack Igniter

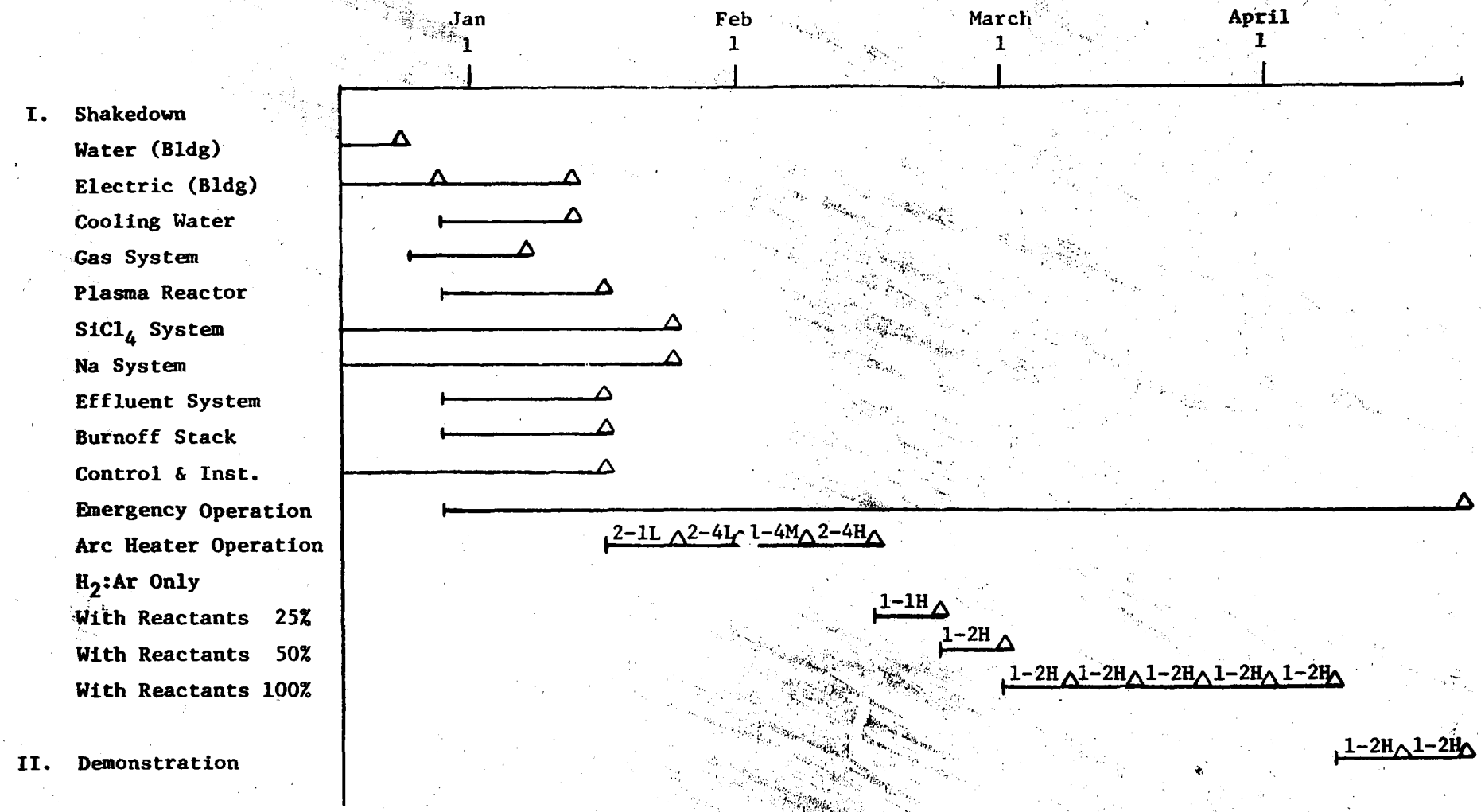
5. TESTING

5.1 Operating Procedures Manual (T. N. Meyer)

Preparation of the Plasma Reactor Operating Manual was initiated during the quarter. The purpose of this manual is to assemble critical information necessary for the successful performance of the testing program. The testing program is to result in operation of the reactor at a silicon production rate of 100 lb_m/hr. The data obtained from the operation and physical analysis of the silicon product will be utilized for final process evaluation. The manual necessarily includes nominal operating conditions with expected or estimated data readouts for immediate assessment of operation, identification of key physical parameters which are crucial to data analysis, and/or control, a detailed test plan for the four month testing program, start-up/shutdown and emergency operating parameters, and a procedure for data reduction and analysis.

A preliminary test plan indicating the testing to be performed during shakedown and demonstration tests is outlined in Table 5.1. The testing program is scheduled to begin on December 15 and end on April 20, 1979. The first 3 to 4 weeks will involve hardware checkout of the system operating under the arc heater electrical noise environment. In addition, an energy balance can be performed and thus provide diagnostics for instrumentation operating in this environment. The next three weeks will involve a continuous increase in operating level with two full power-four hour runs to be performed during the second week of February. These runs using no reactants will provide an evaluation of reactor wall heat losses to be compared with those predicted by the computer model. Thus, a determination of internal liner temperature can be obtained. It is intended to obtain operating characteristics for the arc heaters during these tests. Such characteristics will include thermal heating efficiency, arc voltage as functions of arc current, magnetic field current and the hydrogen-argon gas composition and flow rate.

Table 5.1 - Testing Schedule



*2-1L indicates two one-hour runs at low power. (M, H represent medium and high power.)

During the third week of February, testing with reactant injection will begin with 25% of nominal reactant flow rate (i.e., see Table 5.2 for nominal values corresponding to 100 lb_m/hr of silicon production). This test will provide an initial verification of the reactant subsystem operation, injection means, and permit a determination of potential reactor operating problems. On March 1, test will be run at 100% nominal reactant flow rate. Although these tests are all indicated to be two hour runs, an extra long run may be required due to a possible liner replacement. The collector can contain 200 lb_m of silicon (i.e., 2 hour run under nominal steady state conditions). However, an estimate of 4 to 6 hours of running is required for the skull wall buildup. During this final series of tests, the shakedown phase, a number of partial disassemblies will be required to assess the reactor condition (e.g., skull, scrubber, nozzle). The testing during the last two weeks (demonstration tests) will include two runs under steady state conditions to provide JPL with two representative silicon product samples.

5.2 Safety Manual (W. H. Reed, J. McDonald)

Preparation of the Safety Manual was initiated during the quarter with the compilation of pertinent procedures for safety and handling of chemical materials planned for use during testing. Various information sources were consulted for this safety data. A representative sample of these references is as follows:

a) Westinghouse Safe Practice Data Sheets

b) Chemical Safety Data Sheets published by the Manufacturing Chemist Assoc. (MCA)

c) OSHA - General Industry Safety and Health Standards

d) Westinghouse Accident Prevention Manual

Table 5.2 - Nominal Operating Conditions

Arc Heater:	Flow Rate Of Hydrogen	141 scfm, 2.98 moles/s, 48 lb_m/hr
	Flow Rate Of Argon	35.5 scfm, 0.747 moles/s, 236 lb_m/hr
	3Ø Electrical Power	1.76 MW
	Estimated Arc Voltage	890 Volts
	Estimated Arc Current	660 Amperes
	D.C. Field Coil Current	870 Amperes
Sodium Injection:	Flow Rate Of Sodium	0.66 gpm, 150 kg/hr
	Flow Rate Of Argon	49.2 scfm, 150 kg/hr
Silicon Tetrachloride Injection:	Flow Rate Of Silicon Tetrachloride	0.81 gpm, 275 kg/hr

The following is a tentative outline that was assembled for the safety manual:

Title: "A Guide For Safety In The Arc Heater-Silicon Laboratory"

1. Introduction
   - 1.1 Need for safe practices and purpose of guide
   - 1.2 Westinghouse concern about safety

2. General Laboratory Safety
   - 2.1 Basic rules

3. Process Description for the Production of Solar Grade Silicon

4. The handling and use of process chemicals
   - 4.1 Sodium
   - 4.2 Silicon tetrachloride
   - 4.3 Hydrogen
   - 4.4 Argon
   - 4.5 Hydrochloric acid
   - 4.6 Sodium hydroxide

5. Personal protective equipment
   - 5.1 Respirators
   - 5.2 Leather coats and leggings
   - 5.3 Coveralls
   - 5.4 Breathing unit
   - 5.5 Black vinyl overshoes
   - 5.6 Vinyl-acid wear suits
   - 5.7 Hard hat and face shield
   - 5.8 Goggles

6. Safety in laboratory operation and maintenance
   - 6.1 Silicon tetrachloride system
   - 6.2 Sodium system
   - 6.3 Arc heaters and reactor
   - 6.4 Effluent disposal system
   - 6.5 Electrical equipment
   - 6.6 Instrumentation

7. Emergency procedures
   - 7.1 Sodium spill or leakage
   - 7.2 Silicon tetrachloride spill or leakage
   - 7.3 Fire and fire extinguishing equipment
   - 7.4 Emergency System Shutdown

This manual contains the pertinent safety information, procedures, and appropriate references required for personnel working at the Arc Heater Laboratory during the upcoming experimental verification testing for silicon production. Copies of this manual will be available to all test personnel, maintenance personnel, and visitors in addition to forming the basis for safety training.

