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RESEARCH AND DEVELOPMENT OF A CONTINUOUS ATMOSPHERIC MEASUREMENT SYSTEM

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FOREWORD

This report was prepared by Atlantic Research Corporation, Alexandria, Virginia, on Air Force Contract AF 33(615)-1383 under Project No. 6373, "Equipment for Life Support in Aerospace," and Task No. 637302, "Respiratory Support Equipment." The work was administered under the direction of Aerospace Medical Research Laboratories, Aerospace Medical Division. Mr. Richard E. Bennett was technical monitor for the Air Force.

Effort under this contract began in December 1963, and was concluded in December 1964. Dr. Richard H. Johns was Project Engineer for Atlantic Research, and Mr Alton M. McKissick, Jr., was the Development Chemist on the project. Dr. Michael Markels, Jr., Director, Advanced Technology Division, contributed to project administration. The over-all activity was carried out under Dr. Raymond Friedman, Vice President, Research, Atlantic Research Corporation.

This technical report has been reviewed and is approved.

WAYNE H. McCANDLESS
Technical Director
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ABSTRACT

The application of electrolytic hygrometer cells for the measurement of atmospheric moisture and carbon dioxide was investigated. A carbon dioxide—moisture converter was devised. After system feasibility was proved, a laboratory model was built. Some difficulties were experienced with capillary electrolytic cells and flow control. The selection of optimum components is important.

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SECTION I

INTRODUCTION

This project investigated the application of electrolytic hygrometer cells to the measurement of moisture and carbon dioxide in the atmosphere. The commercially available cells consisted of a pair of helical electrodes enclosed in capillary tubing and coated with phosphorus pentoxide. As air is drawn through a cell, moisture is scavenged by the phosphorus pentoxide and is continuously electrolyzed to hydrogen and oxygen by an impressed voltage. The resulting electrolysis current provides a measure of the moisture content of the flowing airstream. Through the use of a chemical converter, carbon dioxide can be converted to an equivalent amount of water and measured by the same technique.

A breadboard model was built to test the operation of the hygrometer cells over a wide range of moisture levels and to investigate the carbon dioxide conversion principle. Difficulties were experienced with several of the components, but the principle of operation of the proposed instrument appeared to be sound. In particular, difficulty was experienced in attaining adequate flow control across the varying flow resistances of the system.

When the preliminary design investigation had been completed using the breadboard instrument, a laboratory model was built and calibrated. Many modifications were incorporated in the redesign, including the adaptation of the system to operation on 28-volt, d-c power. The flow control system was refined to attain a constant and reproducible sampling rate.

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SECTION II

PRINCIPLE OF OPERATION

The principle of measurement in the Atmospheric Monitor is the electrochemical equivalence of a given amount of water to a measurable quantity of electricity. The measured quantity of electricity is the amount used in electrolytic decomposition of water according to the following reaction:

$$H_2O \longrightarrow H_2 + 1/2\ O_2$$

$$q = 2 \times 96,500 \text{ coulombs/mole} \tag{1}$$

Hence, if a quantity of water is absorbed from an air sample and completely electrolyzed, each mole (18 grams) of water is represented by a current-time integral of 193,000 coulombs.

If the water to be determined is a component of a flowing air stream, the measurement can be made even more simply, provided that the flow rate is constant. In this case, the instantaneous current level indicates the mole fraction or volumetric fraction of water in the stream. For example, a flow of 20 ml/min of air containing 1.0 percent moisture by volume produces a current of 28.6 ma. The value is calculated as follows:

$$1.0\% \text{ } H_2O \times 20 \text{ ml/min} = 0.2 \text{ ml/min } H_2O$$

$$\frac{0.2 \text{ ml/min } H_2O}{22,414 \text{ ml/mole}} = 8.9 \times 10^{-6} \text{ mole/min } H_2O$$

$$8.9 \times 10^{-6} \text{ mole/min} \times 193,000 \text{ coul/mole} \times 1/60 \text{ } \frac{\min}{\sec}$$

$$= 2.86 \times 10^{-2} \text{ coul/sec} \cong \text{ amperes} \tag{2}$$

The absorption-electrolysis cell is the heart of the Atmospheric Monitor. It consists of a 26-inch plastic capillary on whose inner wall are wound a pair of helical rhodium wire electrodes. The electrodes are coated with a thin layer of phosphorus pentoxide, a strong water absorbent. The entire capillary is formed into a helix and potted in an outer metal casing.

As a moist gas is passed through the absorption-electrolysis cell, the water reacts instantaneously with the phosphorus pentoxide layer to form phosphoric acid, a strong electrolyte. A voltage impressed across the electrodes causes electrolysis of the acid, restoring the system and liberating an amount of hydrogen and oxygen equivalent to the water absorbed. Meanwhile, the current in the electrolysis circuit is a measure of the transient amount of water being absorbed in the cell.

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A chemical converter enables the electrolysis principle to be used similarly for the determination of carbon dioxide. The effluent stream from the moisture determination, now dry, is passed through a heated tube packed with lithium hydroxide. Each molecule of carbon dioxide is exchanged for a molecule of water according to the following reaction:

$$CO_2 + 2LiOH \longrightarrow Li_2CO_3 + H_2O \tag{3}$$
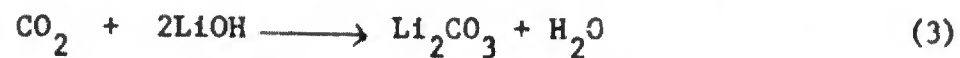
The stream containing the liberated water is fed to a second absorption-electrolysis cell and the water is measured exactly as before.

