ENGINEERING RESEARCH INSTITUTE

COLLECTION AND ANALYSIS OF UPPER AIR SAMPLES

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by

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COLLECTION AND ANALYSIS OF UPPER AIR SAMPLES

1. INTRODUCTION

This is the first in a series of Quarterly Reports on Contract No. DA-36-039 SC-56737 describing an experimental program of collecting and analyzing upper-air samples. The work is a continuation of one phase of a program of upper-air research which has been carried out since 1946 by the University for the Meteorological Branch of the Signal Corps. The other phase of the work, that of measuring pressure, density, temperature, and winds, will continue on a separate contract. For background material, the reader is referred to the Final Reports of Contracts W-36-039 SC-32307, DA-36-039 SC-125, and DA-36-039 SC-15443. The latter report summarizes the current status of the sampling program, the principal objective of which has been the investigation of diffusive separation.

2. PURPOSE

The purpose of the research as given in Signal Corps Technical Requirements SCL-2370 of 19 January 1954 is as follows:

"This specification covers the research into the necessary techniques for the collection and analysis of air samples in the region of 30 to 100 km altitude and a continuing review of the field of upper air research for the purpose of keeping in contact with work of interest to the Signal Corps.

"The techniques shall be confirmed by field experiments using Aerobee or other rockets as vehicles. Emphasis shall be placed on the following experiments.

"(a) The analysis of the upper air and control samples using the gas adsorption analysis and/or other techniques.

"(b) The collection of samples in the region 30 to 100 kilometers.

"(c) The performance of subsidiary experiments, reduction of data, calculation of results and preparation of reports."

3. ABSTRACT

Progress in the construction of a selective adsorption analyzer for upper-air samples is described. Development of a new method of constructing control and upper-air sample bottles is described. Continuing investigations of the effect of sampling on composition and of possible new analysis techniques are noted.

4. NEW ANALYZER

The necessity for constructing a new analyzer, the reason for choosing a charcoal adsorption type, and the design of the new analyzer were discussed in Progress Reports of the previous contract. The work during the quarter was devoted to the construction, testing, and assembly of components. With one exception, the construction of the analyzer proceeded without difficulty according to design. Fig. 1 is a schematic of the analyzer and Fig. 2 a view of the nearly completed apparatus. Figs. 3 to 9 show details of various parts of the analyzer.

The transfer of the gas from the upper-air sample bottle to the analyzer storage vessel has been a difficult problem. A toepler pump was used in the Durham and first Michigan analyzers. Becuase it is slow and requires 24 pounds of mercury for an 800-cc pump (which imposes a mechanical problem), it was hoped to replace the toepler. The best possibility seemed to be a mercury-vapor pump, either single- or multistage. Tests showed that either pump would transfer the samples quickly without contamination. Soft-glass models of each pump were made with some difficulty, and a three-stage mercury-vapor pump was installed in the analyzer. On two trials the pump fractured at the bottom mercury-return joint where the temperature gradient is highest. A successful soft-glass pump for this application presumably could be worked out. However, it was decided that something else should be tried in order to get the analyzer in operation.

Various alternatives were then considered. One of these, charcoal-trap pumping, was tried. It was found to work, but had no advantage in speed over the toepler in transferring helium. Other pumps such as mechanical displacement, Archimedes, carrier-gas toepler, etc., were temporarily rejected as involving considerable development. It was decided to defer the development of a fast transfer system and install a large (800 cc) toepler with automatic operation. This adds 3 to 4 hours to an analysis time. However, the other time-saving features of the new analyzer, i.e., bake-out oven, small size, and final ionization gauge, will still make it possible to perform an analysis much more rapidly than heretofore.

A commercially available pirani gauge, RCA 1947, was purchased. The gauge, which is in a soft-glass envelope, will be used in a temperature-compensating bridge circuit to monitor the sample-bottle pressure when the bottle is attached to the analyzer. Two gauge tubes and the electric controlling device are seen in Fig. 9.

