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CONSTRUCTION OF A PROTOTYPE SULFURIC ACID MIST MONITOR

Environmental Sciences Research Laboratory
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U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

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CONSTRUCTION OF A PROTOTYPE SULFURIC ACID MIST MONITOR

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ABSTRACT

A prototype sulfuric acid mist monitor has been constructed for the purpose of detecting sulfuric acid-sulfur trioxide. The monitor utilized the selective condensation method with subsequent determination of sulfuric acid by measuring the conductivity of an aqueous isopropanol solution. The instrument is fully automated with a mass flow controller and standard TTL logic to allow for easy modification of the timing circuit. After collection of the H_2SO_4-SO_3 in the temperature controlled spiral condensor, the sample is washed into a conductivity cell for measurement. During conductivity measurement the condensor is rinsed with methanol and air dried prior to the next sample collection. The instrument was designed to measure from 1 to 100 mg/m^3 H_2SO_4 and presents the measurement within a 10 mv range. The sample timing cycle can be varied from 105 seconds to 999 seconds.

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FIGURES

TABLES

I. INTRODUCTION

The Sulfuric Acid Mist Monitor (SAMM) designed and constructed for the Environmental Protection Agency (EPA) is described in this report. The objective of this program was to provide one prototype acid mist monitor for use on contact sulfuric acid manufacturing plants. The monitor utilizes the selective condensation method with subsequent determination of sulfuric acid by measuring the conductivity of an aqueous isopropanol solution. Currently, only manual methods are available for this purpose. The manual methods (EPA Standard Method 8) require extensive manpower and have several problems associated with them. Among these are losses in the probe, conversion of SO_2 and delays in analyzing samples. The SAMM is capable of continuously monitoring the stack gas in the range of 1 to 100 mg/m^3 unattended. The detrimental effects of sulfate aerosol to animals, plants, life and property has been established (1). The specific amount of sulfuric acid emitted by stationary sources is important in the understanding of local pollution and the mechanism of sulfate production in an area.

II. MONITOR CONSTRUCTION

A. CABINET

The components of the monitor are housed within an all-weather cabinet consisting of modular compartments. Figures 1 and 2 show the front and rear views of the cabinet, respectively. These same views, with the doors open, are shown in Figures 3 and 4. The cabinet is of sandwich construction, with a layer of insulation between two layers of aluminum. The sandwich was applied over an aluminum frame with a welded aluminum plate on the bottom. All interior partitions are of the same construction. Supports for the glassware are of aluminum angle designed to "float" in space to protect delicate components. The final outside dimensions of the cabinet are approximately 0.76 m x 0.63 m x 0.94 m, with a weight of about 90 Kg. The heated compartment (Figure 3-A) is thermostatically regulated to 65^{o}C to keep heat loss from the glassware to a minumum. Feed-throughs are provided between compartments for gas flow, solution flows, waste liquids, waste gas and electrical connections. Openings through the cabinet are provided for air intake and exhaust, electrical connections for the probe heater, recorder output and power input, the probe itself, and waste connections for liquid and gas.

B. OPERATION DESCRIPTION

The general system as described by Richter and Sattelberg (2) is shown in Figure 5. Basically, the system condenses sulfuric acid out of a sample emission stack air, washes the condenser with aqueous isopropanol

Figure 1

LEGEND:

- A Heated Compartment
- B Spiral Condenser Compartment
- C Methanol and Water Compartment
- D Exhaust Hood

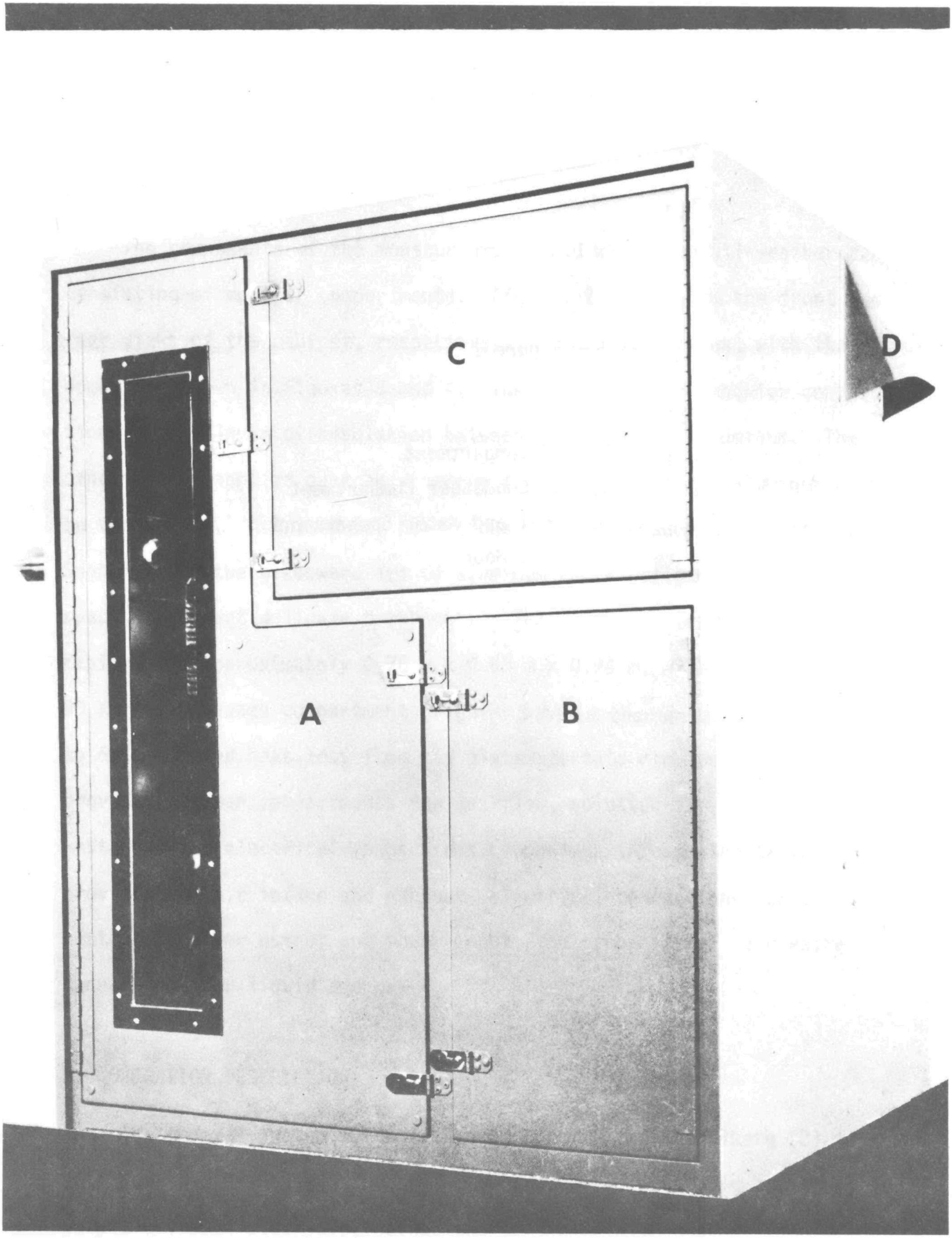


Figure 1 Front View

Figure 2

LEGEND:

- A Electronics Compartment
- B Power Inlet Connection
- C Probe Heater Connection
- D Recorder Output
- E Probe Holder
- F Air Inlet

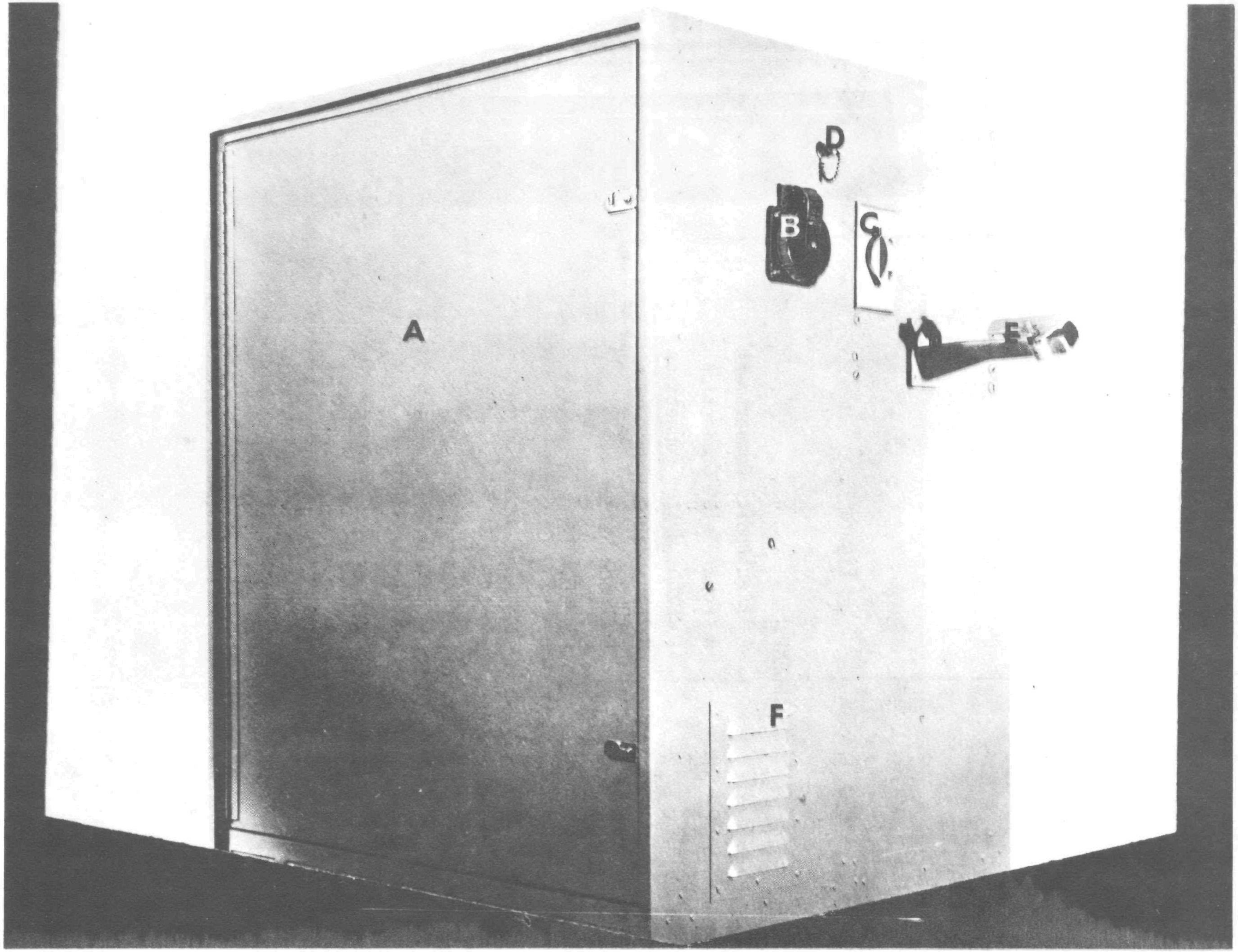


Figure 2 Rear View

Figure 3

LEGEND:

- A Heated Compartment
- B Spiral Condenser Compartment
- C Methanol and Water Compartment