SECTION III

SYSTEM DESIGN, COMPONENT SELECTION, AND ASSEMBLY

One of the major problems involved in designing new equipment is the selection of components best suited for the job they are to perform. Thus, much time was devoted to the investigation and selection of the components for this system. Consideration was given to such factors as weight, power requirements, size, durability, and cost.

The choice of cells for the Atmospheric Monitor was dictated by several factors: Standard Swagelok fittings are used, making for easy connections; the electrodes are made of rhodium rather than platinum, said to lessen the possibility of reverse reactions; and the outer construction is of a very sturdy and durable design.

After long operation, a fault was revealed in the construction of the absorption-electrolysis cells employed, which might have been prevented by the use of glass capillary tubing. The capillary tubing, made of Teflon in cells employed, tends to soften slightly with normal cell heating, causing a change in flow resistance.

Because carbon dioxide converters were not commercially available, one was designed and built in our laboratory. It consists of a nickel pipe nipple (1/4-inch pipe $\times$ 7 inches length) heated by an external heating jacket. The tube is simply packed with lithium hydroxide and controlled to 150° C by a thermostat. The 5-gram charge of lithium hydroxide has a theoretical conversion capacity of 0.10 mole carbon dioxide; this amounts to 1866 hours continuous instrument operation at 0.1% carbon dioxide concentration and 20 ml/min flow. Tests showed that complete conversion was obtained for at least 80 percent of the theoretical converter life. Thus the converted should be recharged every 1500 hours if the average measured carbon dioxide level is 0.1 percent or every 300 hours if the average measured level is 0.5 percent.

As a precaution against erroneous carbon dioxide measurements, a drying tube was provided ahead of the carbon dioxide converter. Thus a malfunction of the moisture measuring system is prevented from affecting the carbon dioxide system. It is good practice to recharge the drying tube with calcium sulfate each time the converter is recharged.

An area of unexpected problems lay in the design of the flow control and metering system. A fundamental difficulty is the lack of commercial availability of components such as pumps, valves and regulators designed for the very small gas flows needed in the Atmospheric Monitor. A diaphragm-action pump was chosen for sampling the air into the instrument. The pump is oversized by a factor of at least five in capacity, but is the smallest available. The substitution of a smaller pump, now under commercial development, may result in a considerable reduction in the weight and power consumption of the instrument.

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The airflow was controlled by fine metering valves frequently used to control small gas flows in gas chromatographs.

A differential flow controller was incorporated in the breadboard model to provide flow stability. It was eventually discarded, however, because it tended to break into oscillation, causing wide fluctuations in gas flow. Flow stability was finally achieved using two needle valves, affording flow regulation in two stages as described in the instruction manual accompanying the instrument.

The breadboard model Atmospheric Monitor is shown in front and rear fiews in figures 1 and 2. The first instrument was set up in this manner to permit open accessibility to components and easy adaptability to design changes. This instrument operated on 115 volts a-c; the d-c electrolysis current was supplied by a solid-state, line-operated power supply, regulated to 75.0 volts ±0.05 percent.

A series of standard concentrations of moisture and carbon dioxide was used for instrument tests and calibration. For the carbon dioxide measurements, two premixed samples of carbon dioxide in nitrogen were prepared in gas cylinders. These had nominal concentrations of 0.5 and 2.0 percent by volume, and were analyzed in our laboratory by mass spectrometry. The preparation of moisture standards is problematic, and this task was approached in two ways. First, an accurate, blower-equipped hygrometer was obtained so that ambient air under varying weather conditions could be utilized as a calibration standard. Second, an apparatus was devised to saturate air with water at the ice point. Satisfactory equilibrium is difficult to achieve, but was finally accomplished using two gas bottles in series, followed by a glass wool filter to trap entrained water. The entire apparatus was kept submerged in an ice bath.