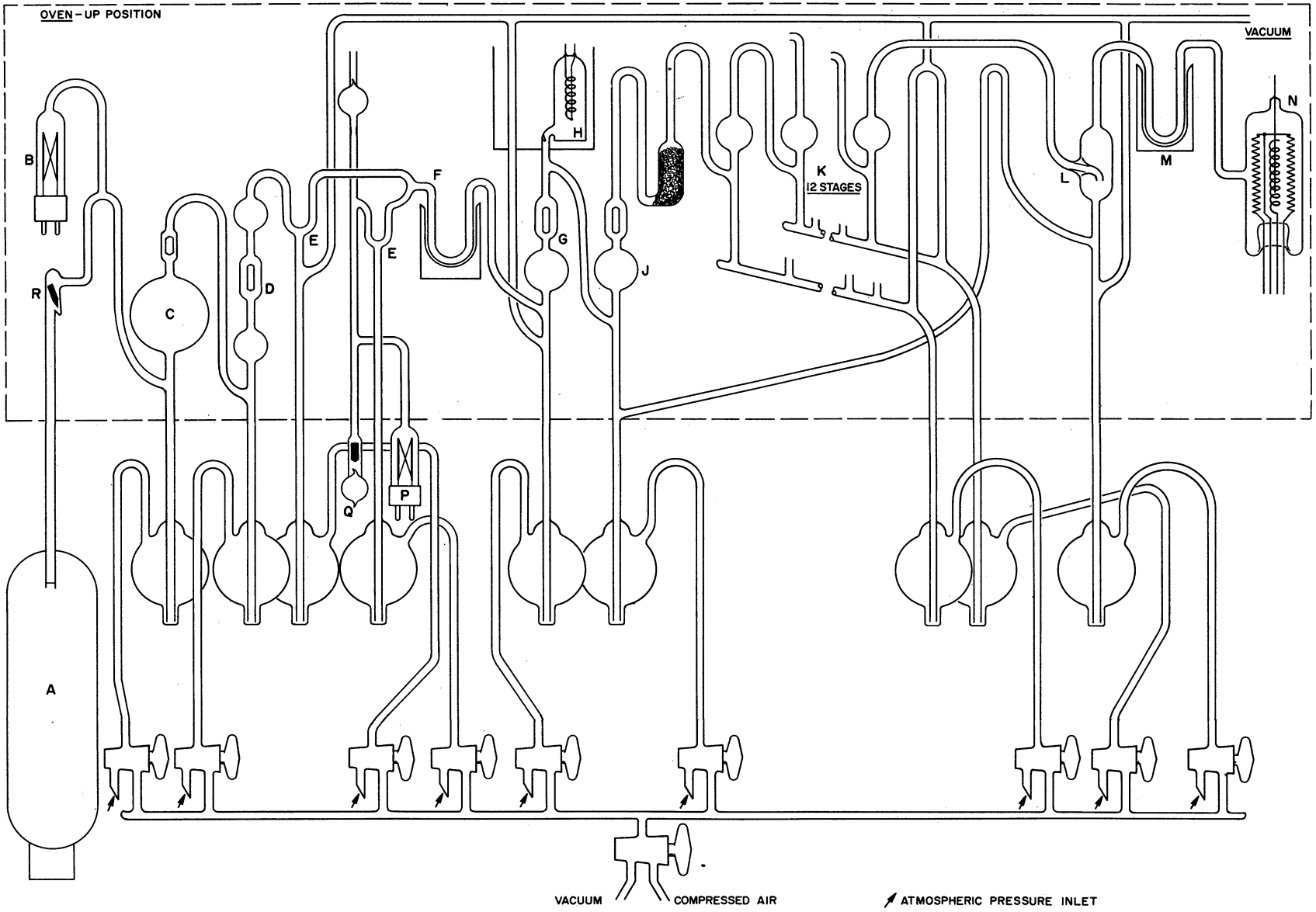

Fig. 1. Schematic of New Analyzer. A. Sample, B. Pirani Gauge for Bottle Pressure, C. Large Toepler Pump, D. Small Toepler and Storage Vessel, E. Mercury Cut-Offs, F. CO_2 Cold Trap, G. Oxygen Cell Toepler, H. Oxygen Cell, J. Column Toepler, K. Fractionating Column, L. Pipette, M. Ion Gauge Cold Trap, N. Ion Gauge, P. System Pirani Gauge, Q. Control Sample Vial. R. Breaker, Mercury containers and stopcocks in the lower part of the diagram are for operating the pumps, valves, or columns to which they are connected.