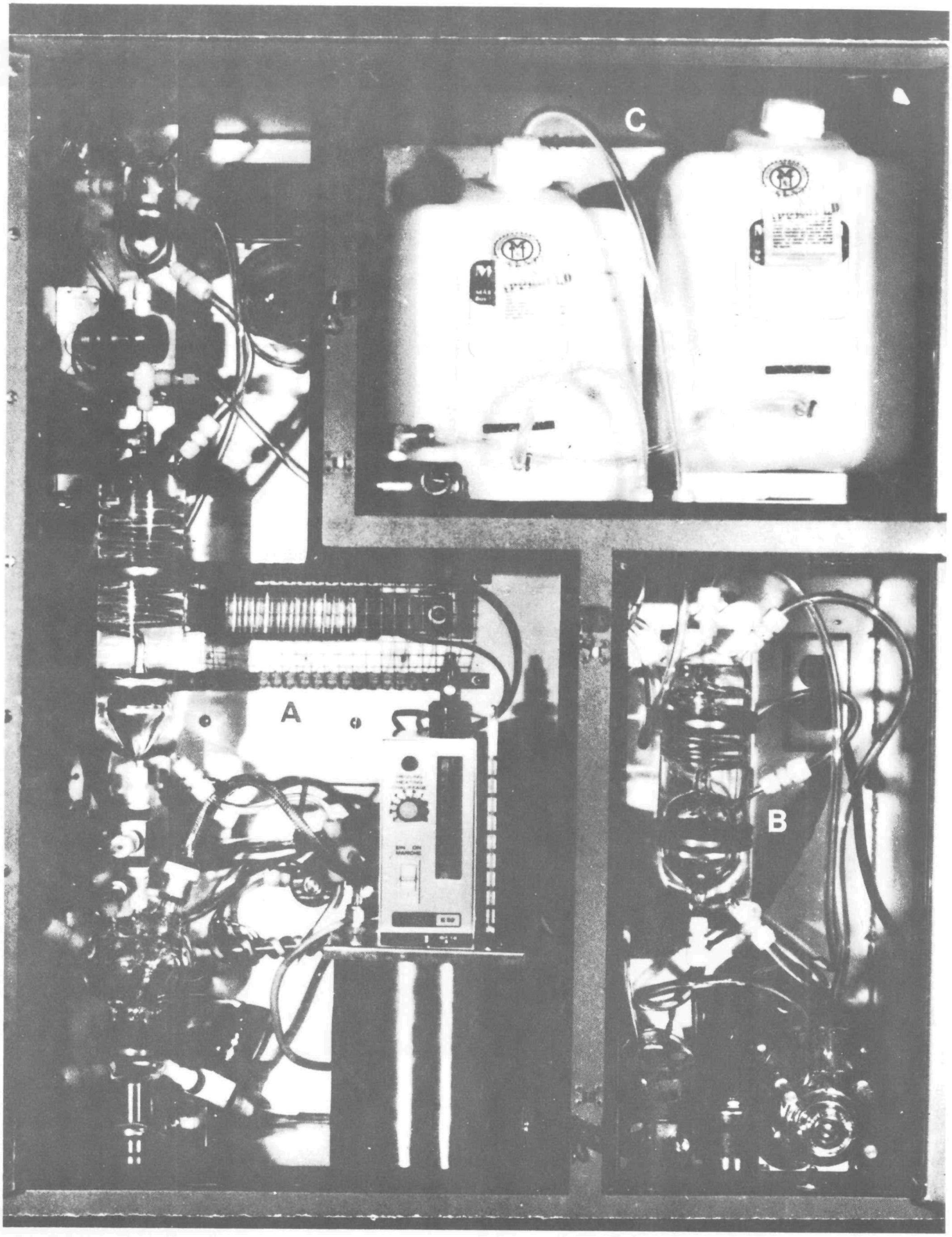


Figure 3 Front View, Doors Open

Figure 4

LEGEND:

A Relay Panel
B Timing Panel
C Distribution Panel
D Flow Indicator
E Conductance Meter
F Isopropanol Pump
G Main Pump
H Mass Flow Controller
I Isopropanol Reservoir
J Temperature Controller
K Magnetic Stirrer Controller

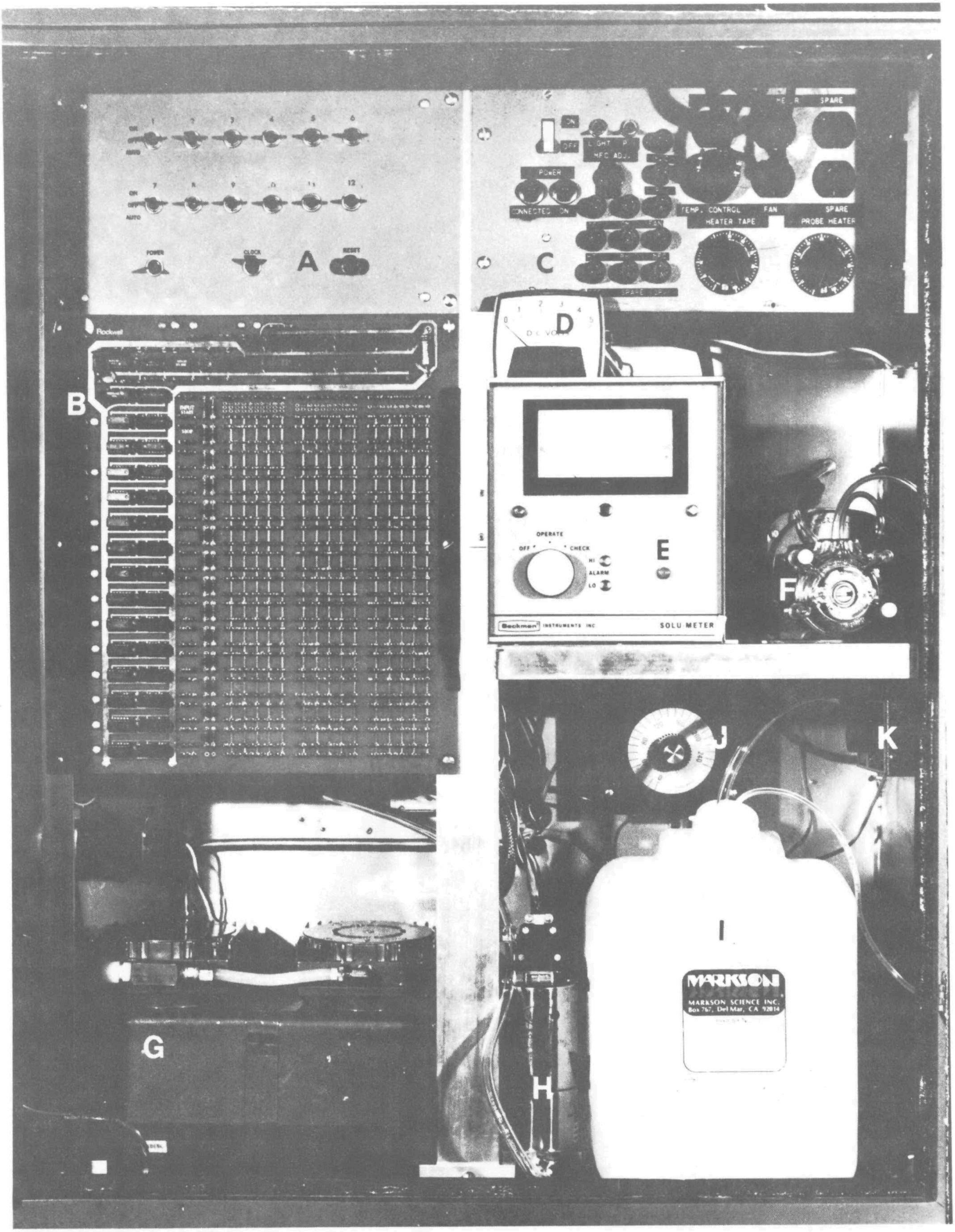


Figure 4 Rear View, Door Open

Figure 14

LEGEND:

A Measuring Vessel
B Solenoid SV-1
C Shell Condenser
D Solenoid SV-2
E Measuring Cell
F Magnetic Stirrer
G Solenoid SV-6
H Solenoid SV-3
I Constant Temperature Bath
J Drain
K Heating Element
L Light
M Fan

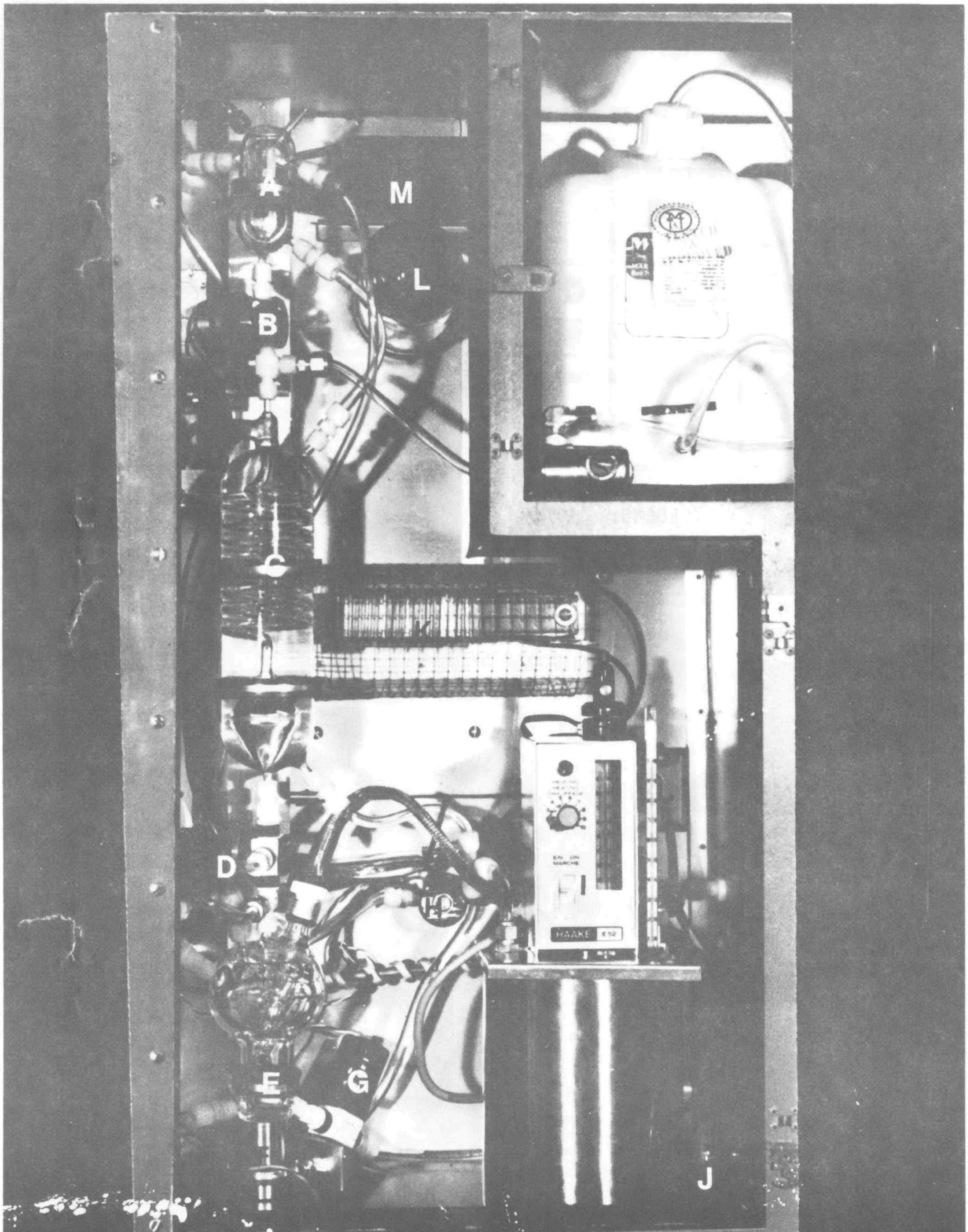


Figure 14 Heated Compartment

Figure 6

LEGEND:

A 000-900 Timing Pins

B 00-90 Timing Pins

C 0-9 Timing Pins

D Start Row

E Stop Row

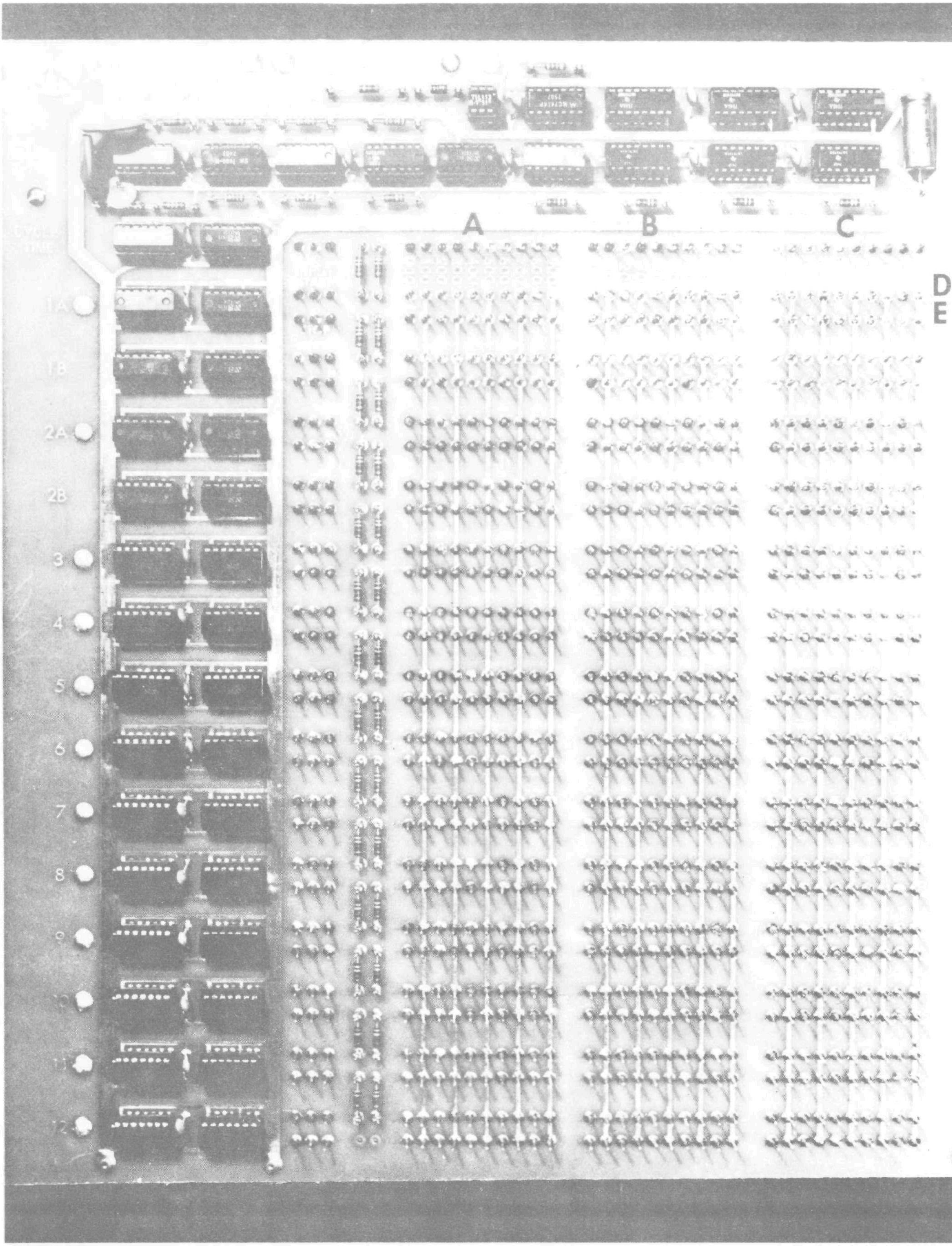


Figure 6   Timing Panel

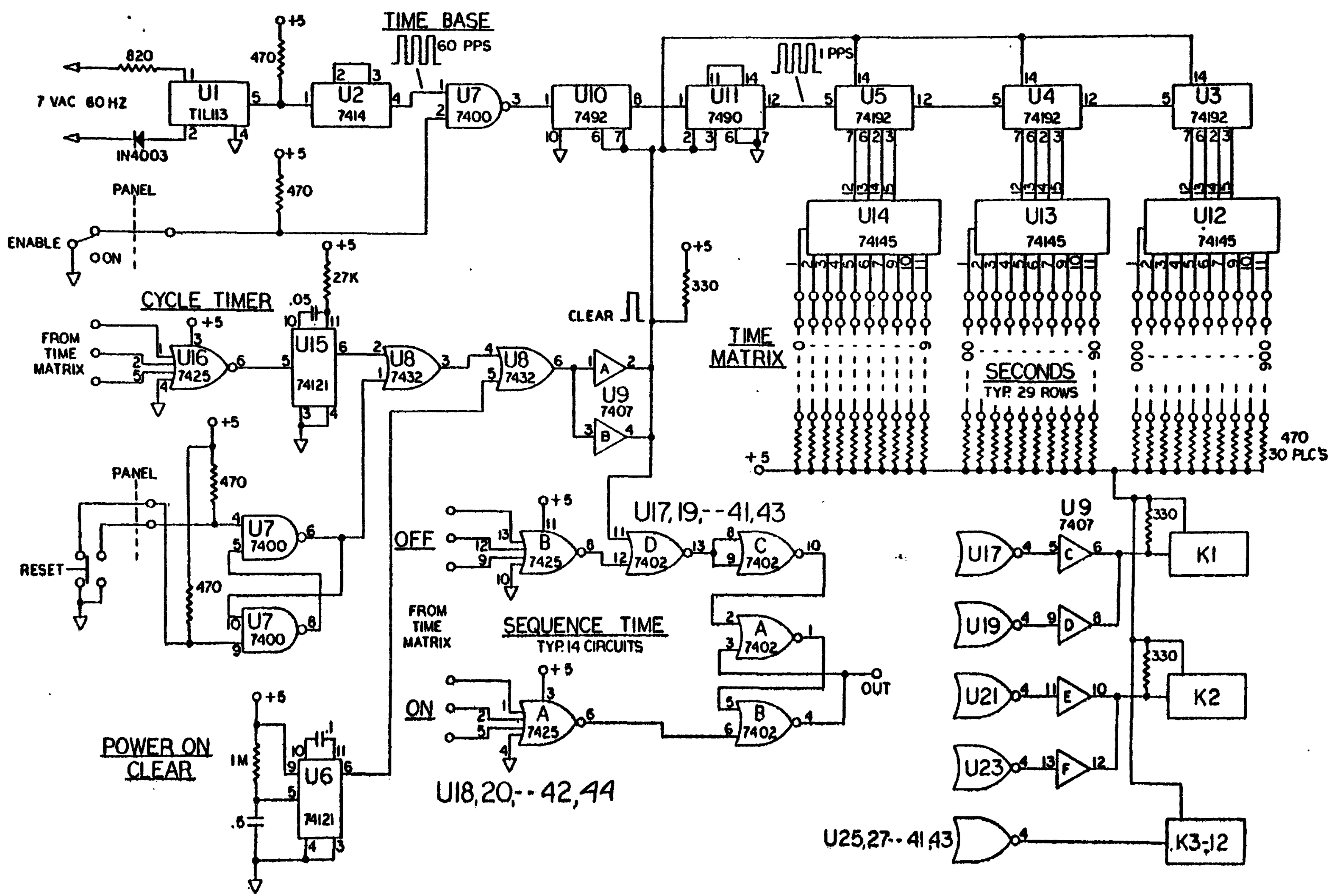


Figure 7 Timing Circuit

generator). These pulses are counted using a three digit BCD counter (cycle timer). When the count reaches a predetermined limit, the BCD counter is reset to zero and the count started over. The cycle time can be varied from 105 to 999 seconds giving a maximum cycle time of 16.6 minutes, and a maximum sample time of 15.6 minutes. One minute is used in the shell condenser washing-rinsing sequence. The various components of the system are turned on and off using this cycle timer and a start-stop timer. The start-stop timer consists of two 12-bit comparators which set or reset a flip-flop when the count equals a preset value. The output is then used to control a solid state relay which is connected to a particular component of the system. The count for each comparator can be set to any value over the entire counting range and is completely independent of the other comparators, except, that the start and stop comparators for each flip-flop cannot be set at the same value. This circuit accurately times and controls the various components of the instrument. All component start/stop times are preset, however, any time may be changed by removing and replacing the wire wrap when the need arises. The preset times and components corresponding to each relay are as follows:

Table 1. Timing Sequence

Relay Number	Component	Time On	Time Off
1A	MFC (1) Low Flow	25	90
2A	MFC High Flow	1	25
2B	MFC High Flow	90	End of Cycle
3	SV-8	95	105
4	SV-7	95	105
5	P_3	90	107
6	SV-4	80	84
7	SV-1	34	55
8	SV-3	32	77
9	SV-2	32	77
10	SV-6	1	30
11	SV-9	1	30
12	P_2	1	31

(1) MFC - Mass Flow Controller

Cycle time may be set from 105 to 999 allowing sample times from 0.75 to 15.65 minutes. The cycle time is set by placing the jumper wires on the selected time from A, B and C shown in Figure 5 and on the cycle time control points (Figure 7).