Calibration curves of flow rate versus response were run with each standard to determine the range of linearity of the instrument. In general, linearity is good up to concentrations of 2 percent moisture or carbon dioxide by volume when the flow rate is maintained at 20 ml/min. The range may be increased somewhat by correspondingly decreasing the flow rate. Typical calibration curves for moisture and carbon dioxide are shown in figures 3 and 4, respectively.
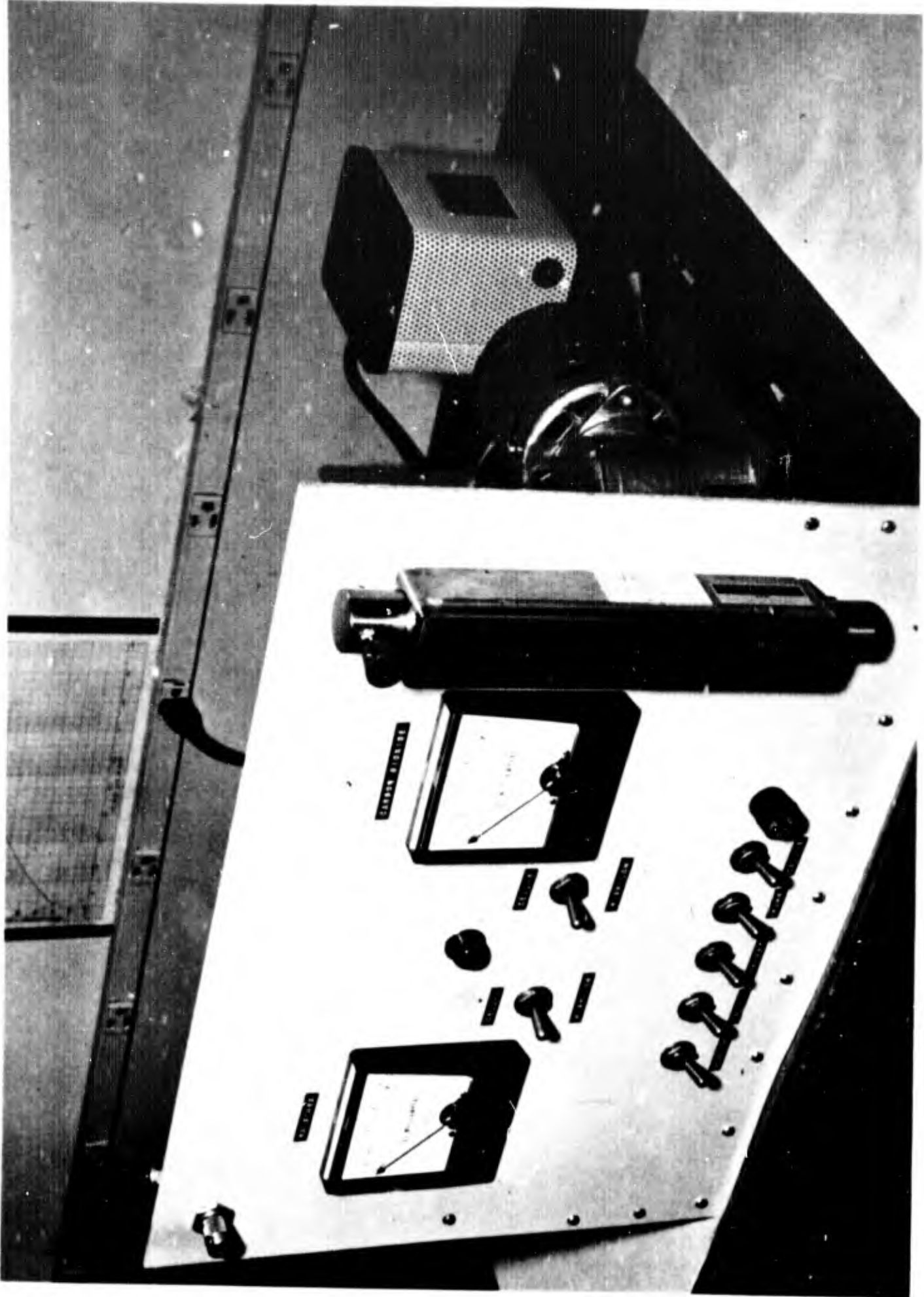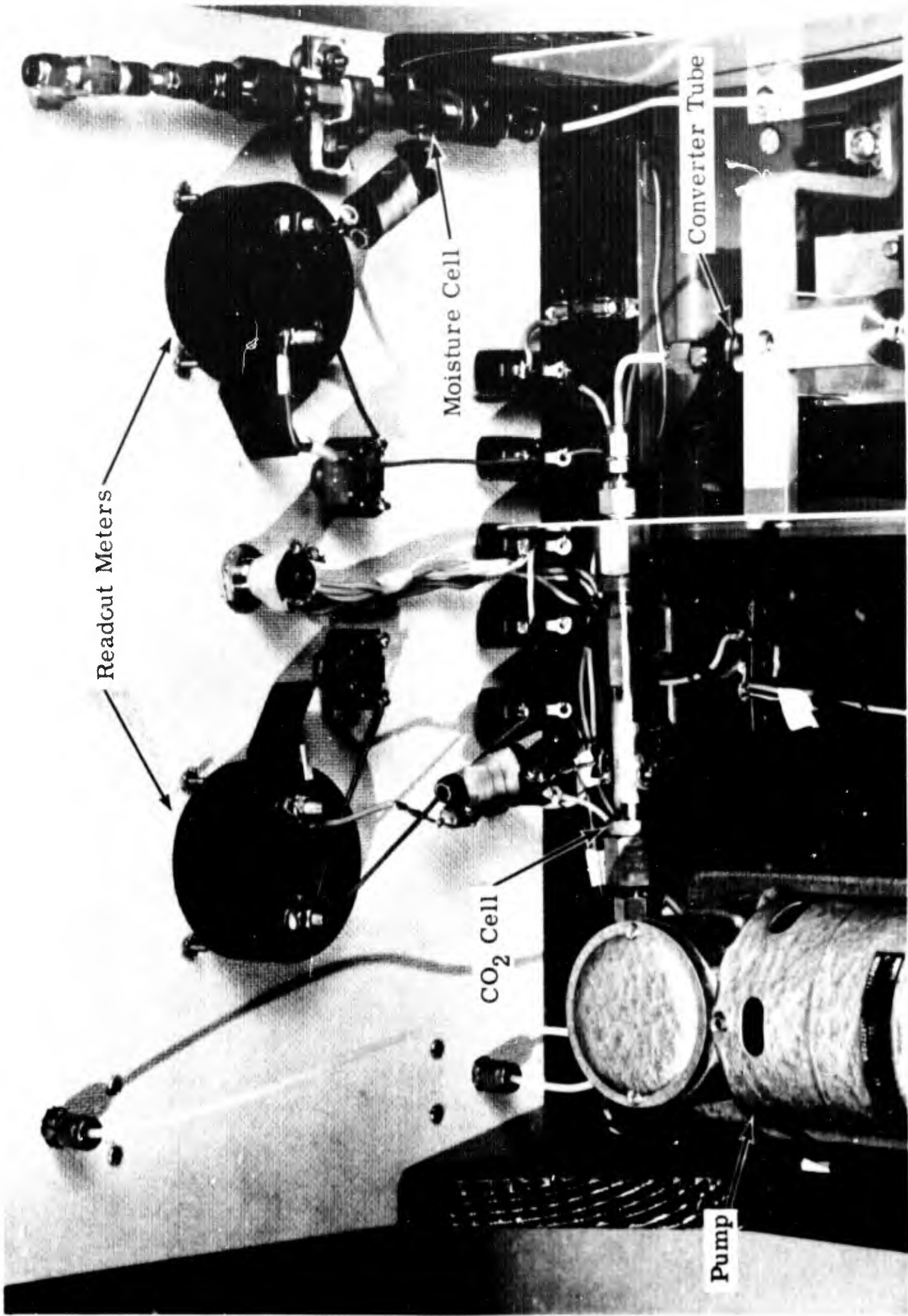
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Figure 1. Front View of Breadboard Monitor.
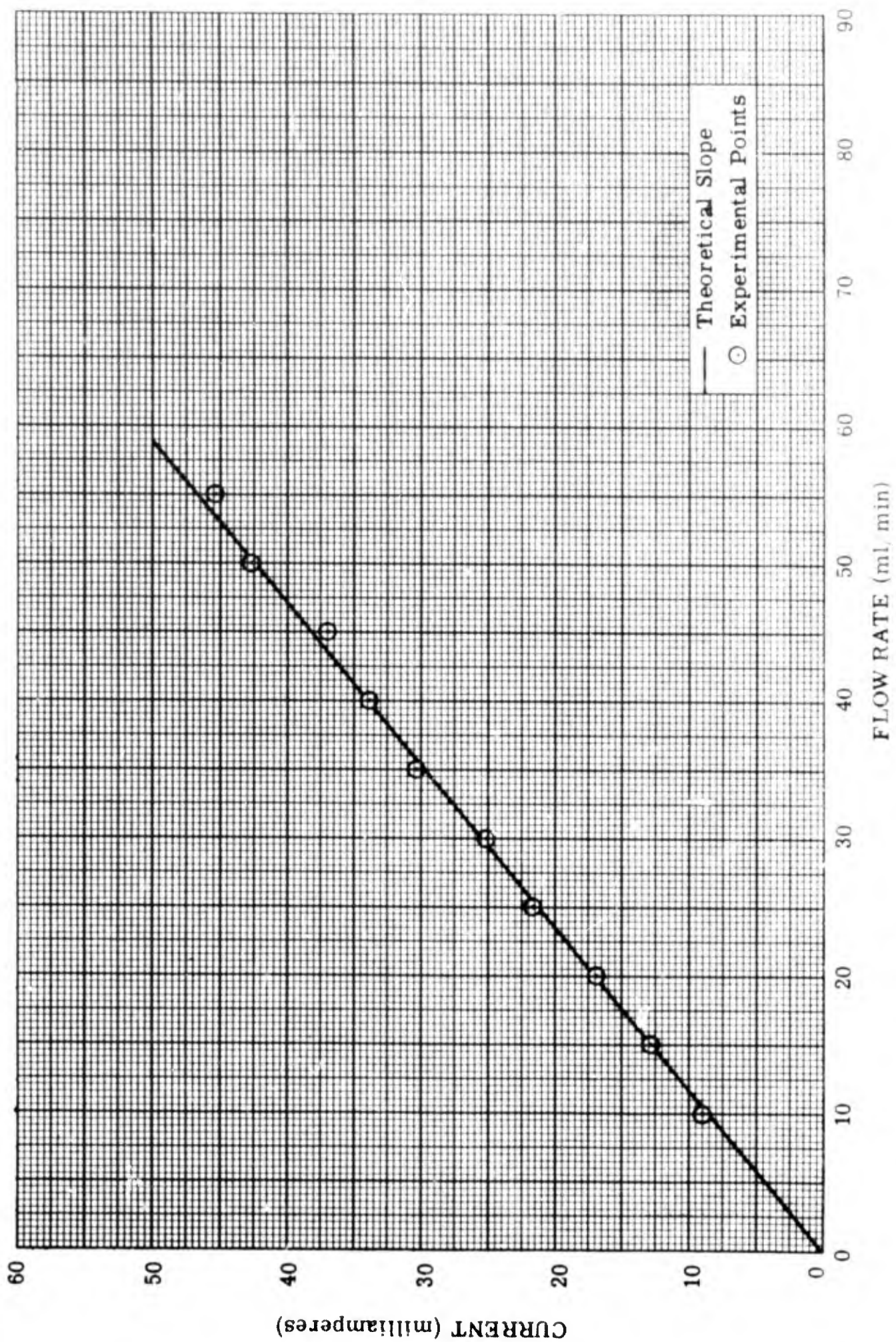
Figure 2. Rear View of Breadboard Monitor.