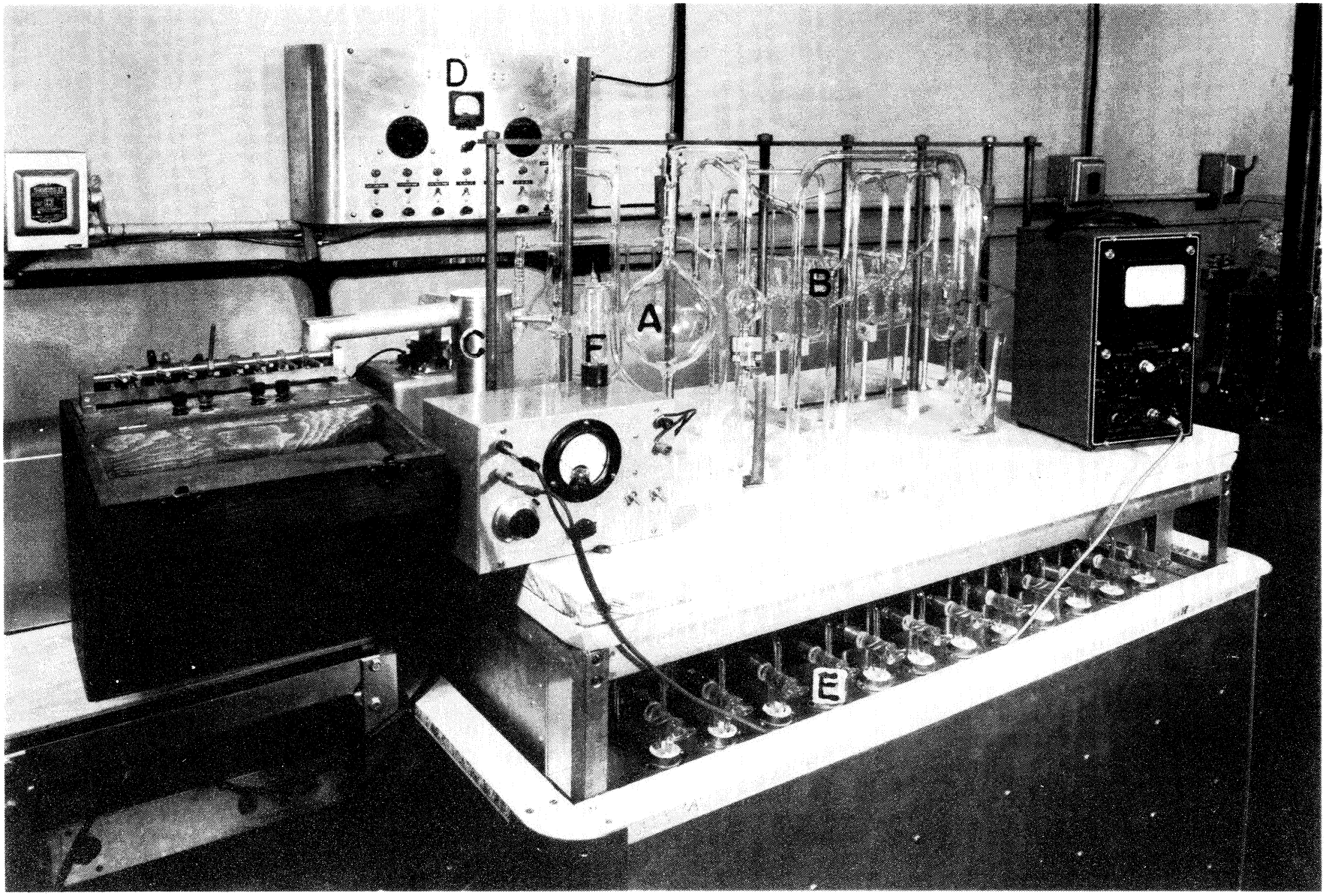

Fig. 2. New Analyzer. A. Large Toepler, B. Fractionating Column, C. Ion Gauge Shield, D. Analyzer Electrical Control Panel, E. Analyzer Operating Stopcocks, F. Pirani Gauge.