Relay Panel

The relay panel (Figures 8 and 9) contains on/off/auto switches for all timed components. The timing power switch, clock actuate switch and

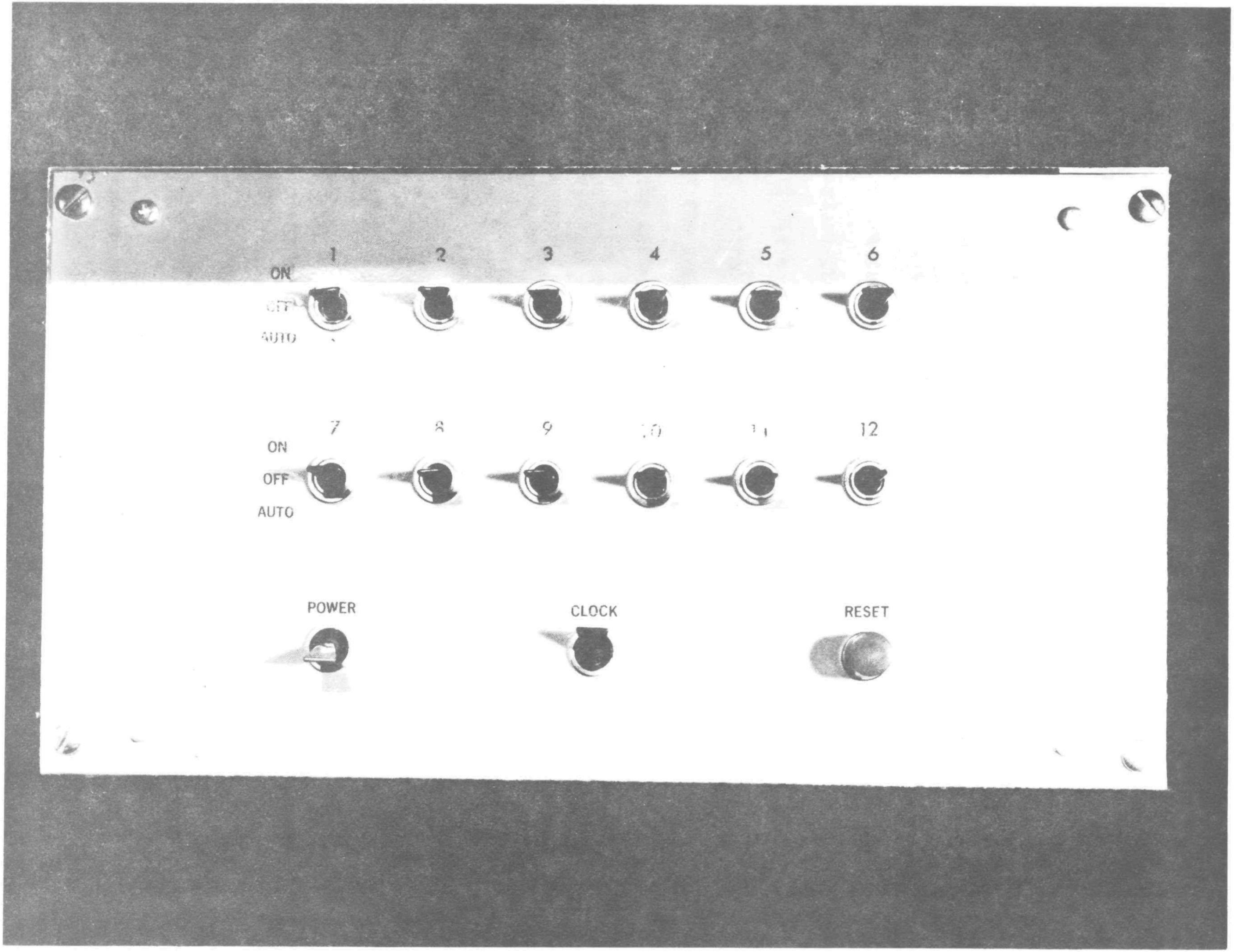


Figure 8 Relay Panel

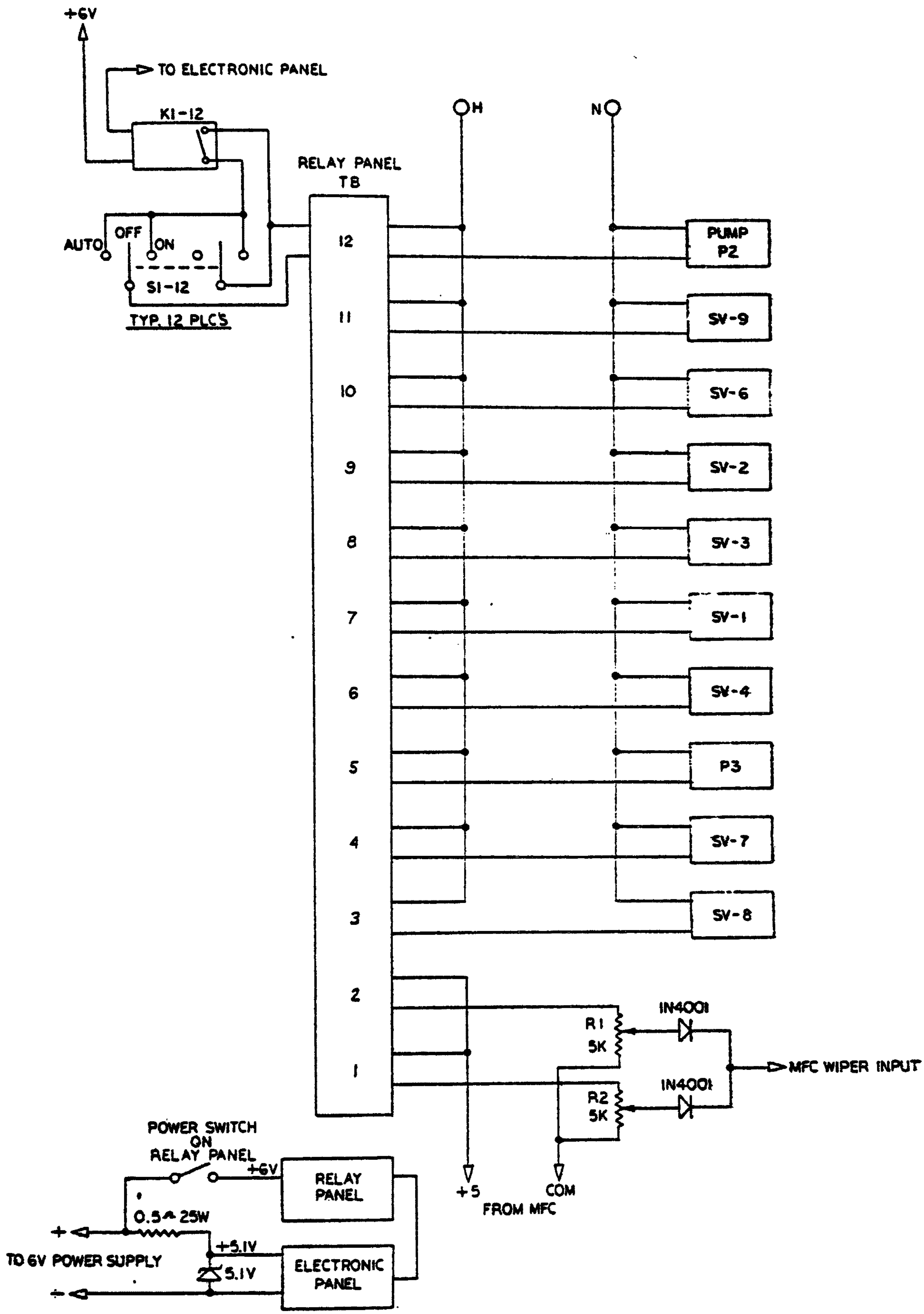


Figure 9 Relay Circuit

clock reset button are also on this panel. Teledyne solid state relays are used for all timed functions. These are located in the relay panel and connected to a terminal strip on the back of the panel. The on/off/auto switches allow manual or automatic control of timed components. Each component is activated when the switch is turned to "on". The "auto" position of the switch changes control of the components to the timed intervals set on the timing panel. During normal operation all switches are placed in the "auto" position. The power switch is placed in the up position to supply power (5 VDC) to the timing circuit. The clock actuate switch is placed in the down position to start the clock running. The clock reset button resets the clock to zero time when actuated.

Electrical Distribution

The electrical distribution panel (Figures 10 and 11) contains the main power switch, switches for the main pump (P1) and heated compartment light, fuses, controls for mass flow controller, probe and tape heaters and plugs for components that are not controlled by the timing circuit. Two spare 115 v AC fused electrical outlets are also provided.

Sample Flow Control

The sample flow rate is controlled by a mass flow controller (Figure 12-B, Tylan Corporation, Model No. FC-202). The flow controller incorporates a valve and electronics to automatically regulate flow proportionally to an external signal. The external signal is provided by the pot (Mass Flow Controller Adjustment MFC Adj.) on the distribution panel (Figure 10).