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Figure 3. Current Versus Flow Rate. Air Saturated at 0°C.
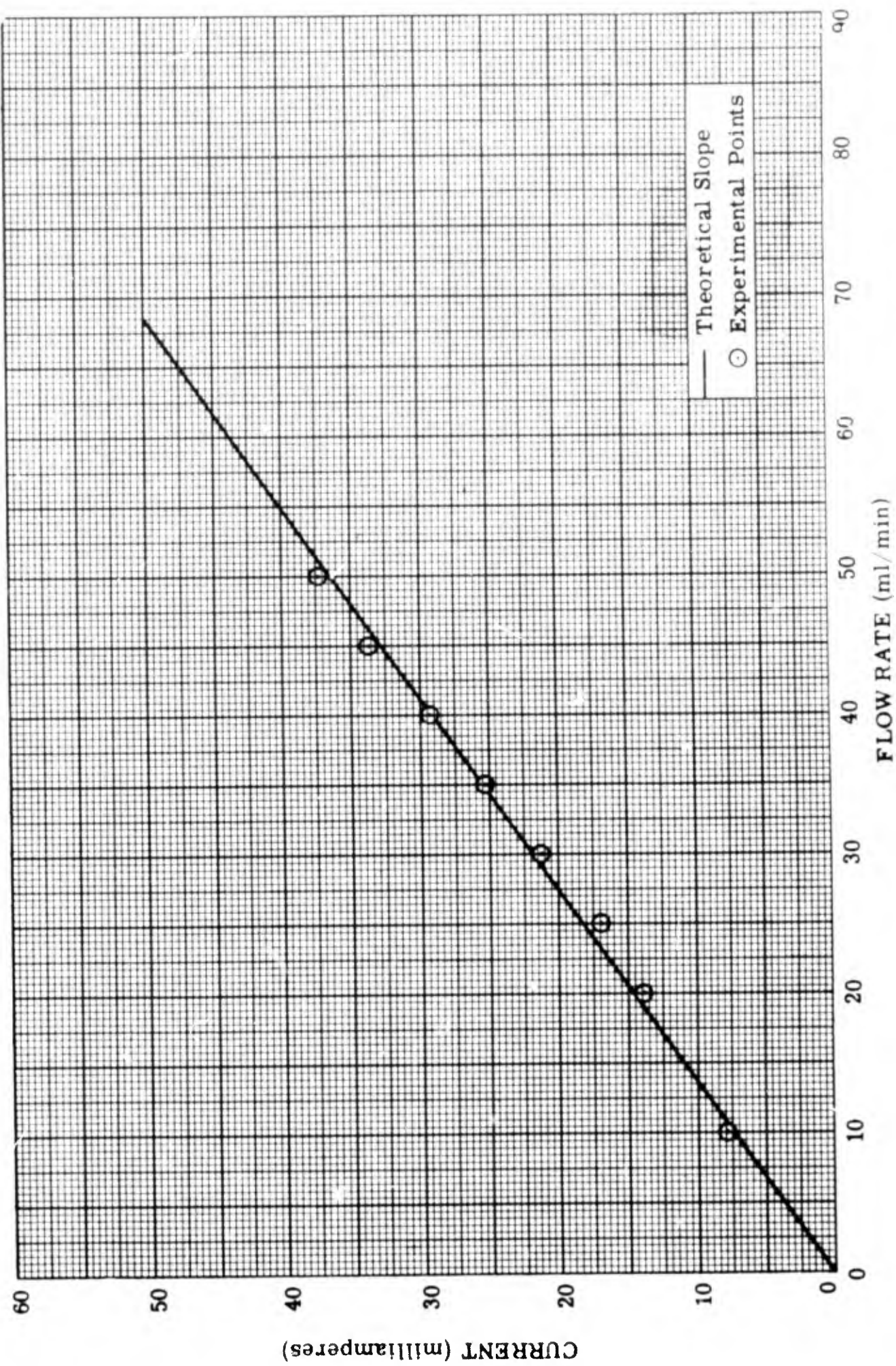
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Figure 4. Current Versus Flow Rate. 0.51 Per Cent Carbon Dioxide in Nitrogen. Converter Temperature 150°C.