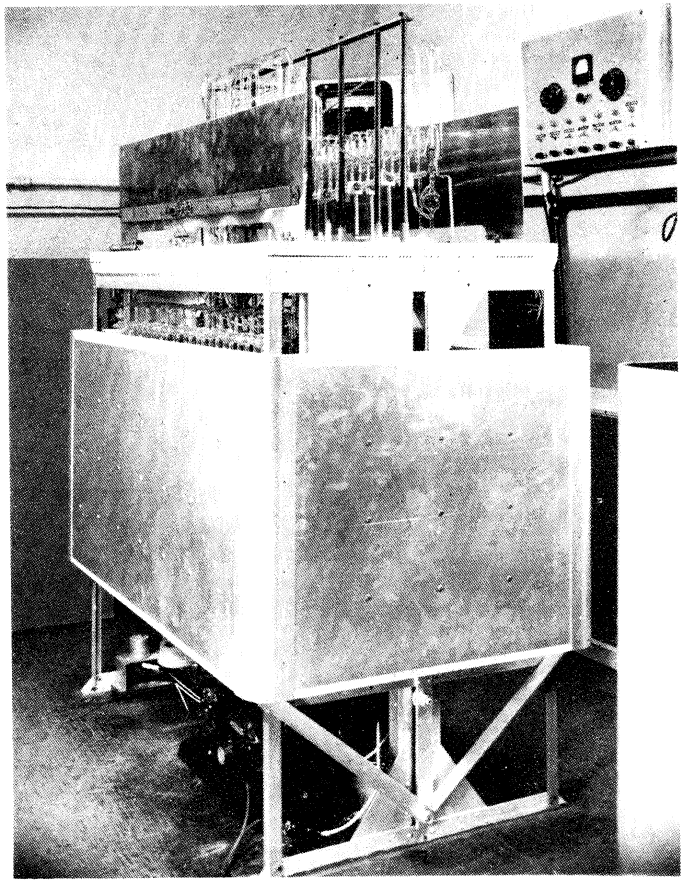

Fig. 3. New Analyzer. Oven in lowered position showing plug-in strip heaters and convection plates. Oven control is behind the analyzer. Electrical control panel is in the upper right corner.

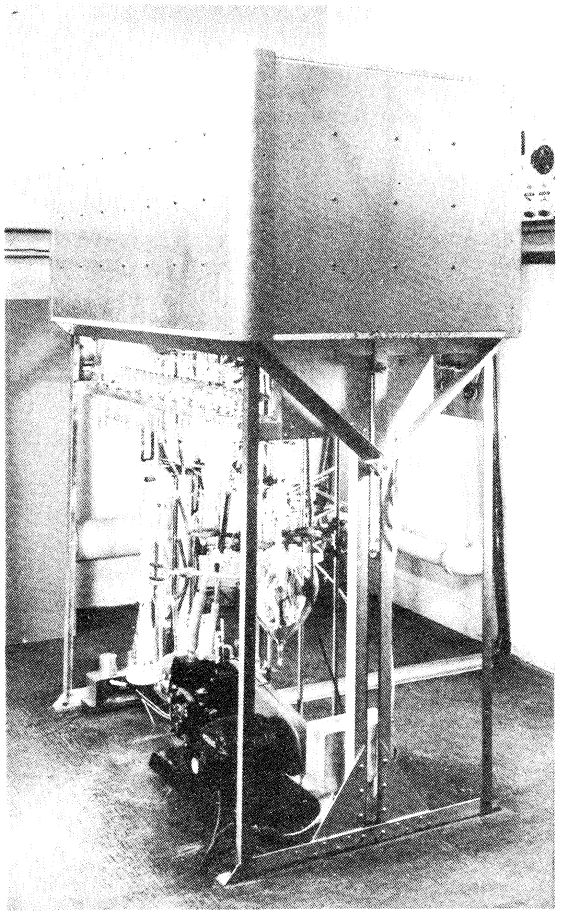

Fig. 4. New Analyzer. Oven in raised position showing main mechanical and diffusion pumps. Analyzer-operating stopcocks are in a row just below the oven.