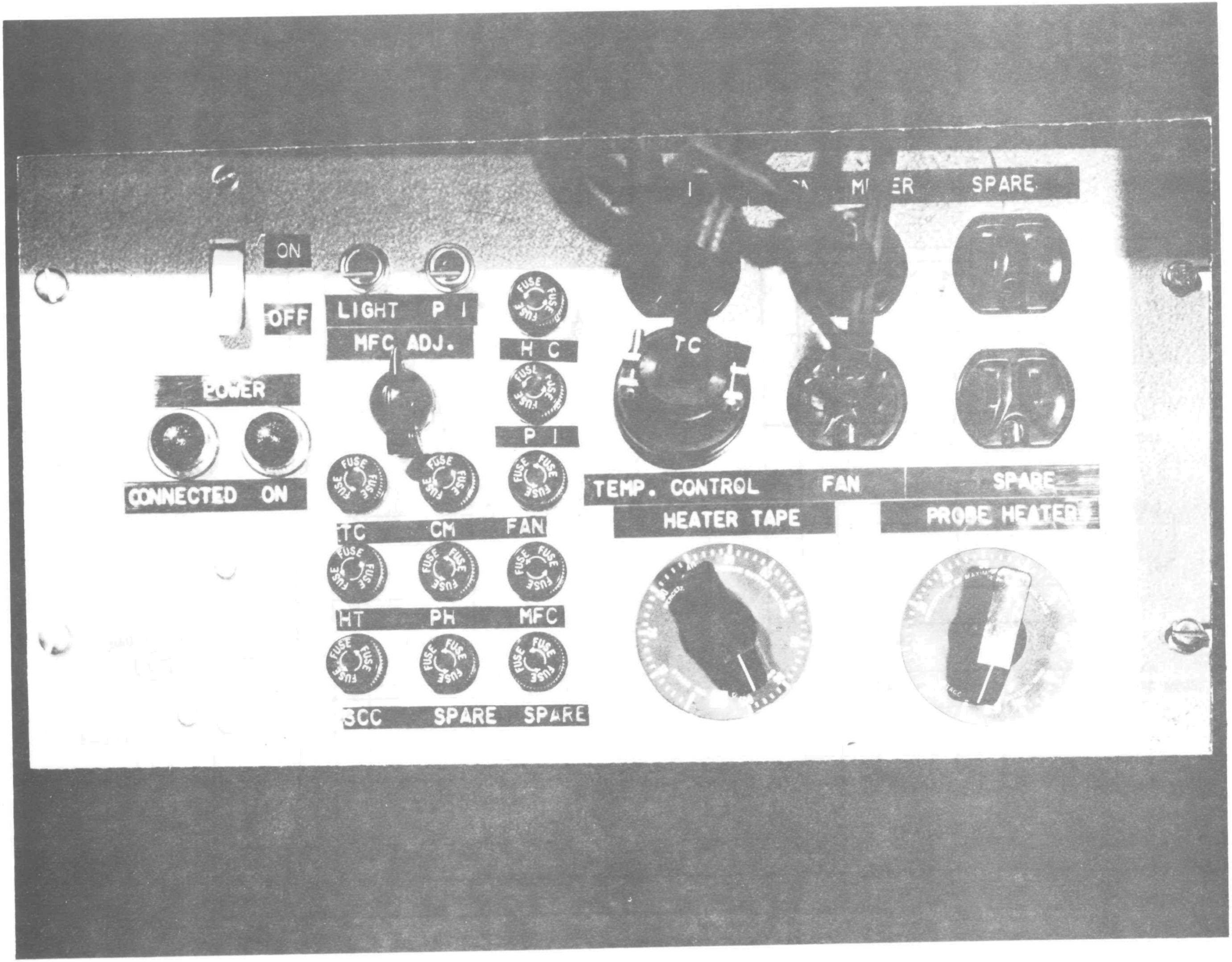


Figure 10 Distribution Panel

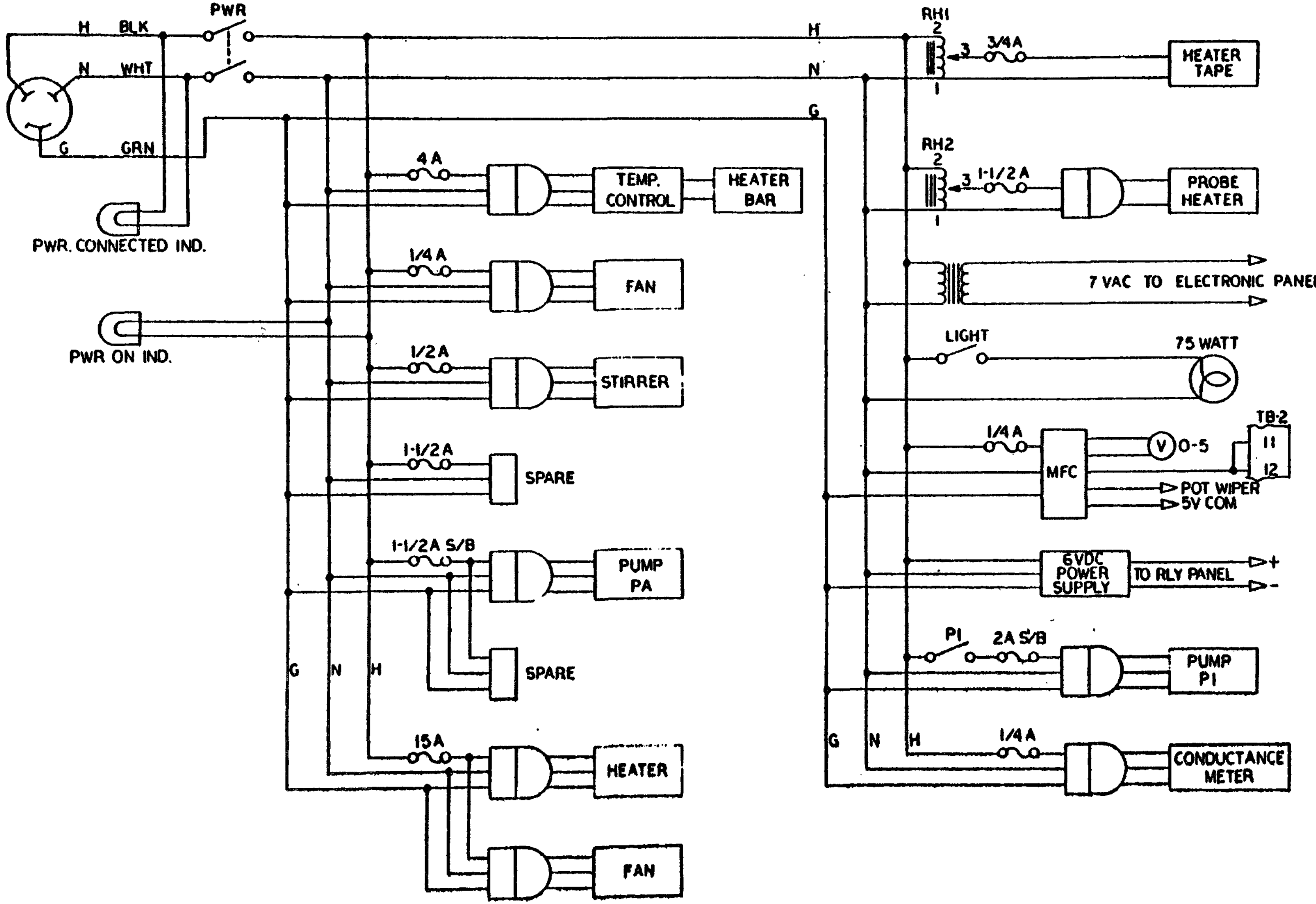


Figure 11 Distribution Circuit

Figure 12

LEGEND:

A Main Pump, P1

B Mass Flow Controller

C Isopropanol Reservoir

D Temperature Controller

E Magnetic Stirrer Controller

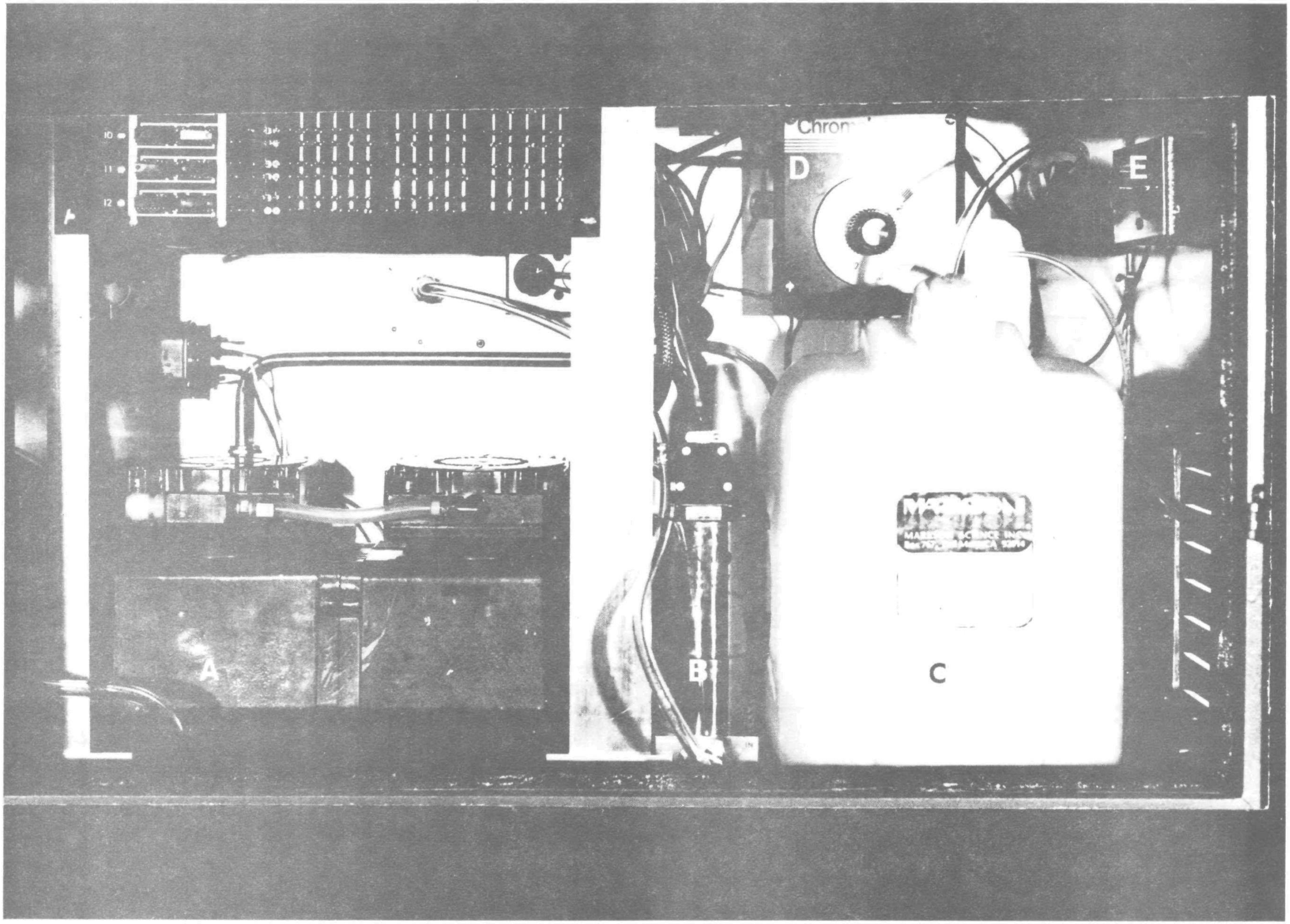


Figure 12   Lower Electronic Compartment

The valve used in the Tylan flow controller is a thermal expansion design that eliminates friction, moving seals and all material except 316 stainless steel. The actuator is a small thin-wall tube with a ball welded to the end and a cone seat. Inside the tube is a heater wire that causes the tube to expand relative to the outer shell, moving the valve, and thereby setting the flow. Two resistence thermometers are wound adjacent to each other on the outside of the sensor tube. These thermometers form part of a bridge circuit. When there is a flow in the tube, the up-stream sensor is cooled and the down-stream sensor is heated, which produces a signal from the bridge which is proportional to flow. The mass flow controller is set by an external command signal from a pot on the distribution panel. The command signal is compared internally with the amplified sensor signal to give an error voltage proportional to the difference between the desired flow rate and the actual mass flow rate. The control valve in the output flow path of the controller acts in response to this signal to reduce the difference between command and actual flow to zero. An indication of the flow is supplied on 0-5 VDC meter. One volt corresponds to 20% of full flow. The flow controller is calibrated for air from 0-20 lit/min. The sample flow rate is adjusted by turning the pot (MFC Adj.) on the distribution panel until the desired flow is indicated on the meter (Figure 13-A). During the period when the condenser is being rinsed and cleaned, the flow is automatically set at 20% of full flow (5 lit/min), to reduce turbulence and carry over by the gas stream.

The flow controller has fine-mesh screens permanently installed on both the inlet and outlet section to prevent accidental clogging of the sensor

Figure 13

LEGEND:

- A Flow Meter
- B Conductance Meter
- C Isopropanol Pump

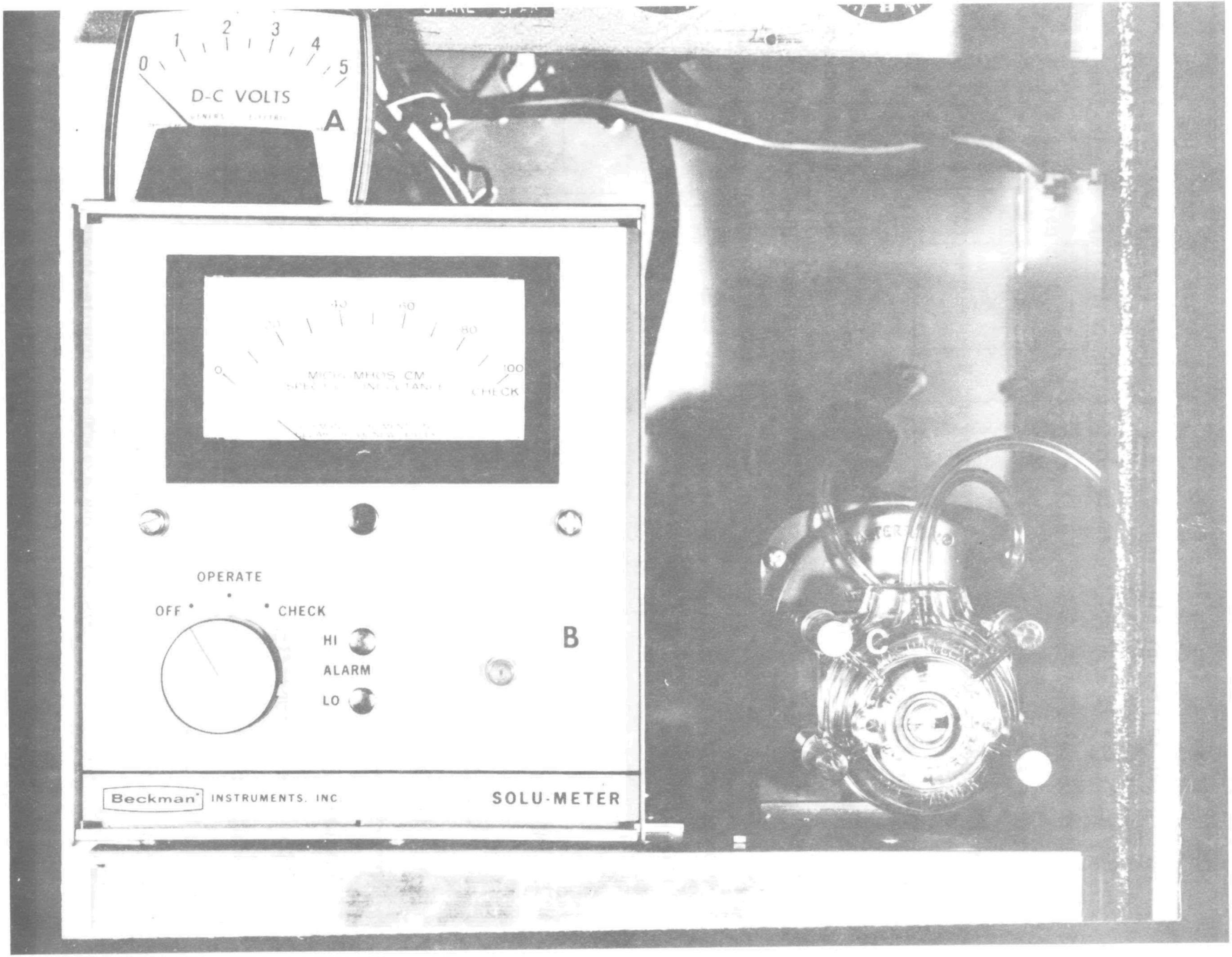


Figure 13 Upper Electronics Compartment

tubing or valve. An in-line filter is placed before the mass flow controller to prevent plugging of these permanent screens and to provide a simple, easily changed system to protect the controller. The in-line filter contains glasswool and desiccant to prevent both particles and moisture from carrying over. The operation and maintenance manual for the flow controller is in the Operation and Maintenance (O&M) Manual.

Conductance Meter

The conductance of the 5% aqueous isopropanol acid solution is measured by use of a wheatstone bridge conductance meter (Figure 13-B) manufactured by Beckman Instruments, Inc. (Model No. RA5). A visual meter indication is provided in the electronics compartment as well as a 10 mv recorder output (Figure 2-D) external to the cabinet. The operation and maintenance manual for the conductance meter is in the O&M manual.

D. MECHANICAL SYSTEMS

Sampling Systems

The sampling system is comprised of a shell condenser (Figure 14-C), a measuring vessel (Figure 14-A), and a conductance cell (Figure 14-E). These glassware components, along with the spiral condenser (Figure 14-A) were custom made to specifications by Cal-Glass (Costa Mesa). A complete set of drawings for the glassware is in the O&M Manual. An extra set of glassware consisting of a measuring cell, a shell condenser, a measuring vessel and a spiral condenser for replacement are provided with the instrument.

The components of the sampling system are double-jacketed, allowing

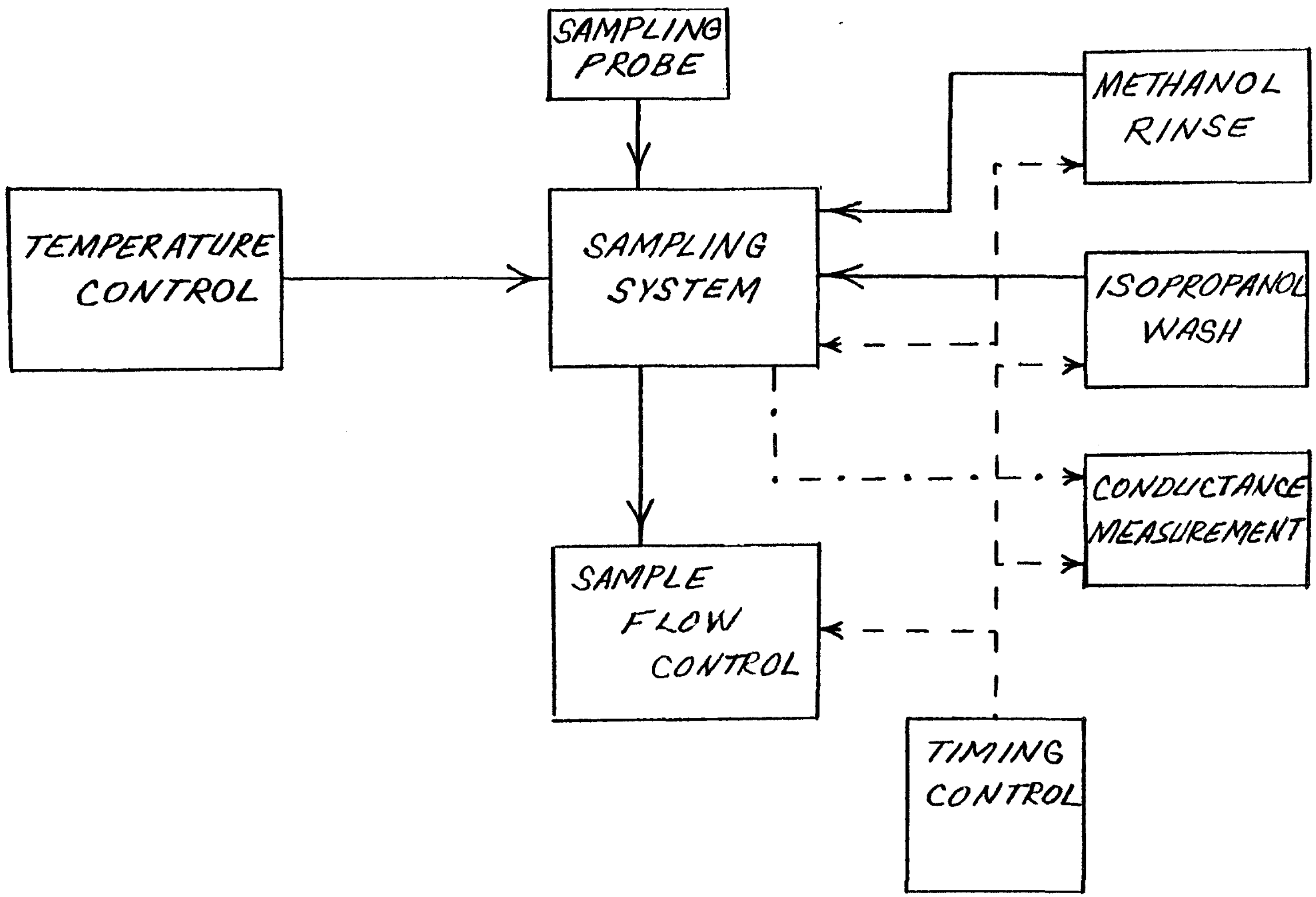


Figure 5 Basic System (SAMM)

and measures the conductance of the isopropanol-sulfuric acid solution. To automate the procedure and obtain valid measurements, other capabilities have been provided: temperature control, control of the sampling steps (timing control), solution volume control, washing or cleaning of the condenser and sample mixing. The operating sequence involves the following steps: gas sampling, washing and drying the shell condenser, and conductivity measurement. During the gas sampling phase stack gases are passed through the heated quartz probe and into the shell condenser where the sulfuric acid is selectively condensed. The condenser is washed after the end of sampling. During the washing process the 5% aqueous isopropanol sulfuric acid solution is introduced to the measuring cell. The condenser is then rinsed with methanol and dried in preparation for the next sampling period. The electrical conductivity of the isopropanol-sulfuric acid solution is measured during the next cycle. The measuring cell is emptied before the next washing cycle. Detailed discussions of the monitor systems are in the following sections.

C. ELECTRICAL SYSTEMS

Timing Circuit

Standard TTL logic was used to allow easy modification, small size and easy adaptation of the timing circuit for production at a later time. The circuit (shown in Figures 6 and 7) has the following characteristics.

The 60 Hz line frequency (60 Hz clock pulse generator) is used as the time base and counted to give pulses of frequency 1 Hz (1 Hz clock pulse thermostatic control. Temperature control is maintained by a constant temperature Haake circulating bath. The O&M manual contains the instruction and maintenance manual for the temperature bath.

The measuring vessel is filled by a parastaltic pump (Figure 13-C) operated by the timing system. The pump and reservoir (Figure 12-C) are located in the electronics compartment. The pump is manufactured by Cole-Parmer Instrument Co. (Model No. 7540-14).

The main sampling pump (Figure 12-A) is a dual diaphram type, manufactured by Gast Pump Co. (Model No. DAA 110).

The measuring cell has a number of important design features. The cell is maintained at a constant 70^{o}C by thermostated circulating water (Figure 14-I). The water bath is made by Haake (Model No. E51). The cell has a provision for a stirring bar which allows for an electronically controlled magnetic stirrer to be placed as close as possible. A threaded connector is provided for the conductance cell. Provision for removing the 5% aqueous isopropanol acid solution and drying with heated air is made. The purge air pump is located in the Spiral Condenser Compartment (Figure 16-B) (Neptune Products, Inc., Model No. 54904-006).

The methanol rinse solution volume will be controlled by timing the period in which solenoid valve SV-4 (Figure 15-D) is open. The methanol supply container (Figure 16-B) is outside of the heated compartment because of the low boiling point.

Because heated sulfuric acid mist is highly corrosive, the solenoid valves in contact with the acid are Teflon (Figure 14-D & G). All of the other solenoid valves are stainless steel.

Figure 15

LEGEND:

A Spiral Condenser
B Purge Air Pump, P2
C Cool Water Pump, PA
D Solenoid SV-7
E Solenoid SV-9
F Solenoid SV-8