SECTION IV

INSTRUMENT ASSEMBLY AND SPECIFICATIONS

The laboratory model Atmospheric Monitor, an evolution of the breadboard model, is shown in figure 5. All of the components of the system are designed for operation on 28 volts d-c. The electrolysis voltage is obtained directly from the d-c supply, but is regulated to 22.0 volts by a pair of Zener diodes so that fluctuations in supply voltage will not affect the instrument response. The readout meters are from 0 to 10 ma Simpson meters, provided with switchable hand-wound shunts to give a range increase from 0 to 100 ma. The electrical circuit of the Monitor is shown in figure 6.

Figure 7 shows the flow system. The system uses the same components as the breadboard model in a somewhat different arrangement. A brass manifold, mounted on the inside rear panel, holds the electrolytic cells, converter, and drying tube in an easily accessible position.

Figures 8 and 9 show interior views of the instrument and clarify the placement of components. The two cells must be positioned as shown in figure 9. All flow connections are made with 1/8-inch Teflon tubing and Swagelok compression fittings.

Since it was necessary to place the flowmeter in the system at a point of reduced pressure, the calibration of the meter is arbitrary. It is so calibrated that a setting of 6.0 on the flowmeter scale represents a flow of 20 ml/minute at the inlet of the instrument. Recalibration is best done by means of an external displacement meter.

The final calibration charts for the instrument, given in the instruction manual, were drawn from calibration points obtained with standard mixtures.

The temperature of the carbon dioxi⟨...⟩ noncritical. At a temperature of 120° C, conversion was f⟨...⟩ plete, whereas a temperature of 175° C produced no improvem⟨...⟩. Accordingly, the heating jacket was set to maintain 150°