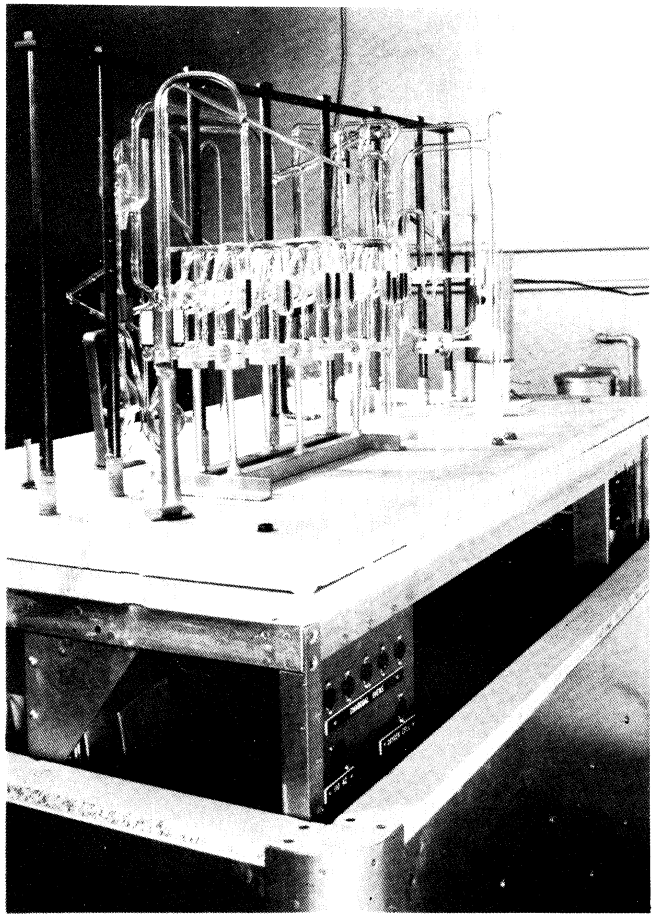

Fig. 5. Details of Column and Charcoal Oven Panel.

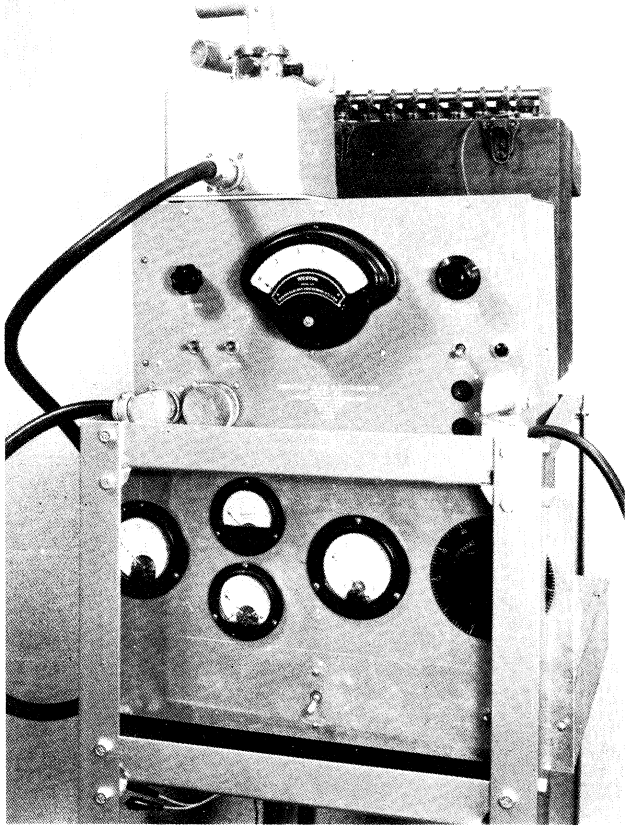

Fig. 6. Final Ion Gauge Electrometer Control Circuit.