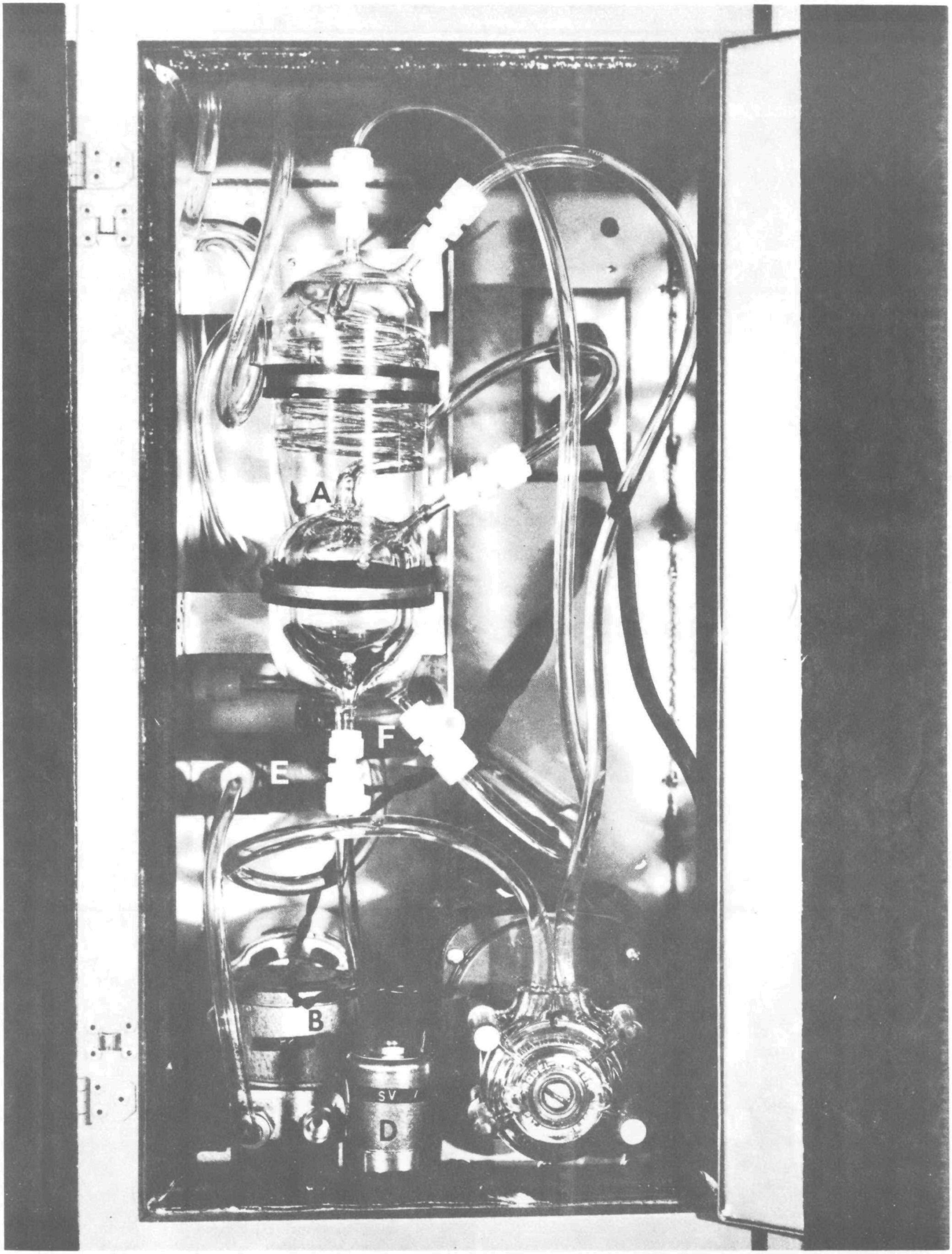


Figure 15 Spiral Condenser Compartment

Figure 16

LEGEND:

- A Cool Water Reservoir
- B Methanol Reservoir
- C Methanol Shelf
- D Solenoid SV-4

Figure 16   Methanol and Water Compartment

The spiral condenser is located outside of the heated compartment. It is supplied with cool water (Figure 15-A) to condense methanol and other vapors preventing damage to the mass flow controller. The cool water pump (Figure 15-C) is manufactured by Cole-Parmer Instrument Co. (Model No. 7540-17).

Sample Probe

The sample probe is a commercially purchased vitreous quartz lined stack sampling probe manufactured by Research Appliance Co. (Model No. STK-LQ-5). The probe was supplied with various interchangeable stainless steel nozzles and a stainless steel protective covering. A pitot tube with quick disconnect couplings was also supplied to aid in isokinetic sampling. The commercial probe, however, was not designed to reach the necessary temperature (300^{o}C). This high temperature is required to keep sulfuric acid from condensing on the probe wall and being lost from the sample. This will require that the probe be modified (see Recommendations for Modification section for details).

III. TESTING AND CALIBRATION

A. TESTING

Following the construction phase, the instrument's mechanical and electrical systems were tested. During the testing, adjustments were made to the timing of certain functions to optimize operation. Certain problems arose while testing which will require modification before the instrument can be operated outside of the laboratory environment.

Testing Procedures

The heated compartment temperature control system was tested by placing a thermometer in the center of the compartment. The control (Figure 9-D) was set at 145^{o}F and the system turned on. After a warm up period, the temperature at the center of the compartment reached 145^{o}F ± 2^{o}F. The primary purpose of temperature control in the heated compartment is to reduce the temperature loss of the condensation apparatus and solution delivery and measurement system. The constant temperature bath was tested and calibrated with the built-in thermometer. The constant temperature bath is used to maintain 70^{o}C temperatures in the measuring cell, measuring vessel and shell condenser.

The relays, timing sequence and sample flow system were tested for proper operation with the specific solenoids and pumps. Solution transfer operations for isopropanol and methanol were tested. Carryover of isopropanol occurred when gas flows above 8 lit/min were used. This required that during calibration the flow be reduced to 5 lit/min during the rinsing/

washing operation. Modifications were made to automate this procedure.

In the initial testing of the gas flow system, after a period of operation, the mass flow controller no longer properly regulated the flow. Flow would drop to almost zero while the reading on the 0-5v DC meter remained normal. This was traced to the spiral condenser. The cooling water to the spiral condenser was warming up, and consequently was not condensing the alcohol vapors, resulting in their carry-over to the flow controller. These vapors were condensing in the controller making it inoperative. This problem was solved by using cool water for the spiral condenser. No further problems were encountered with these components. Testing of the commercial sample probe revealed that the maximum temperature obtainable was 320^{o}F. This temperature is not sufficient for quantitative transfer of SO_3 without condensation. To complete calibration a specially designed probe which could reach 550^{o}F was used as the sample probe.

Recommendations for permanent solutions to these problems are contained in Section V., Recommendations for Modification.

B. CALIBRATION

The experimental system used for calibration is shown in Figure 17. The special probe consists of a quartz tube heated electrically by a spiral of nicrome wire insulated by several layers of asbestos tape. With this arrangement, the probe temperature is adjusted with a variable transformer. Dilute sulfuric acid solution is added at a constant rate by a calibrated syringe pump through a hypodermic needle and serum cap in the top opening

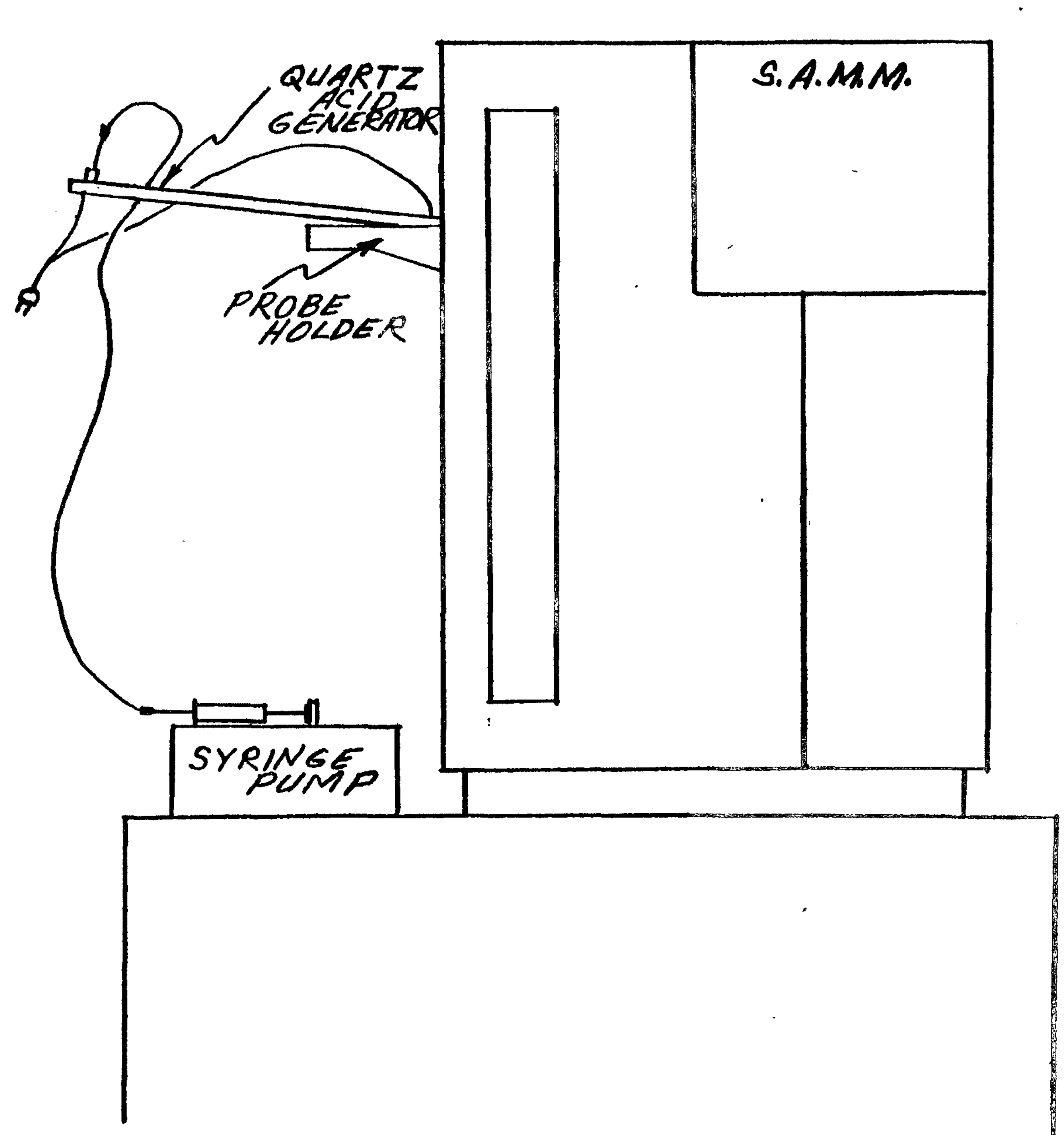


Figure 17 Experimental System

of the probe. The rate of acid addition was altered by adjusting the speed of the syringe pump. Using 0.02 N H_2SO_4 in the syringe, the pump was calibrated at the following rates: 1.02 ml/min, 0.73 ml/min, 0.53 ml/min, 0.38 ml/min, 0.19 ml/min, and 0.073 ml/min (Table 2 shows the calibration data). The generator ground glass fitting was connected directly to the shell condenser in the heated compartment. This set-up simulated the sample probe.

Calibration results are presented in two manners, micromhos vs. milligrams H_2SO_4 (Figure 18) and micromhos vs. milligrams H_2SO_4 per cubic meter (Figure 19).

Table 3 contains experimental values as well as calculated values for added mg H_2SO_4 and concentration in mg/m^3. An example of the calculations of the added mg H_2SO_4, volume sampled and concentration in mg/m^3 are as follows:

mg H_2SO_4 = (ml H_2SO_4/min) x (0.98 mg/ml) x (sample time in min.)

Vol. (M^3) = (Gas flow rate in lit/min) x (sample time in min.) x 10^{-3M^3}/lit

conc. mg/m^3 = mg H_2SO_4/Vol (M^3)

Figure 18 requires only that the total sampled volume be divided into the milligrams H_2SO_4 to give concentration, while Figure 19 requires that both the sample rate and sample time be referenced to the standard of the calibration curve (10 lit/min and 5.75 min).

TABLE 2

SYRINGE PUMP CALIBRATION DATA

Pump Setting	Vol. of Flask	Time	Pump Rate
1.1 ml/min.	10 ml	9;49.7	1.02 ml/min.
0.82 ml/min.	10 ml	13;45.8	0.73 ml/min.
0.58 ml/min.	10 ml	19;02.3	0.53 ml/min.
0.42 ml/min.	10 ml	26;39.1	0.38 ml/min.
0.21 ml/min.	10 ml	52;50.0	0.19 ml/min.
0.073 ml/min.	10 ml	136;59.2	0.073 ml/min.