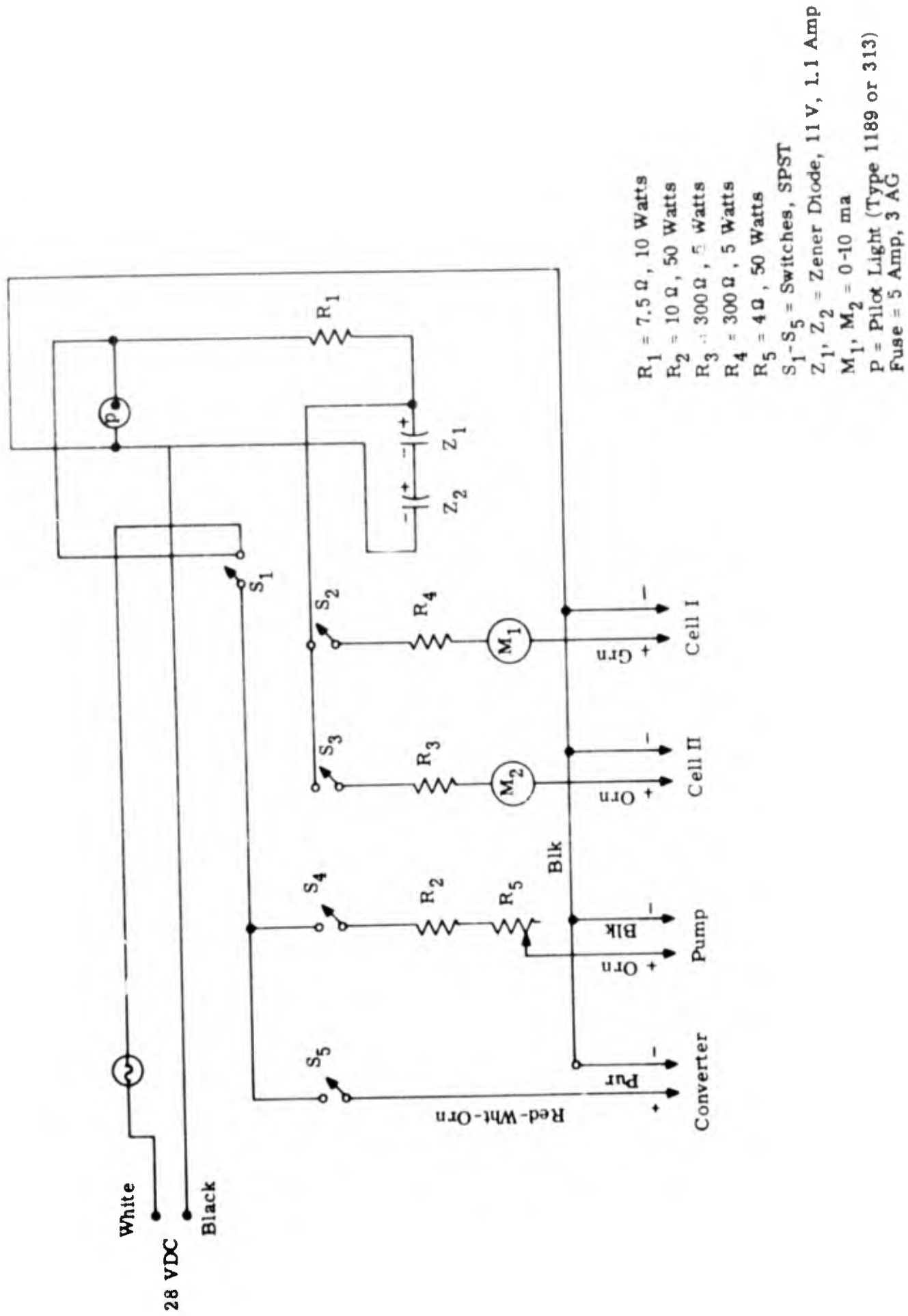Figure 5. Laboratory Model Atmospheric Monitor.

R₁ = 7.5 Ω, 10 Watts
R₂ = 10 Ω, 50 Watts
R₃ = 300 Ω, 5 Watts
R₄ = 300 Ω, 5 Watts
R₅ = 4 Ω, 50 Watts
S₁-S₅ = Switches, SPST
Z₁, Z₂ = Zener Diode, 11V, 1.1 Amp
M₁, M₂ = 0-10 ma
P = Pilot Light (Type 1189 or 313)
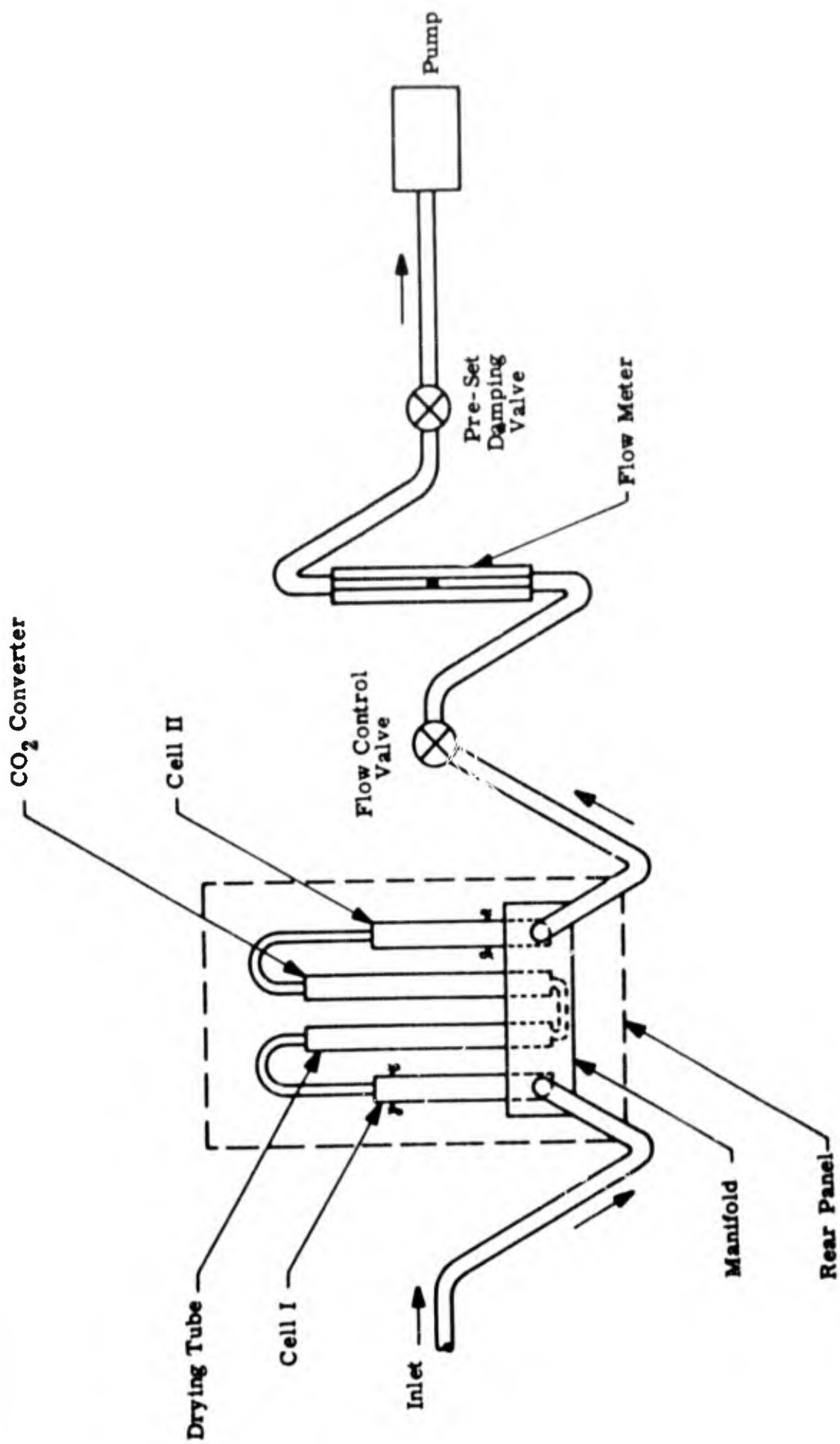Fuse = 5 Amp, 3 AG