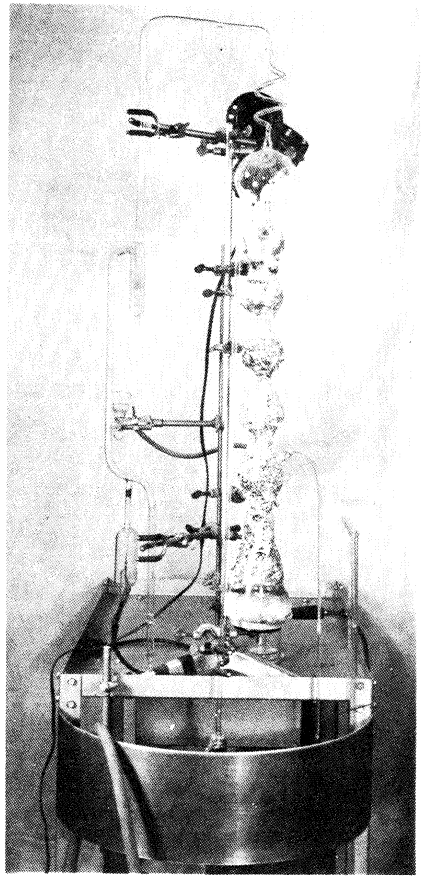

Fig. 7. Mercury Still.

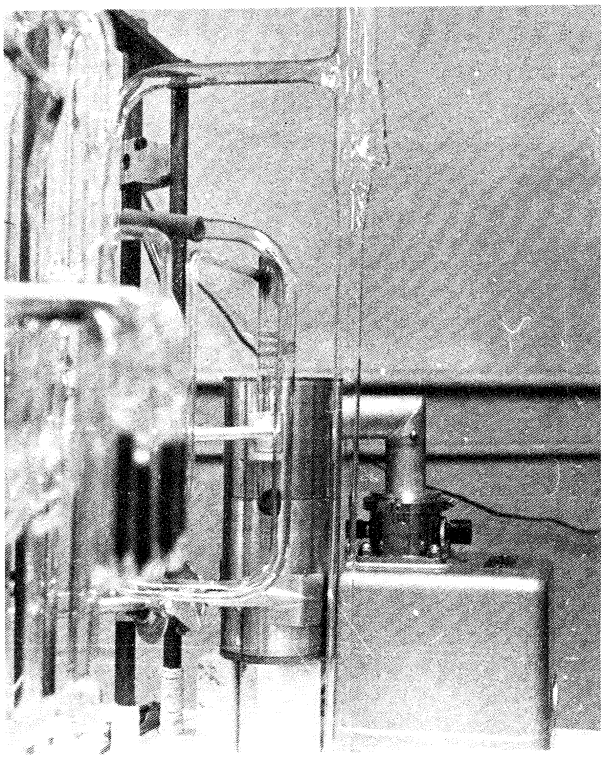

Fig. 8. Ion Gauge and Electrometer Input Box.

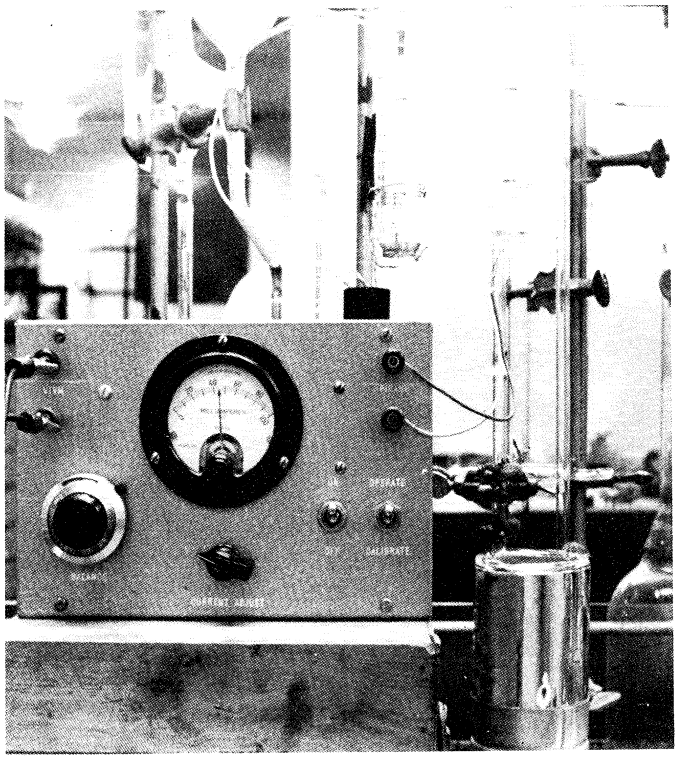

Fig. 9. Measuring and Compensating Piranis and Control Box.

The distribution coefficient* of charcoal for a gas determines the percentage of gas that will be transferred in each cycle of operation of a pumping device using active charcoal at the temperature of liquid nitrogen as a pumping medium.