TABLE 3

CALIBRATION VALUES

Gas Flow Rate Lt/Min.	Sample Time Min.	(0.98mg/ml) 0.02N H_2SO_4 Rate ml/min.	Conductance Meter Reading µmhos	Total mg H_2SO_4 Added	Mean of Meter Readings µmhos	Conc. mg/m^3
10	5.75	0.73	59.0	4.1		
10	5.75	0.73	59.0	4.1	59.3	71.3
10	5.75	0.73	60.0	4.1		
10	5.75	0.38	34.0	2.1		
10	5.75	0.38	37.0	2.1	34.9	36.5
10	5.75	0.38	34.5	2.1		
10	5.75	0.38	34.5	2.1		
16	15.73	0.073	15.0	1.1		
16	15.73	0.073	15.0	1.1	15.0	4.4
16	15.73	0.073	15.0	1.1		
10	15.73	0.19	38.0	2.9		
10	15.73	0.19	38.0	2.9	38.2	18.4
10	15.73	0.19	38.5	2.9		
10	5.75	1.02	85.0	5.7		
10	5.75	1.02	89.0	5.7		
10	5.75	1.02	87.0	5.9	85.8	99.1
10	5.75	1.02	83.0	5.7		
10	5.75	1.02	85.0	5.7		
10	5.75	0.53	54.0	3.0		
10	5.75	0.53	55.0	3.0	54.3	52.2
10	5.75	0.53	54.0	3.0		

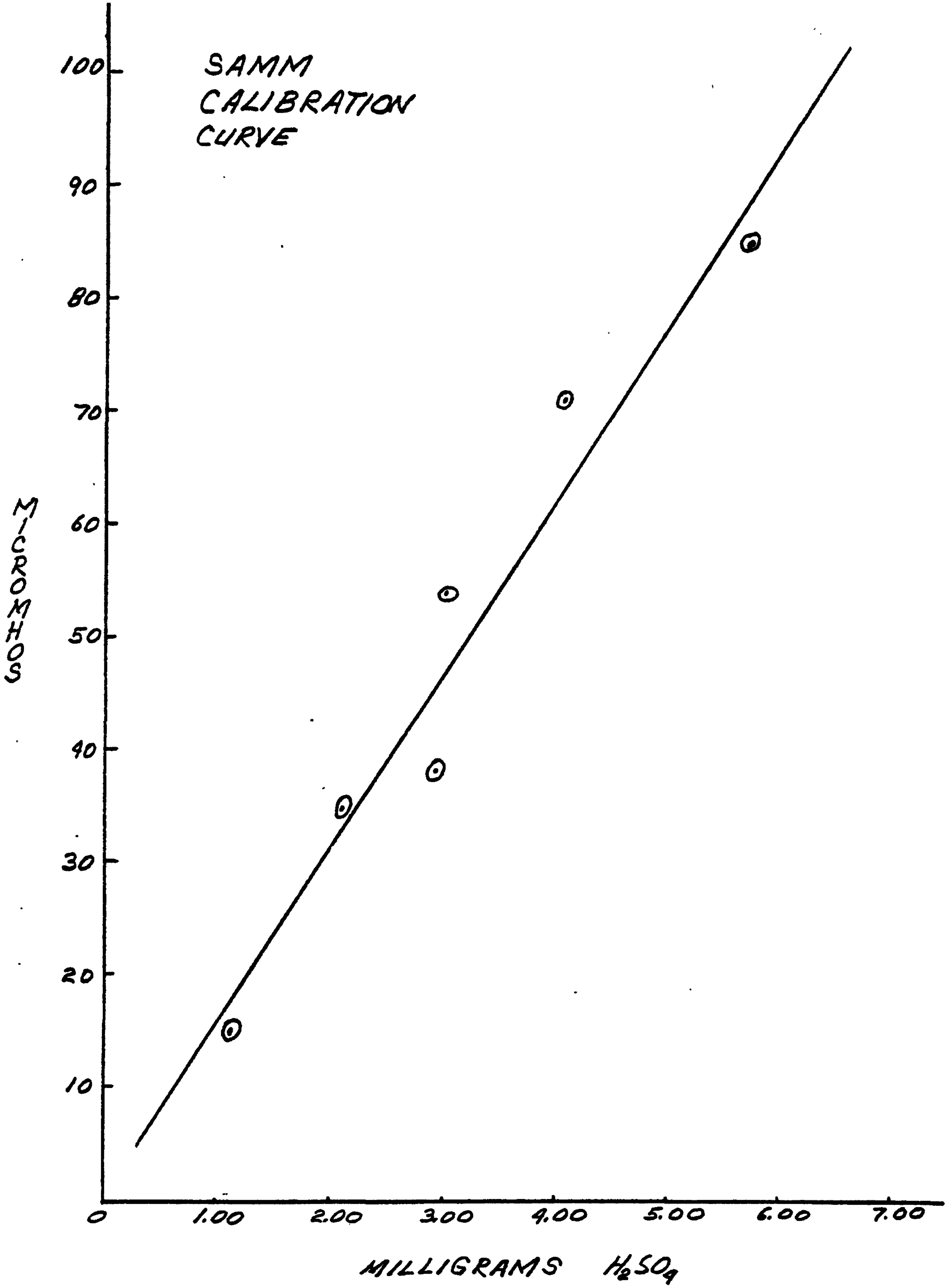
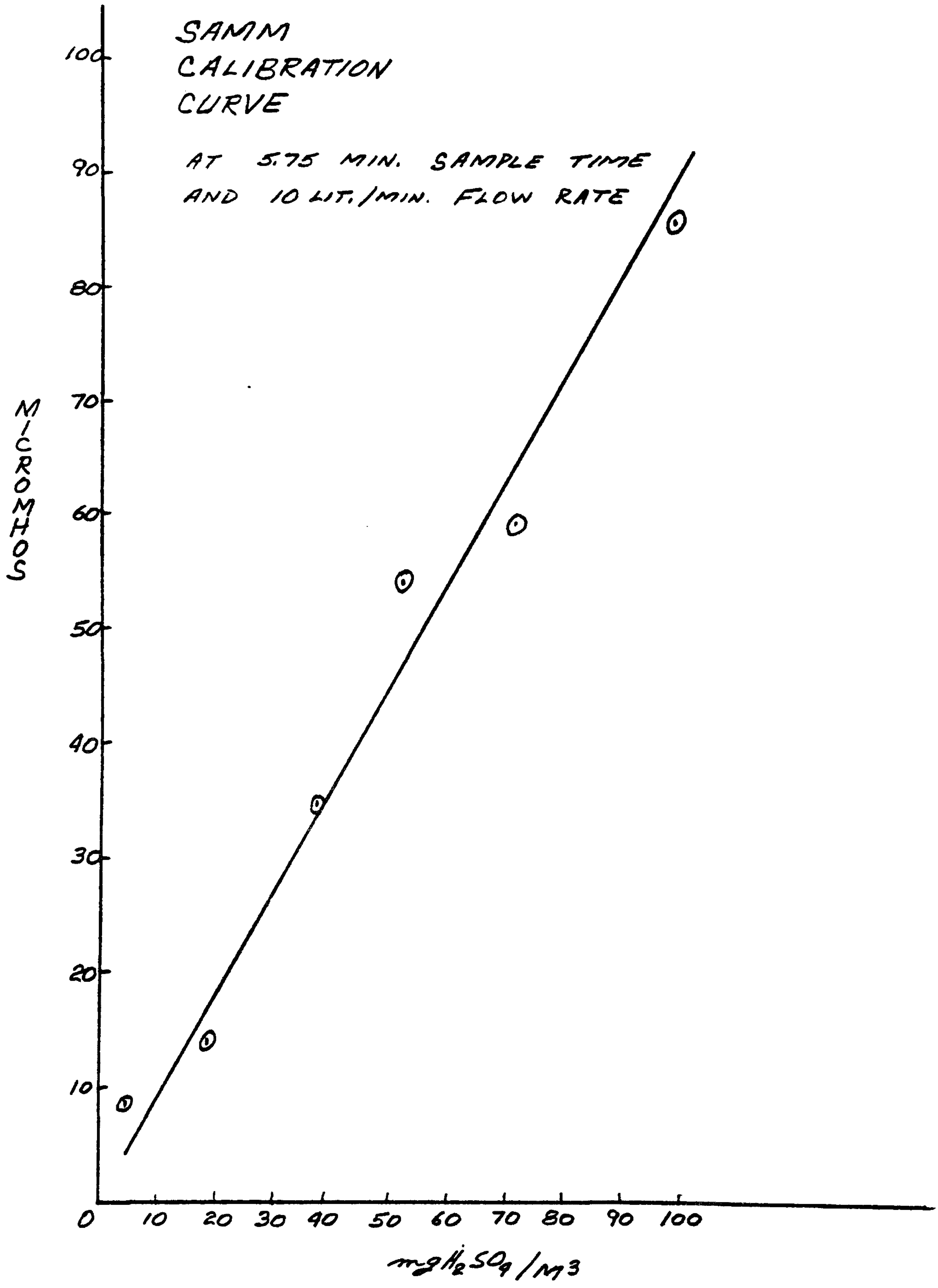

SAMM
CALIBRATION
CURVE
MICROMHOS
100
90
80
70
60
50
40
30
20
10
0
1.00
2.00
3.00
4.00
5.00
6.00
7.00
MILLIGRAMS H_2SO_4

Figure 18



SAMM
CALIBRATION
CURVE
AT 5.75 MIN. SAMPLE TIME
AND 10 LIT./MIN. FLOW RATE
MICROMHOS
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
100
$mg\ H_2SO_4/M^3$

Figure 19

IV. SYSTEM DOCUMENTATION

A. OPERATION AND MAINTENANCE

The operation and maintenance manual is under separate cover. This details the steps for set-up, sampling and maintenance of the monitor.

V. RECOMMENDATIONS FOR MODIFICATION

A. SAMPLE PROBE

It is recommended that the commercially purchased probe be modified by adding new heating material and thermal insulation. Sufficient heating material will be added to assure a temperature of at least 550^{o}F as measured in the air stream at the exit of the probe.

B. COOLING SYSTEM

It is recommended that the spiral condenser system be modified by addition of a closed loop cooling system utilizing a water chiller. This would insure a low enough temperature in the spiral condenser for methanol condensation.

REFERENCES

1. Stern, A. C., "Air Pollution, " 2nd Edition, Vol. 1, Academic Press, N.Y.

2. Richter, E. and Sattleberge, S., "Brennst-Warme-Kraft," 24, 339 (1972).

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(Please read Instructions on the reverse before completing)

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16. ABSTRACT

A prototype sulfuric acid mist monitor has been constructed for the purpose of detecting sulfuric acid-sulfur trioxide. The monitor utilized the selective condensation method with subsequent determination of sulfuric acid by measuring the conductivity of an aqueous isopropanol solution. The instrument is fully automated with a mass flow controller and standard TTL logic to allow for easy modification of the timing circuit. After collection of the H_2SO_4-SO_3 in the temperature controlled spiral condenser, the sample is washed into a conductivity cell for measurement. During conductivity measurement the condensor is rinsed with methanol and air dried prior to the next sample collection. The instrument was designed to measure from 1 to 100 mg/m^3 H_2SO_4 and present the measurement within a 10 mv range. The sample timing cycle can be varied from 105 seconds to 999 seconds.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
* Air pollution * Sulfuric acid * Aerosols * Sulfur trioxide * Monitors * Prototypes		13B 07B 07D

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