6. INJECTION TECHNIQUES

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

6.1 Summary

Detailed characterization of the Sonicore sodium injection nozzle using water as the test fluid has been completed. Results indicate that flow rates of 45 gph sodium and 50 scfm argon should produce sufficiently small droplet sizes. Verification of the nozzle operation with liquid sodium is almost complete. The nozzle functioned properly and showed no tendency to plug. Sieving of sprayed sodium particles is in progress. The silicon tetrachloride feed system is being constructed and it is planned to commence testing of the hydraulic spray nozzle within the next few weeks.

Final assessment of the sodium nozzle particle size data will be made and post-test inspection of the nozzle will be performed. The significant results are being relayed to the personnel responsible for assembling the sodium system for the demonstration reactor. Qualitative testing of the silicon tetrachloride injection nozzle will be initiated.

6.2 Work Description

6.2.1 Nozzle Characterization

The leading candidate nozzle for liquid sodium injection is the Sonicore 312T, with the Sprayco being regarded as a back-up, should unforeseen difficulties arise. With the majority of the testing complete, the Sonicore appears to be suitable for application in the plasma reactor. A considerable amount of data has been collected concerning the particle size produced by these two nozzles using water as the test fluid. This data was obtained using laser imaging spectrometers supplied by the Particle Measuring Systems Company (PMS) and described below.

The basic principal of operation of these instruments can be described with the aid of Figure 6.1. A collimated laser beam illuminates a particle in the vicinity of the object plane of an imaging system. The shadow of the particle is projected at a certain magnification and moves across a linear photodiode array. The shadow of a particle is related to the particle size by the magnification.

Two types of processing electronics are used in conjunction with these arrays. The Optical Array Spectrometer simply determines the number of elements shadowed by the particle and thus provides a particle diameter measurement (1-D Spectrometer). The second type obtains full two-dimensional shadow images of particles (2-D Spectrometer) by simultaneously sampling the output of each photodiode element in the array at a very fast rate to provide sequential image slices as the shadow transits the array.[4]

For testing, both the 1-D and 2-D Spectrometers were used, covering the particle size ranges 20-300μ and 25-600μ, respectively.

To a first approximation, the sample cross section in these Optical Array Spectrometers is the product of the array width and the particle depth-of-field. In the case of the 1-D Spectrometer, end elements of the array are used to reject partial shadow conditions which would result in undersizing. In the case of the 2-D Spectrometer, partial shadows are handled through software in the analysis of the image data. To complete the sample cross section definition one needs only to define the depth-of-field. If the illuminated regions are well collimated, the depth-of-field is limited only by diffraction-induced divergence of the shadow region behind the particle which causes gradual filling and fading of the shadow boundaries.[5] For present discussions, one needs only to recognize that this divergence leads to

OPTICAL ARRAY SPECTROMETER

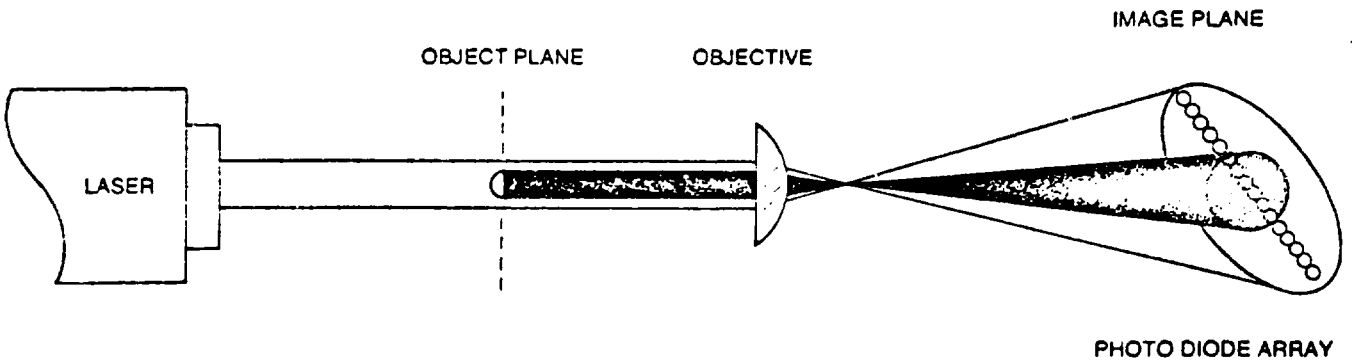


Figure 6.1 - Operating Principle Of The PMS Laser Spectrometer

inaccurate measurements at large depths-of-field and that the available depth-of-field where accurate measurements can be made varies as the square of the particle radius. A particle of 20 μm diameter has a 1 mm depth-of-field, while a particle several hundred microns diameter may have up to a meter depth-of-field. In an instrument sizing these larger particles, the depth-of-field is invariably truncated by the mechanical limits of the sampling aperture. However, with sizing small particles the depth-of-field must be truncated by other than mechanical means if a large percentage of the particles are to be sampled in situ. The method most commonly used to provide optical depth-of-field truncation is through the use of multiple thresholding in the processing of the shadow signals from each photodetector element. One threshold is obviously required for sizing and a second for depth-of-field limiting. For accurate particle sizing, the appropriate shadow threshold is approximately 50% (roughly the amount equivalent to the noise band). If maximum depth-of-field is desired a setting of about 60% is used. If a shorter depth-of-field is desired settings of up to 80% are used.

In the 1-D Spectrometers, depth-of-field calibration is performed over the size range through actual measurements. It can be calibrated to better than 10% for spherical particles.

When the 2-D Spectrometer is applied to measurements of small particle size, the method just described can be used or multiple shadow density levels can be simultaneously measured and displayed in the imagery to provide a grey-scale which contains the needed depth-of-field information. This again can be processed by software. The depth-of-field can be more accurately specified since particle morphology information is also available.

The range of tests run at PMS are outlined in Table 6.1.

TABLE 6.1

Tests Performed at PMS

SONICORE NOZZLE (Model 312T)

Run No.	Liquid Flow (GPM)	Mass Ratio (Air / Liq.)	Spectrometer
1	0.75	0.29	1D
2	0.75	0.22	1D
3	0.75	0.19	1D
4	0.75	0.14	1D
5	0.75	0.33	1D
6	0.75	0.28	2D
7	0.75	0.22	2D
8	0.75	0.19	2D
9 (without cup)	0.75	0.31	2D

SPRAYCO NOZZLE (Model 49267650)

10 (6.35 mm)	0.7	0.30	2D
11 "	0.4	0.46	2D
12 "	0.3	0.59	2D
13 "	0.7	0.24	2D
14 (6.50 mm)	0.7	0.26	1D
15 "	0.55	0.31	1D
16 (5.98 mm)	0.75	0.24	1D
17 "	0.45	0.39	1D

A total of 17 tests were made for the two candidate sodium nozzles at varying water flowrates and atomizing gas to liquid ratios. The Sprayco nozzle (Model 49267650) has an adjustable outlet which controls spray pattern and particle size - the distances in parentheses are a measure of this adjustment.

The results printed out by the computer are in three different forms. First, a simple mass distribution, Figure 6.2, which plots the percentage of the total liquid mass which is present as particles of a particular size. The size axis is divided into 15 channels each with a 20μ span and covering the total range 20-300μ. Figure 6.2 is an example from run No. 1 and shows that the distribution varied from 1.8% within the 20μ channel to a maximum of 8.9% within the 120μ channel.

The second type of plot obtained is a cumulative mass distribution, as presented in Figure 6.3. This example is also for run No. 1 and shows that 27.6% of the total mass appeared as particles less than 100μ in diameter, 70.5% less than 200μ and so on. The third form in which the results are presented is shown in Table 6.2, which gives not only the precise data from which Figures 6.1 and 6.2 were compiled but also the number count of the particles. This clearly shows that the vast majority of the spray particles are in the 20-120μ range but that the cube dependence for converting radius to mass, shifts the mass distribution to higher sizes.