The distribution coefficients of charcoal for helium, neon, and nitrogen are shown below. The "calculated" values were calculated from experimental values by Glückauf[1] to accommodate the fact that distribution coefficient is a function of the amount of gas and charcoal used. The measured values were obtained at Michigan during the quarter.

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* Distribution Coefficient $= \dfrac{V_g}{V_g + V_a}$

where V_g = vol. of gas in gaseous phase

V_a = vol. of gas in adsorbed phase at same press. and temp. of V_g.

1 E. Glückauf, "A Microanalysis of the Helium and Neon Contents of Air," Proc. Roy. Soc., A 185 (1946), p. 98.

Gas	Volume of Gas Phase (ml)	Weight of Charcoal (gm)	Distribution Coefficient
Helium	8200	5	0.9935 calc.
	8200	50	0.94 "
	8990	20	0.977 "
	8990	20	0.986 meas.
Neon	8200	5	0.924 calc.
	8200	50	0.583 "
Nitrogen	785	20	0.0005 meas.

A low distribution coefficient results in the transfer of a higher percentage of gas per cycle of operation. The relation between percentages of gas transferred and the distribution coefficients after operating this pumping device a given number of cycles is shown below:

Gas	Distribution Coefficient*	% Gas Transferred			
		5 cycles	10 cycles	20 cycles	40 cycles
Helium	0.9935	3.2	6.3	12.2	23
	0.94	26.6	46	71	91.6
	0.977	11	21	37	60
	0.986	7	13	25	43
Neon	0.924	33	55	80	96
	0.583	93	99.6	99.99	≈ 100
Nitrogen	0.0005	≈100	≈100	≈100	≈100

5. BOTTLE PREPARATION

The development of a method of closing a bottle without heat and providing a reusable vacuum-tight seal was continued. A successful flare-type fitting was described in the previous report. A simpler device, shown in Figs. 10 and 11, was designed, tested, and found to be successful. Subsequent designs of a seal using Teflon and silicone rubber "O" rings were built and tested. These methods were discarded as unreliable. The seals discussed here are used during the laboratory preparation of the bottles and should not be confused with the cold-weld sealers used in the collection of samples during a rocket flight (See previous report).

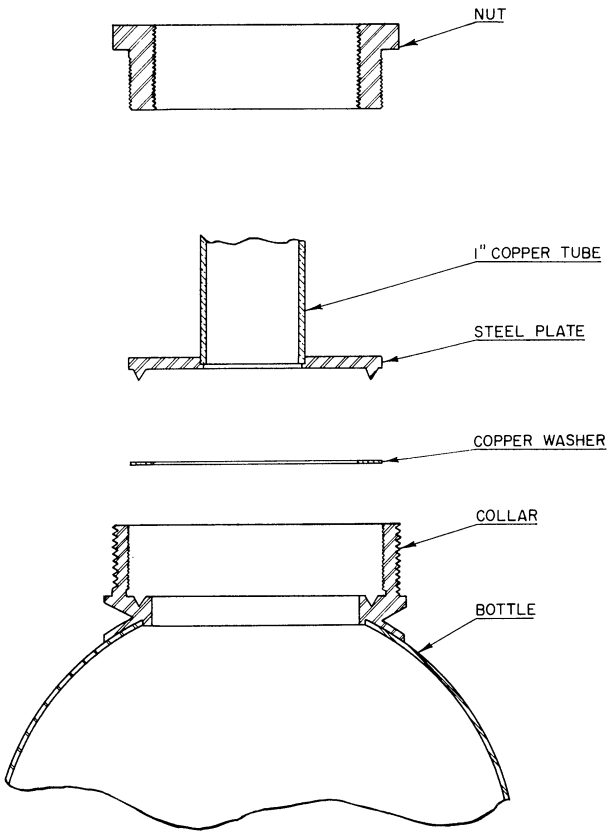

Fig. 10. Bottle Seal.