Figure 6. Electrical System

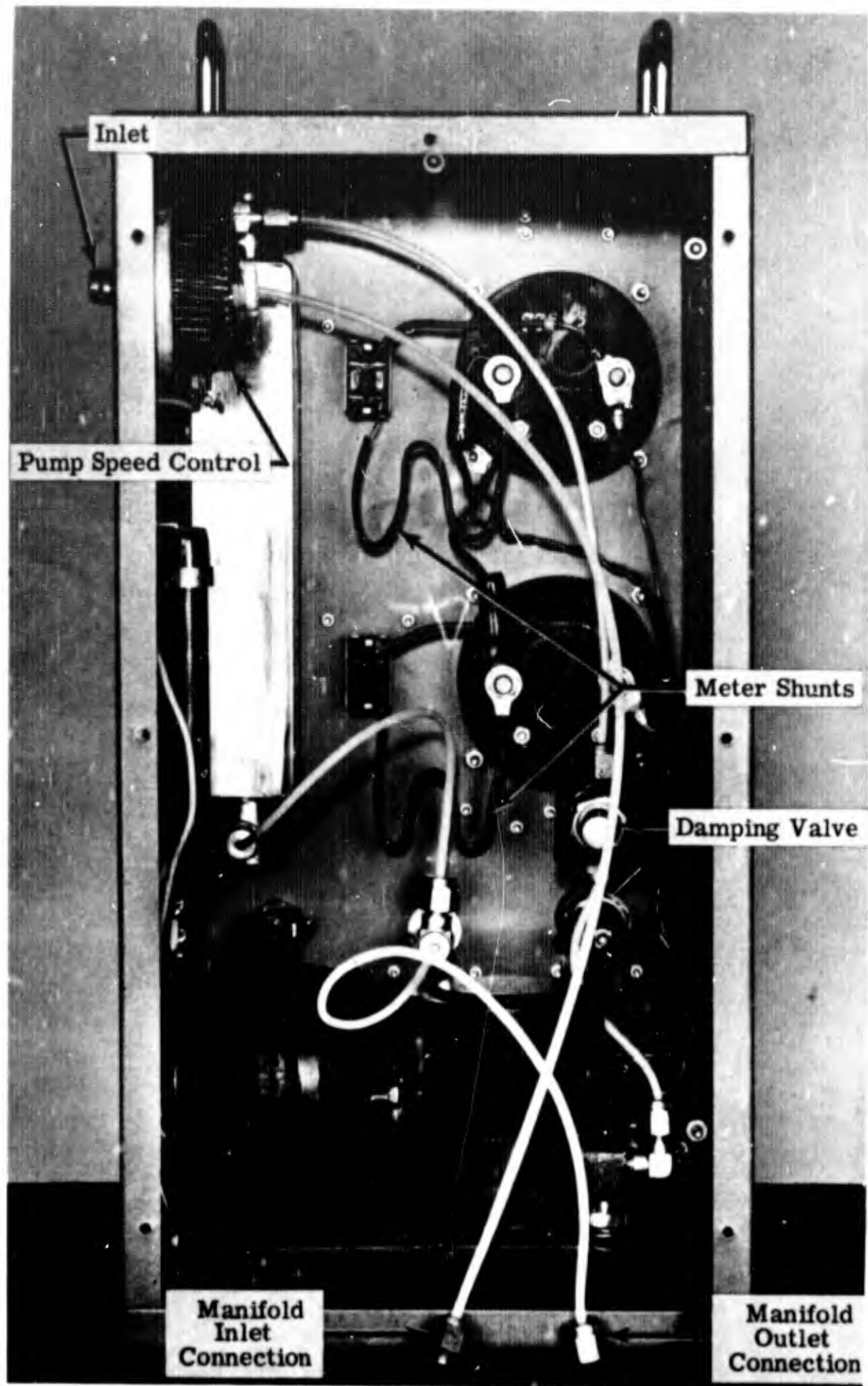Figure 7. Flow System.
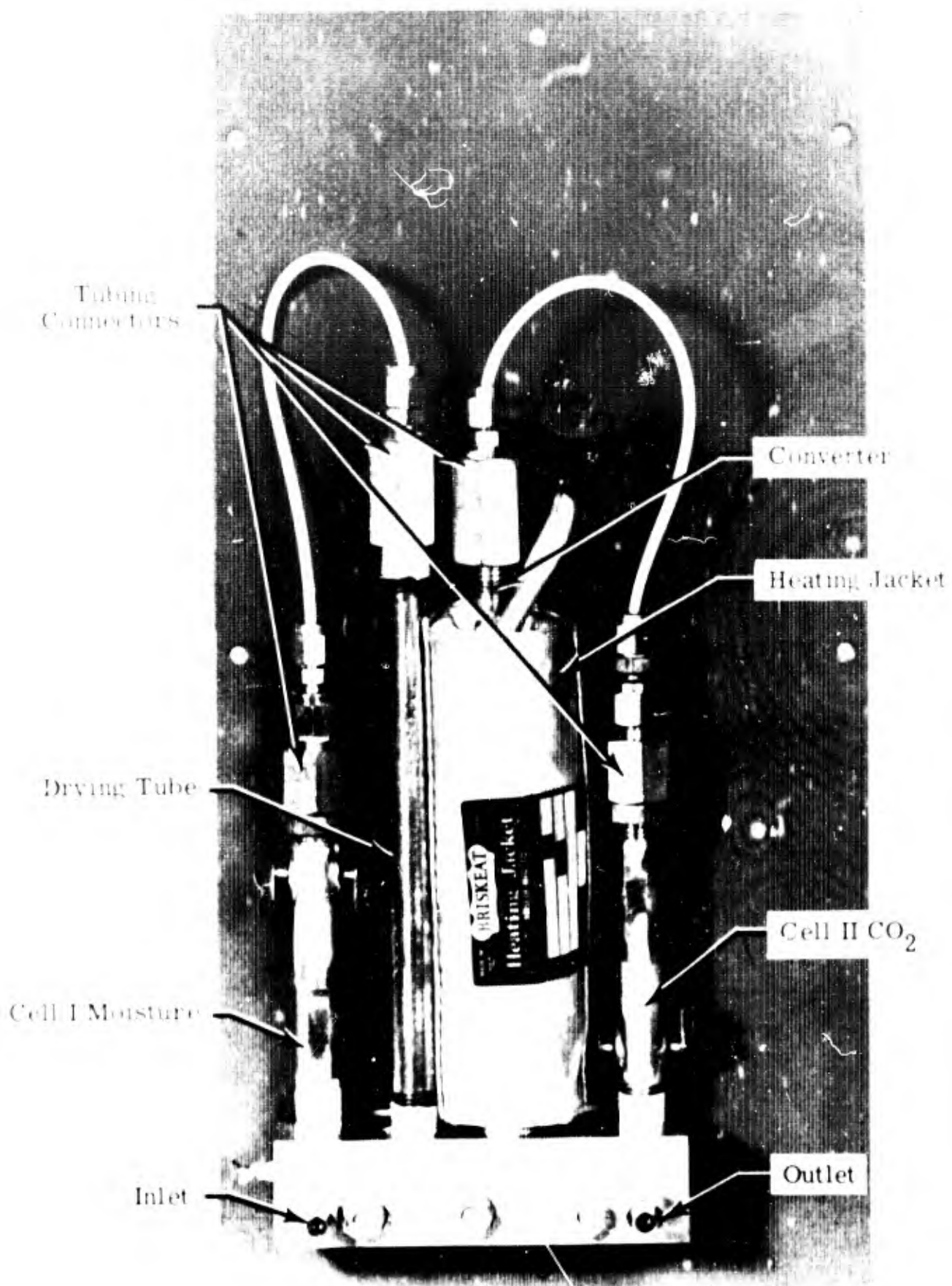
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Figure 8. Interior of Atmospheric Monitor.

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Figure 9.   Positioning of Cells.

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

Experimental work indicated that the electrolytic method for the measurement of atmospheric moisture and carbon dioxide is feasible. A workable carbon dioxide—water converter was devised which functioned reliably throughout.

Moisture is classically difficult to measure because of the problem of attaining a totally anhydrous background in the test environment. Any moisture-measuring system is subject to atmospheric moisture adsorption during periods of idleness, and precautions must be taken to avoid the influence of adsorbed moisture on subsequent measurements. In the Atmospheric Monitor, care must be taken to avoid cell flooding from condensed or adsorbed moisture entering the instrument while it is not in use. A heated inlet system could be installed to help solve this problem in the laboratory model, although this should not be necessary in the space application where continuous operation is assured.

The development of electrolytic hygrometer cells has not been sufficiently advanced to realize the full potential of this device. Presently available cells are designed primarily for low levels of moisture, and the need is evident for a macro cell.

The availability of suitably small pumps, valves and flow control systems is a problem in the development of air-sampling instruments. A new type of maniature pump is being commercially developed. Within the space capsule, pressure differentials undoubtedly exist across various points in the air-circulation system. Utilization of such a differential for sampling should eliminate the need for a pump.

Future efforts on an electrolytic atmospheric measurement system should be directed toward improved components, particularly cells. Miniature pumps and other components are necessary to turn the present instrument into flight-weight hardware. Except for these components needs, the instrument has excellent potential for an in-flight continuous measurement system.