For each of the 17 nozzle tests, at least 3 or 4 sets of data (i.e., 2 curves and 1 table) have been obtained. Reduction of the laser imaging data, particularly the 2-D, involves a complex computer program especially written by PMS for this series of tests, and there are still some differences between the 1-D and 2-D results which remain to be rationalized. Therefore, the results presented here are

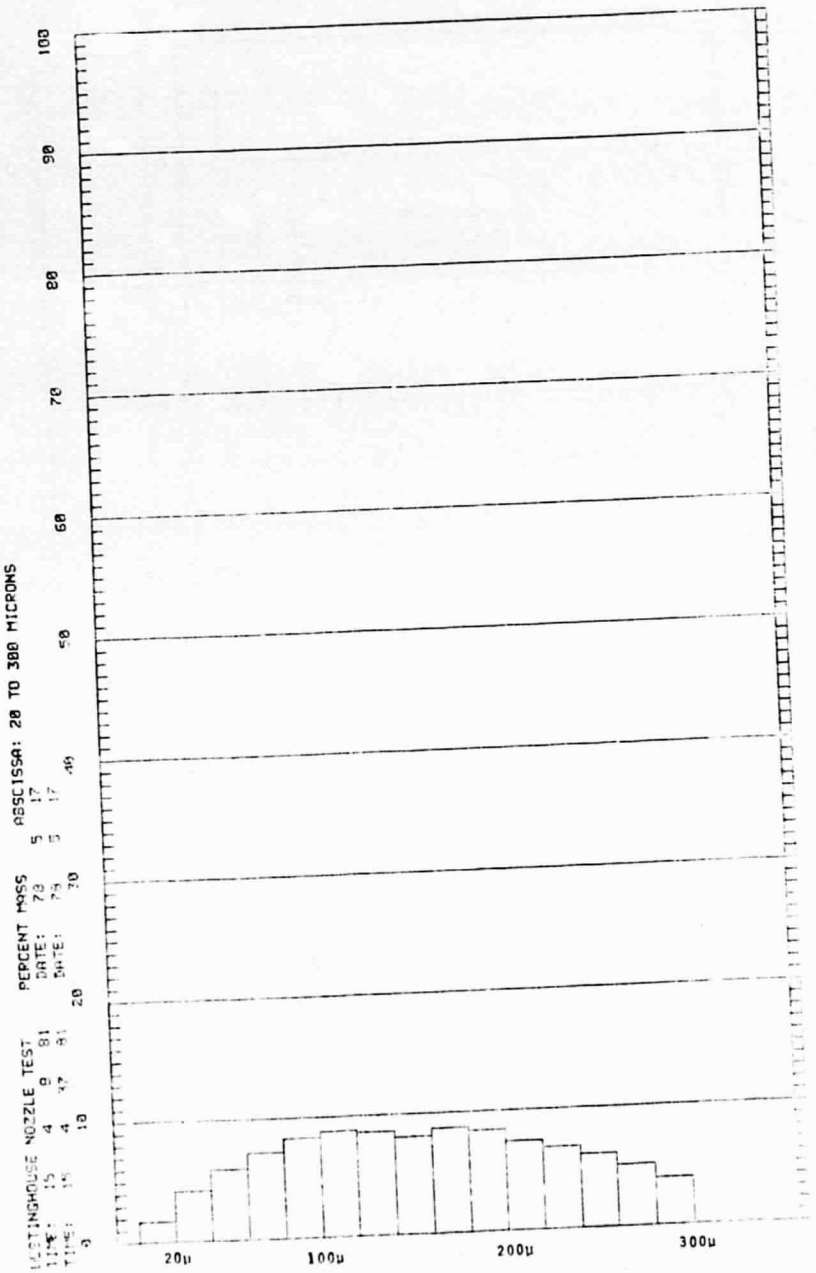


Figure 6.2 - Mass Distribution Plot From Run #1 At PMS

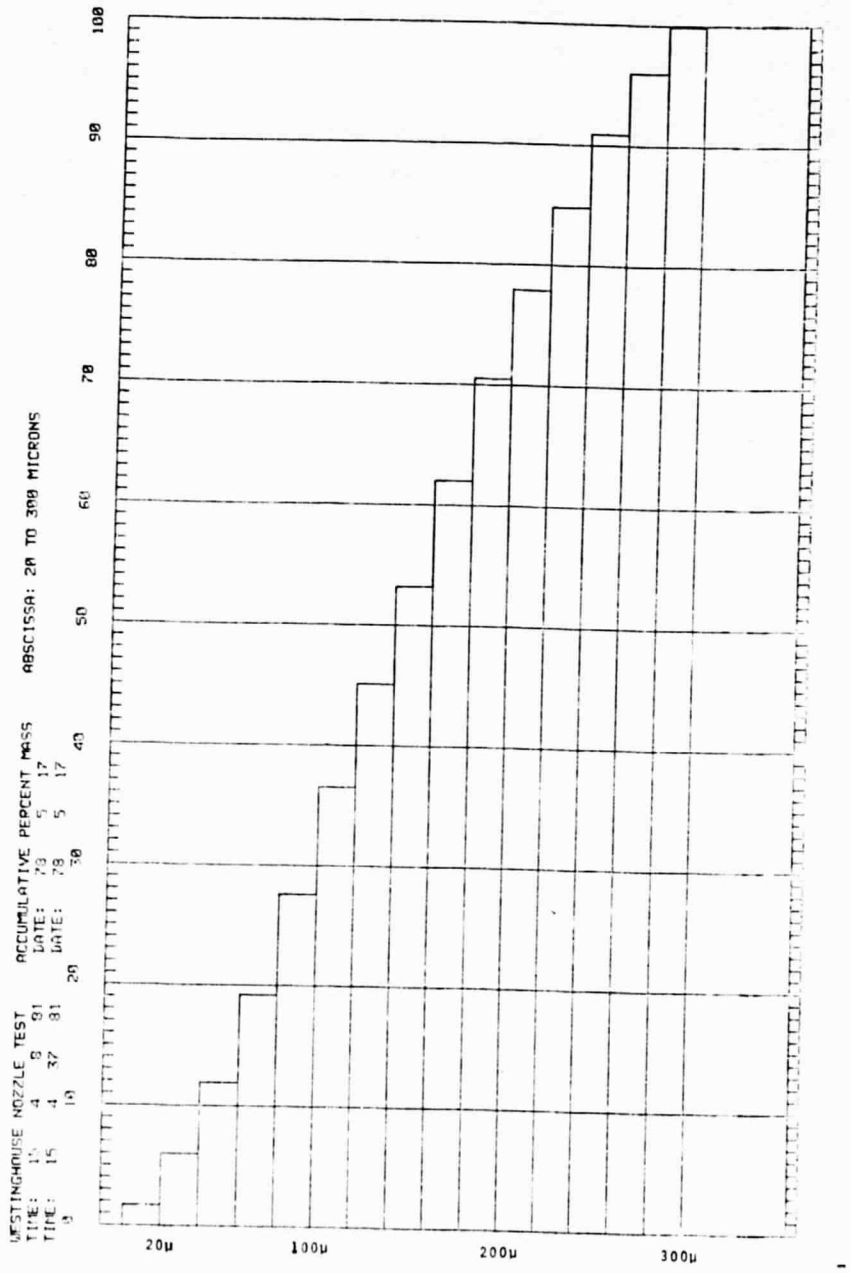


Figure 6.3 - Cumulative Mass Distribution From Run #1 At PMS

TABLE 6.2 - Test Data From Run #1 At PMS

TIME: 15 4 8 81 DATE: 78 5 17
TIME: 15 4 37 81 DATE: 78 5 17

I	NUMBER	MASS	PM	AM
1	83933.820	0.000615160	1.794	1.794
2	32985.000	0.001461201	4.262	6.056
3	14994.806	0.002029359	5.919	11.974
4	8139.161	0.002481069	7.236	19.210
5	4987.630	0.002878690	8.396	27.606
6	3130.832	0.003050526	8.897	36.503
7	1942.993	0.002950051	8.604	45.107
8	1254.098	0.002807287	8.187	53.294
9	939.513	0.003009840	8.778	62.072
10	664.801	0.002899031	8.455	70.527
11	449.752	0.002559142	7.464	77.991
12	318.322	0.002335917	6.813	84.804
13	226.434	0.002103123	6.134	90.937
14	153.119	0.001769409	5.160	96.098
15	94.454	0.001337993	3.902	100.000

I = Channel No.

PM = % Mass

AM = Accumulative Mass

preliminary in nature and will be subject to further refinement, but one or two general points can be made. The most striking, though not unexpected, feature of the results is that, in general, smaller particles are obtained for both the Sonicore and Sprayco nozzles when the air:water mass ratio is increased. This finding is in agreement with previous studies of gas atomization of fluids.[6] A conservative extrapolation of the mass distribution versus gas:liquid ratio indicates that all the spray particles should be below at least 200μ if a ratio of 1:1 can be achieved.