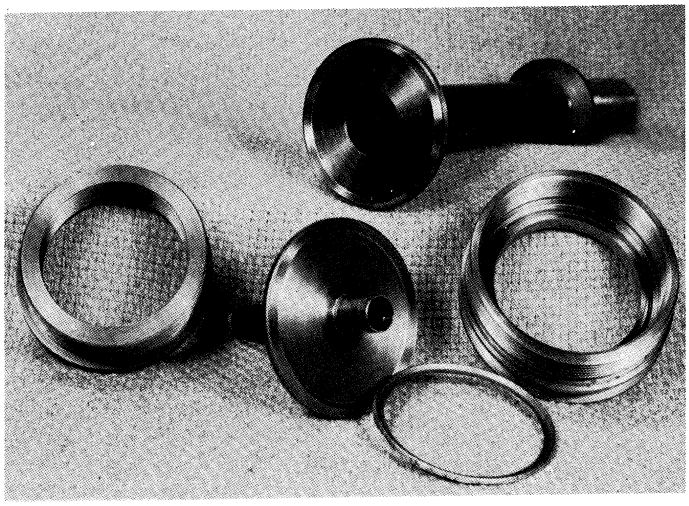

Fig. 11. Sealer Parts.

6. NEW TECHNIQUES

In November, R. M. Howe discussed several aspects of the analyzer problem with W. Nottingham, J. Houston, and W. Lange of the Physical Electronics Group at MIT. Further discussions are anticipated with people engaged in high-vacuum work in a continuing effort to keep informed on new techniques which may have application to the air analysis program. At the meeting the following topics were discussed:

6.1 Diffusivity of Helium Through Glass. A reference[2] to recent work on this subject was noted. The possibility of using pyrex in an analyzer for helium was discussed. It was felt that this might be done successfully if a) the pyrex were cooled well below room temperature, b) the pyrex were coated either by evaporation or electrolytically with metal, c) the vacuum system were immersed in a second, medium vacuum system.

6.2 Methods of Removing Active Gases, N_2 and O_2, From A Sample. It was suggested that an ion gauge operated at about 18 volts would clean up all active gases by dissociating them, whereupon they would be so active chemically as to combine with any part, particularly metal, in the system. It was thought that noble gas clean-up would be negligible at this voltage. Various new getter techniques using tantalum and titanium, were noted to be effective.

6.3 Use of Ion Gauge for Quantitative Pressure Measurements. A new, commercially available ion gauge capable of reproducing sensitivity to 10 per cent or better at 1 micron Hg pressure was discussed.

6.4 Small Mass Spectrometer and the Omegatron. A new mass spectrometer having possible application to air analysis was discussed. The possible application of the ion resonance gauge or omegatron to our problem was reviewed. The omegatron has been under development at MIT for several years. The current design, which was discussed in detail, is thought to be applicable to the analysis of helium, neon, and argon in air, particularly if the amounts of N_2 and O_2 are reduced.

6.5 Miscellaneous. Various problems and techniques of manipulating and measuring small amounts of gas were discussed.

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[2] G. J. Norton, Helium Diffusion Through Glass, General Electric Research Laboratory Report.

7. ANALYTICAL INVESTIGATIONS

A discussion of the sampling results to date was given in the previous report. It was pointed out that some serious objections to Martin's[3] interpretation of separation as being caused by a flow phenomenon exist. Nevertheless, such a phenomenon may operate during sampling, and work on the problem continued. The current approach is to calculate the pressure and temperature distributions in the field of the inlet flow. The mass rate of flow into the bottle of each component will then be calculated. The total amounts accumulated during the sampling interval will be obtained by integration.

Two aerodynamic problems concerning pressure measurements on rockets are being investigated. In the case of a pressure gauge connected to the point of measurement with a tube, the temperature gradient along the tube is a source of error. The magnitude and possibility of correcting this error is being analyzed. In a pitot-tube operating at supersonic velocity at low density, the relaxation time is a source of error. The magnitude and correction of this error is being determined.

8. REPORTS ISSUED AND LABORATORIES VISITED

No reports were issued during the quarter. The following places were visited during the quarter:

Aerojet-General Corporation

Jet Propulsion Laboratory

MIT Physical Electronics Group (See Sec. 6)

9. ACKNOWLEDGMENT

Thanks are due to the Meteorological Branch of the Signal Corps Engineering Laboratories for continued cooperation and financial support.

[3] G. R. Martin, "The Composition of the Atmosphere Above 60 Km," J. of Atmos. and Terr. Phys., Special Supplement, 1 (1954), p. 161.