A second major conclusion is that the particle size produced by the Sprayco nozzle was found to be highly dependent on the setting of the outlet adjustments.

A direct comparison between the Sonicore and Sprayco nozzles under similiar flow conditions indicates that the former was slightly superior (i.e., produced smaller particles). When the Sprayco nozzle adjustment is optimized, however, it should provide an adequate back-up for the plasma reactor, particularly if the need is found for a narrower spray cone angle.

Discussions with the plasma reactor design engineers have indicated that gas:liquid mass ratios approaching 1:1 should not unduly upset the heat balance in the plasma reactor. Figure 6.4 shows the optimum nozzle flow and pressure readings for the Sonicore nozzle. At the required liquid sodium flowrate of 45 gph, an argon flowrate of 50 scfm is indicated. This flow corresponds to a gas:liquid mass ratio of 0.8 and has been used throughout the sodium test injections reported below. Unless future difficulties are identified, this flow ratio appears to offer a good compromise between small particle size and minimum inert gas dilution.

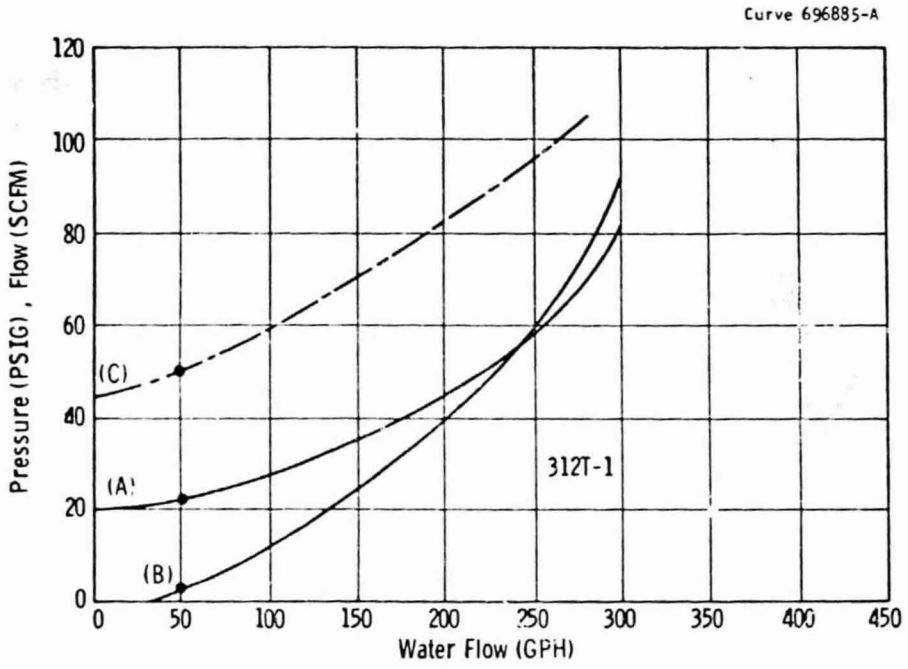


Figure 6.4 - Operational Parameters For Sonicore 312T Nozzle. Water Flow vs. Water Pressure (A), Air Pressure (B), Air Flow (C)

6.2.2 Sodium Testing

Having determined the essential operating characteristics and parameters of the Sonicore nozzle, it remained to verify that the nozzle could be successfully used to spray liquid sodium. A series of test sodium injections have been made and a number of different methods and modifications have been tried in order to determine the particle size produced. Although, as described below, it has not proved possible to collect such comprehensive particle size data as in the laser imaging experiments (section 6.2.1), the major conclusions of those tests have been confirmed. More importantly, considerable experience has been gained concerning the general operation of the nozzle using liquid sodium.

Initially, three shakedown tests were performed to optimize injection conditions. Each was performed at the proposed reactor condition of 50 scfm argon, but the sodium flowrate of 45 gph had to be arrived at by experimentation.

The first two sodium test injections were performed with a pressure of 50 psig on the liquid metal, and gave rise to sodium flowrates of 228 and 138 gph. The first injection was necessarily performed with the line from the pneumatic valve to the nozzle empty. This gave rise to a higher flowrate per unit pressure than in subsequent tests when the line was filled with sodium up to the nozzle. The third test injection, however, was run at 19 psig and gave a sodium flow of $\sim$ 41 gph. The sodium flow profile for test number 2 is shown in Figure 6.5. The electromagnetic flowmeter gives an output in millivolts which was plotted against time for the duration of the 2 second injection. The flow profile exhibits a sharp turn-on and a small initial peak extending for $\sim$ 1/10 second, followed by a gradual drift from 7.5 mV to 5.7 mV before a fairly sharp cut-off. Such a profile enables an accurate calculation of the sodium flowrate, using calibration tables supplied by

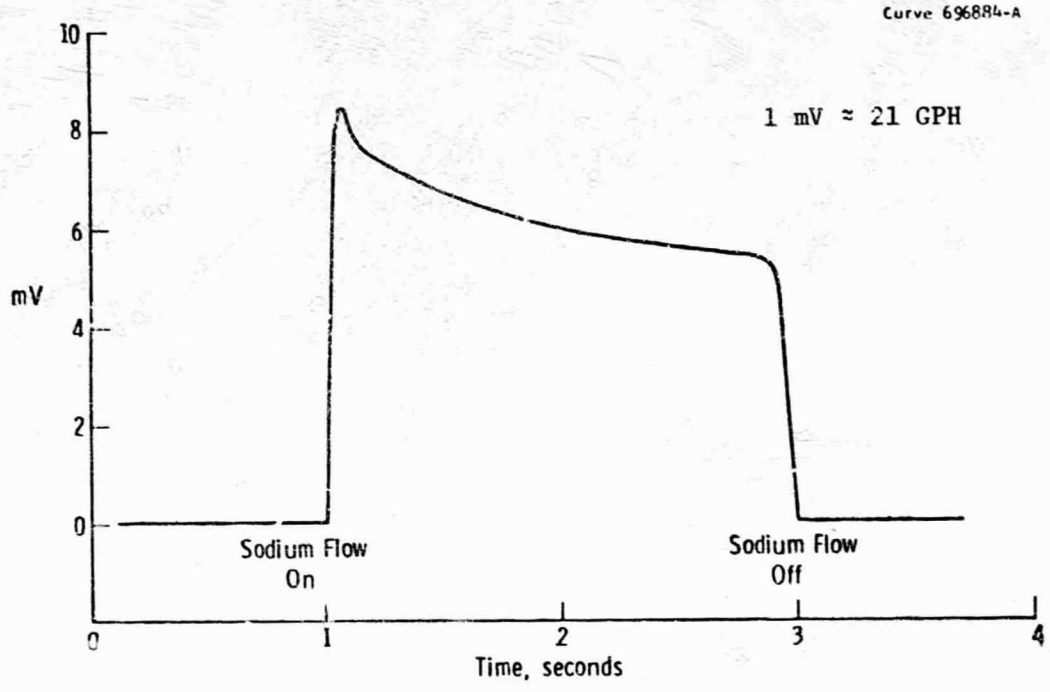


Figure 6.5 - Sodium Flow Meter Output vs. Time - Injection No. 2

the vendor. This profile was achieved, however, using a static head of sodium cover gas pressure, thereby explaining the slight drift in signal over the duration of the 2 second injection. Subsequent tests used a constant over-pressure to eliminate this effect.

Other experimental features which were optimized during these initial tests were the temperatures of the sodium and gas lines, heaters and nozzle. The nozzle appeared, visually, to operate correctly, the third test (41 gph, 50 scfm) producing what appeared to be a particularly fine sodium mist.

During testing, the lower section of the chamber was cooled with flowing liquid nitrogen to facilitate rapid solidification of the sodium particles. Due to the excessively high sodium flowrates in Tests 1 and 2, however, a considerable amount of molten metal hit the chamber walls and adhered. Some particles, however, were successfully collected at the base of the chamber and subsequently removed to an inert atmosphere glove box. These particles were successfully sieved to produce samples in the size ranges 200-400μ, 100-200μ and < 100μ. Microscopic examination showed these samples to consist of lustrous, clean sodium spheres.

During subsequent tests, a number of experimental difficulties arose requiring modification of the chamber. The shakedown tests indicated the need for a blow-down line to remove liquid sodium from the nozzle after each injection. This was designed to stop the tendency for post-test dripping and also facilitate decontamination of the apparatus. In addition, a modified sodium particle collection funnel was fabricated. This funnel was designed to cover the entire diameter of the chamber in order to catch all the particles produced by the nozzle. It also incorporated a small stainless steel cup suspended directly below the nozzle to catch post-test

drips. While these alterations were being made, the opportunity was also taken to connect the sodium reservoir to the reactant supply system of the Kinetics Studies. Also, the large capacity filter (300 scfm) was installed on the roof of the building and connected by pipe to the injection test chamber. This filter is intended for the long duration sodium and coinjection tests and also in the event of an emergency pressure build up.

When testing was resumed, these modifications worked well with the exception of the particle collection system. Repeated difficulty was experienced in collecting the sprayed particles, which, even in the solid state, were so clean and lustrous that these adhered to the chamber walls. One method that was tried in order to overcome this problem was to partially oxidize the particles as formed so that the outer surface would be sodium oxide, Na_2O, rather than clean, metallic sodium. This was achieved by introducing ~ 10% oxygen into the atomizing gas. The resultant spray particles were indeed more powdery in appearance and consequently easier to collect. It is not felt that the reaction between the atomizing gas and the liquid sodium will have materially affected the particle size produced in the spray.

At the moment, particles are being sieved and the size distribution calculated. These results will be presented and compared with the water data in the next report.

Attempts to record the sodium injections using high speed (5000 frames/sec) photography have been hampered by the difficulty in introducing sufficient light into the chamber. The photographs obtained were too underexposed to be of significant value.

6.2.3 Silicon Tetrachloride Injection

Work is in progress on the assembly of a silicon

tetrachloride supply and pump system for testing of the hydraulic spray nozzle. The chosen nozzle, manufactured by Spraying Systems Co., must operate at ∿ 9 gph and requires a liquid pressure of ∿ 100 psi for most efficient atomization. The particle size distribution produced by this nozzle with water as the operating fluid is well established and is shown in Figure 6.6. The laboratory tests to be conducted with this nozzle will not attempt to determine particle size, but will simply demonstrate the feasibility of producing a fine mist under reactor conditions.

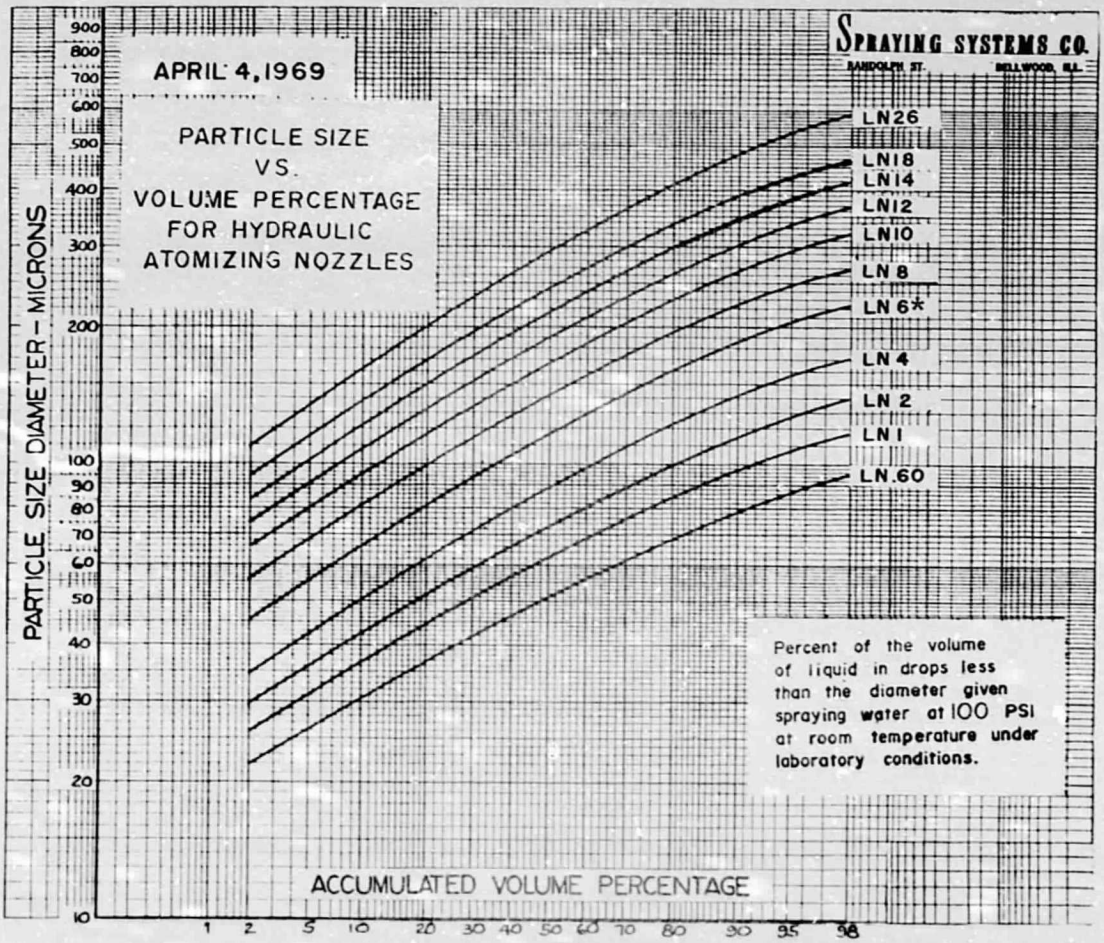


Figure 6.6 - Hydraulic Atomizing Nozzle (Model LN6*) For $SiCl_4$ Injection, Particle Distribution Curve (Curve Supplied By Spraying Systems Co.)

7. KINETICS STUDY

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

7.1 Summary

The reactant injection systems were incorporated into the test apparatus. Full power tests were run with different hydrogen-argon flowrates and $SiCl_4$ injection. Silicon was produced by the hydrogen reduction of $SiCl_4$. The injection flange was modified in order to extend the range of operating parameters. Experiments with Na-injection are being prepared.

7.2 Work Description

7.2.1 Test System

Assembly of the test system was completed by incorporating the reactant injection systems into the main apparatus. A photograph of the test chamber is shown in Figure 7.1. The plasma torch is seen at the left. Above it is a valve for controlling the flowrate of cold argon into the center part of the test chamber. The four front windows of the first two test sections have regular window flanges; the last test section has plexiglass windows. The vertical connection tube to the scrubber has a provision for injecting water for further cooling of the gas stream before it enters the scrubber. A photograph of the system assembly is shown in Figure 7.2. The three flowmeters in the center serve to measure cooling water flowrates to different sections of the test chamber. The scrubber and its control panel, which also controls the burnoff igniter above it, are shown on the right. The control cabinet for the plasma torch, including the safety circuitry, is visible in the background. The torch power supply is located behind it.

The $SiCl_4$-supply system is shown in Figure 7.3. The

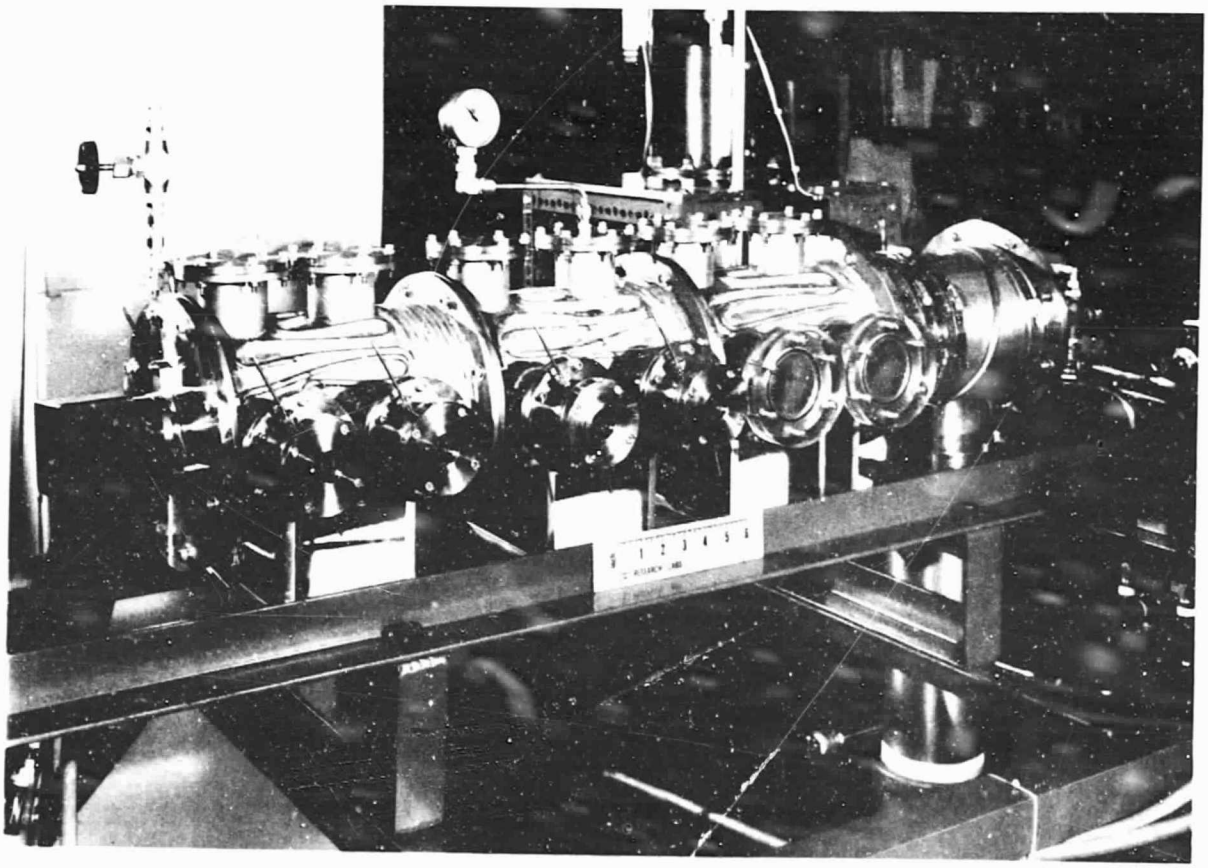


Figure 7.1 - Photograph Of Test Chamber

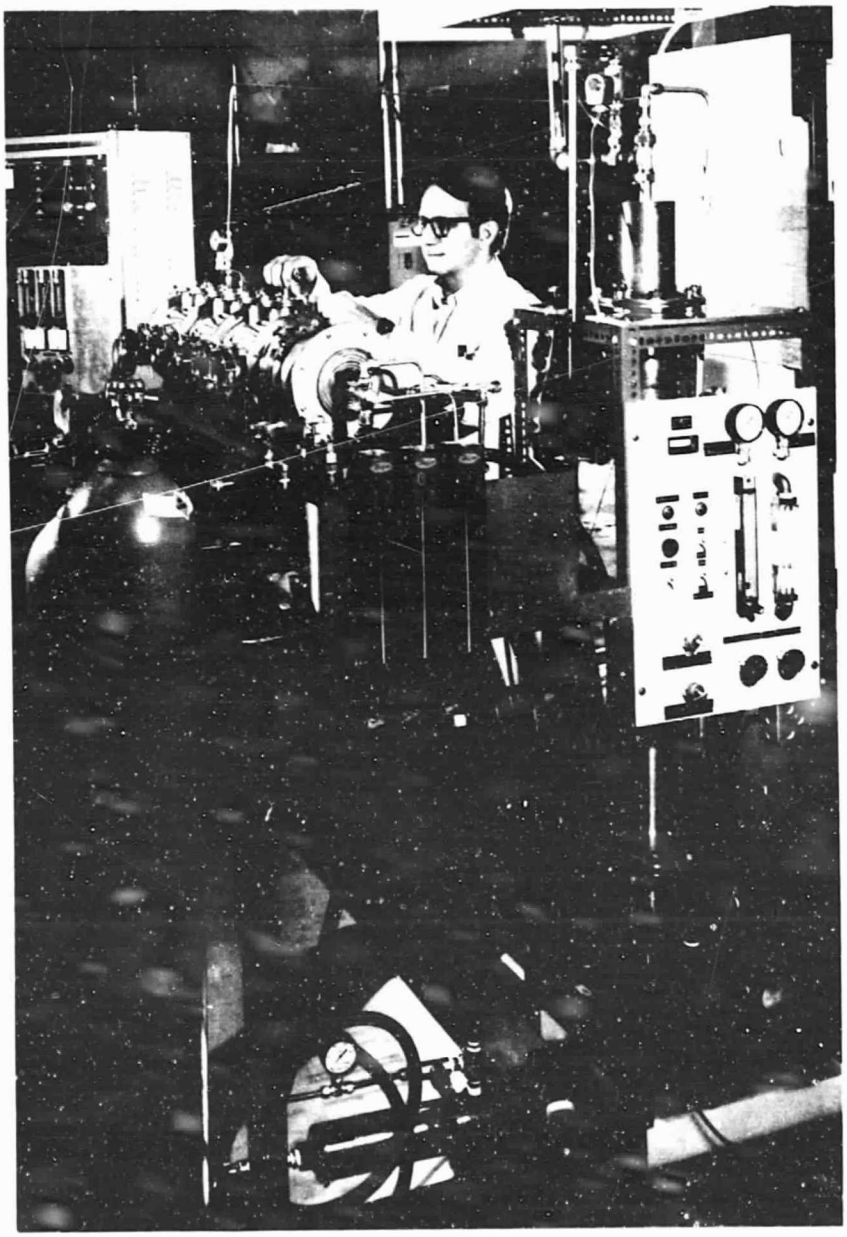


Figure 7.2 - Photograph Of Kinetics Test System Assembly

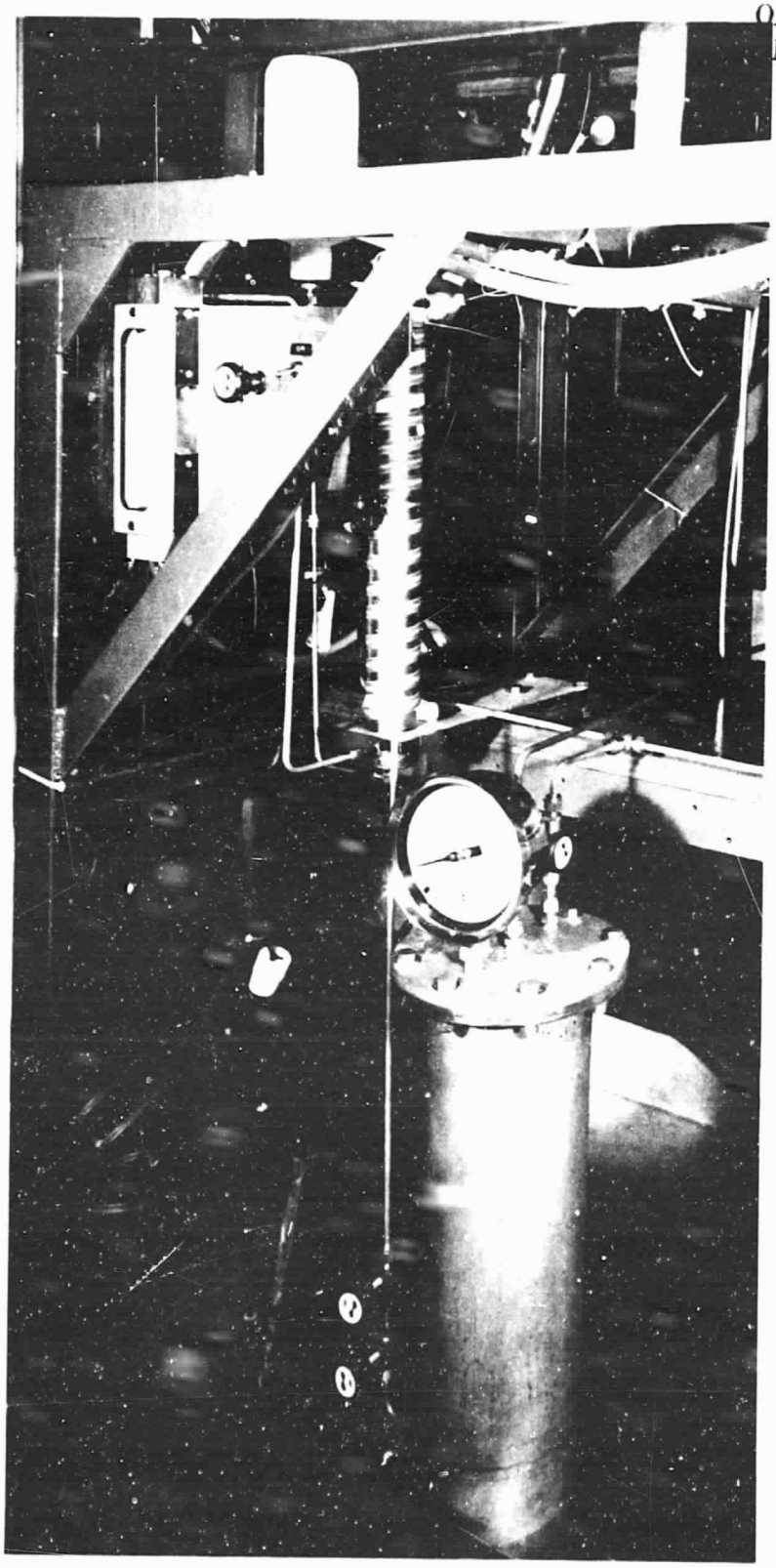


Figure 7.3 - Photograph Of The $SiCl_4$ Injection System Mounted Below The Test Apparatus

reservoir with the attached pressure gauge, pressure relief valve and argon feed line is shown in the center. The control valve and the flowmeter are in the upper left, and the $SiCl_4$-boiler with a helically-wound external heating tape is in the upper center.

Several experiements were run at up to 35 kW torch power with hydrogen flowrates of around 150 scfh and argon flowrates of around 35 scfh. Minor modifications were made on the system as a result of these tests. In a few runs, $SiCl_4$ was injected into the gas stream.

In the runs with $SiCl_4$ injection, a considerable amount of silicon condensed on the wall of the injection nozzle. As a result, the injection holes clogged partially, and the $SiCl_4$ flow into the injection nozzle was difficult to control. The injection nozzle was further modified and a graphite liner was inserted which surrounded the plasma flow channel. Experiments with this nozzle arrangement are in progress.

The silicon which condensed on the nozzle wall was studied with the aid of Energy Dispersive Analysis of X-rays (EDAX), and by an electron beam microprobe analysis. In both cases the substance was confirmed to be silicon with traces of Fe, Ni and Cr. The latter three materials probably originated from the nozzle and injection tubes.

7.2.2 Product Separation Study

A particle filter probe was built and is ready to be installed. A photograph of the different parts is shown in Figure 7.4. The probe stem will be attached through a sliding seal in one of the window ports and inserted into the gas stream during operation. The white disc is the Zirconia filter material while the other parts are made of silicon carbide.

Because the flow conditions in the Kinetics Experiment

Figure 7.4 - Components Of Particle Filter Probe

are different from the ones in the actual test reactor, the analysis predicting Si-condensation for the test reactor was modified to be more suitable to the laminar flow conditions of the Kinetics Experiment. More fundamental modification of the analysis will follow when experimental results are present for comparison.

7.2.3 Data Acquisition

The wall temperature of the silicon carbide inner tube was measured pyrometrically. Under typical running conditions, the wall temperature was 1723^{o}K at the first window, dropping to 1523^{o}K at the second window location. Injection of sodium into the gas stream is expected to raise the wall temperature due to the increase in radiative wall heat transfer.

The thermocouples for the calorimetric measurements are all connected to an automated data acquisition system. Energy balances are being made for the different operating conditions. At 21 kW input power without reactant injection, 38% is lost in the torch electrodes and 19% in the injection flange. The remaining 9 kW corresponds to a temperature of 3150^{o}K for the Ar-H_2 mixture at the entrance of the test section. The mass flow for the 4H_2-Ar mixture was 0.6 grams/second.

8. REACTION DEMONSTRATION

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

8.1 Summary

Fabrication and assembly of the test apparatus has been completed. The Nupro metering valve has been calibrated for liquid sodium flow control. Based on shakedown tests in regard to system leak checking, minor modifications have been made in the test apparatus. A method has been devised for charging an evacuated silicon tetrachloride reservoir directly from the sealed drum supplied by the manufacturer. Partial reactant flow tests have been initiated.

8.2 Work Description

Assembly of the test apparatus was initiated early in the quarter with the installation of the Harper Globar furnace, its instrumentation and the flow rack in the Liquid Metals Laboratory. The components of the sodium and silicon tetrachloride subsystems were mounted onto the flow rack. Six (6) variacs were installed for controlling the various heater assemblies. Thermocouple probes were appropriately positioned and connected to a multipoint recorder. The burnoff stack was mounted below the exhaust hood.

As shown in Figure 8.1, the test apparatus includes the reactant subsystems and the high temperature furnace. The $SiCl_4$ subsystem is located on the right side of the flow rack in Figure 8.1, while the sodium reservoir and the furnace for the sodium vaporizer are on the left side of the flow rack. For testing, sodium and $SiCl_4$ vapors are piped separately into the reaction tube within the Harper Globar furnace. This furnace is shown in Figure 8.2 with its instrumentation panel. The cylindrical cavity which encloses the reaction tube is visible on the left side of the photograph.

The Nupro metering valve was calibrated for liquid sodium flow control. The sodium reservoir was pressurized to 50 psig and

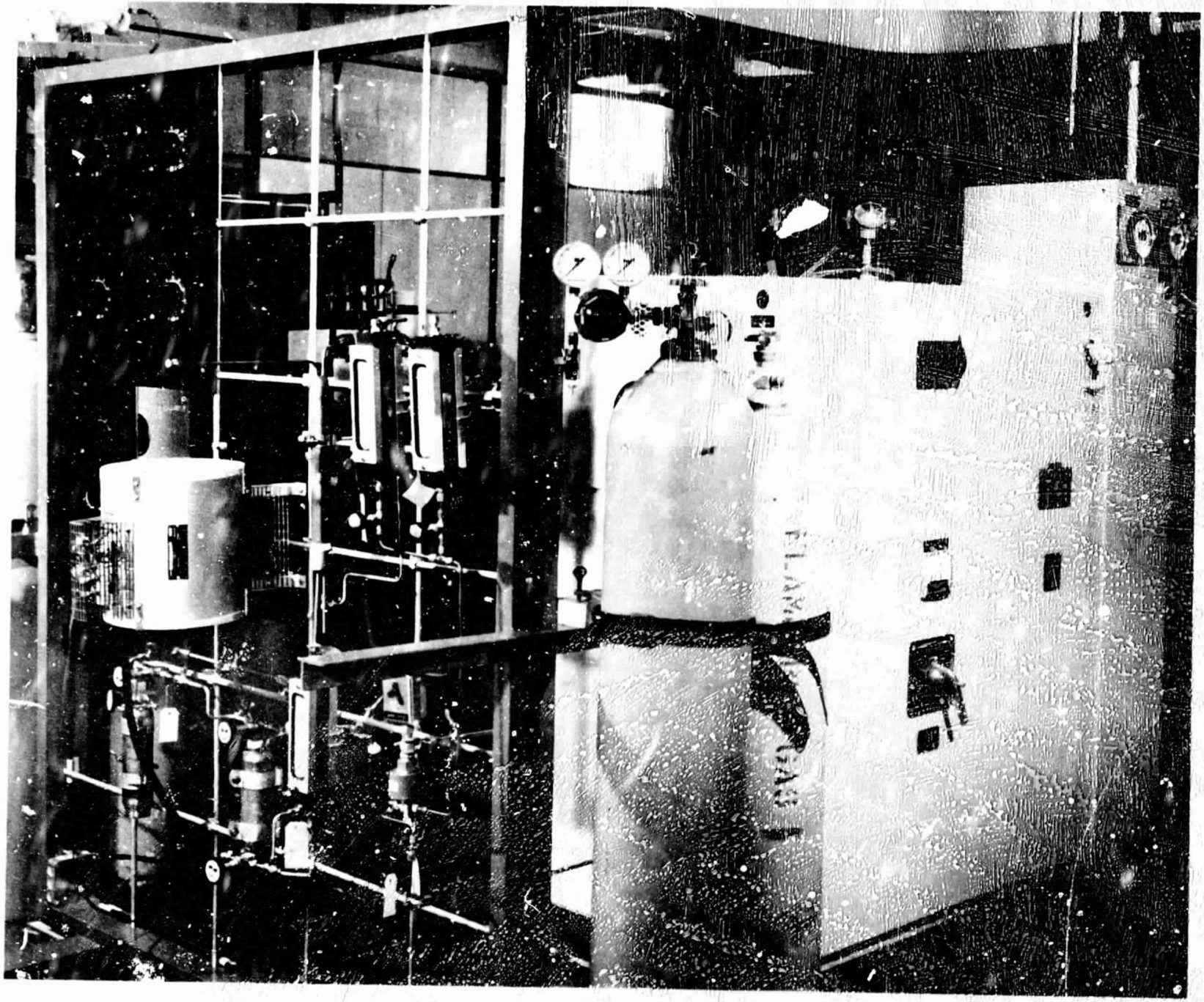


Figure 8.1 - Test Apparatus For The Reaction Demonstration Tests

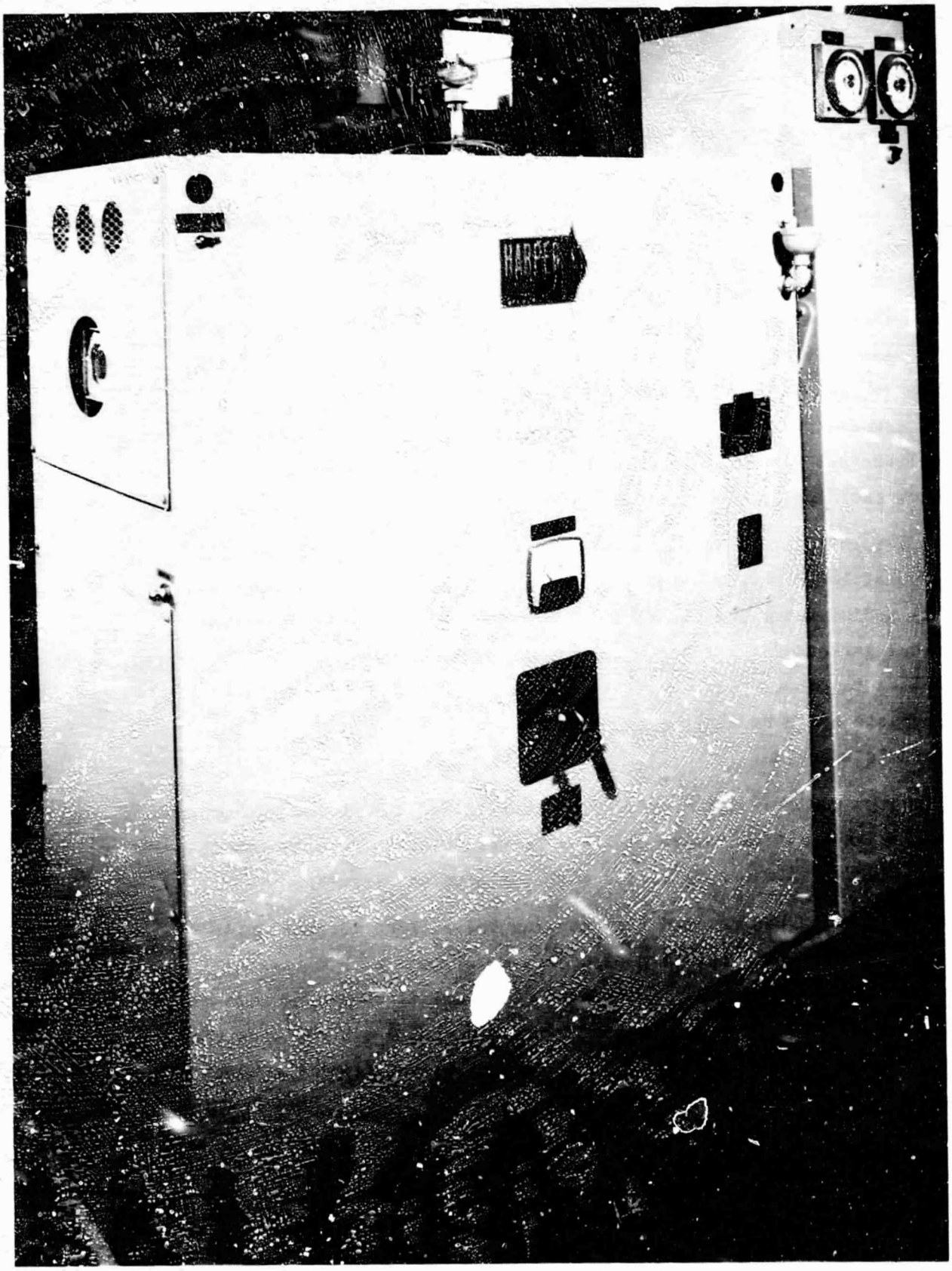


Figure 8.2 - Harper Globar Furnace And Instrumentation Panel

the liquid sodium lines were maintained at 300°C. The calibration curve for the flow control valve is shown in Figure 8.3.

Later in the quarter, discussions were held with personnel from the Liquid Metals Laboratory to evaluate system safety. These discussions resulted in minor modifications to the test apparatus. The threaded end cap (bottom) of the sodium vaporizer was replaced by flanges with gaskets cut from stainless steel foil. The piping was revamped to enable the sodium and silicon tetrachloride reservoirs and purging systems to be pressurized by separate Ar cylinders. A catch pan was fabricated and installed below the flow rack to contain possible spills.

Subsequent shakedown tests for system leak checking revealed several leaks. Persistent leakage occured at the flanged-end of the sodium vaporizer due to insufficient sealing by the stainless steel gaskets. The flanged end of the sodium reservoir was, therefore, welded together. Leakage at the inlet side of the reaction tube endplate could be checked only by the use of a high temperature ceramic adhesive.

The sodium vapor conveying heaters failed during the shakedown tests. A modified design of these heaters was, therefore, fabricated and preliminarily tested with satisfactory results.

A method was devised for charging the silicon tetrachloride reservoir directly from a sealed drum safely and with minimal contamination. A dry and evacuated reservoir is connected to a dip tube protruding from the drum of $SiCl_4$. Predried air replaces the volume of silicon tetrachloride withdrawn, thus filling the test system reservoir.

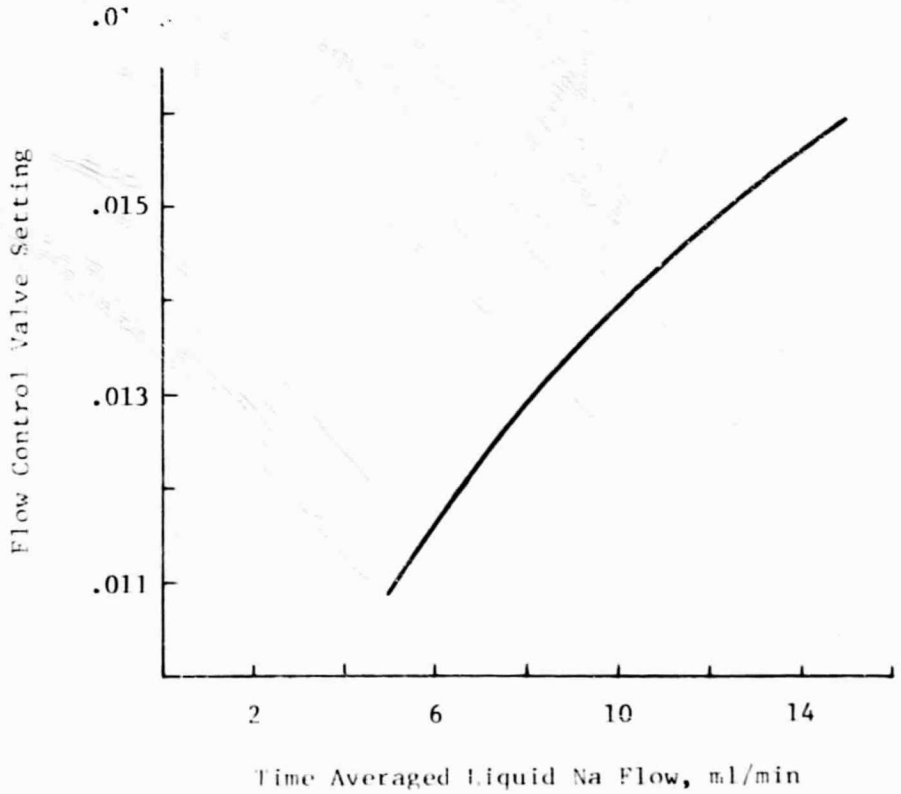


Figure 8.3 - Valve Calibration Curve For Liquid Sodium Flow

9. CONCLUSIONS

- Based on the analyses to date, the reactor configuration (i.e., graphite liners with pyrolytic internal coating) can maintain the Si skull wall design scheme from approximately 1 cm thick skull at the arc heater location to approximately 5 cm at the cyclone region. Heat fluxes will be variable to $\sim \pm 25\%$ of the nominal while maintaining the skull wall.
- Initial tests of the Sonicore nozzle for sodium injection have indicated satisfactory performance and droplet size, thus conditionally verifying this nozzle for use in the plasma reactor during the testing program.
- Preliminary results of the Kinetics Experiment
  a) verify the direct reduction of $SiCl_4$ with hydrogen in a high temperature gas flow
  b) indicate that the required reaction tube temperatures can be achieved.

10. PROJECT STATUS

10.1 Present Status

During the quarter, detailed design was completed for the experimental verification unit for silicon production. Additionally, all purchase orders were placed for the required components and the procurement effort is near completion with many subsystem areas currently fabricating and/or assembling components. Installation of various items at the Arc Heater Laboratory was initiated and continues as the components are received from the vendors. Preparatory efforts for the testing program were initiated with preliminary compilation of both the System Operating Procedures and Safety Manual.

The complementary research programs (i.e., Kinetics, Injection Techniques, and Reaction Demonstration) accomplished initial shakedown tests and system calibrations, in addition, full scale tests for the three projects were initiated.

10.2 Future Activity

- Complete procurement of purchased equipment for the experimental verification unit (EVU).
- Complete fabrication/assembly of the subsystems for the EVU.
- Complete installation of the entire test system assemblies for the EVU.
- Initiate system shakedown tests for the EVU.
- Complete the experimental testing for the respective research programs

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