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THE UNIVERSITY OF MICHIGAN  
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COLLECTION AND ANALYSIS OF UPPER-AIR SAMPLES

Quarterly Report

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by

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TABLE OF CONTENTS

	Page
ABSTRACT	iii
OBJECTIVE	iii
1. INTRODUCTION	1
2. NEW ANALYZER	1
2.1 Ion Gage Tests	1
2.2 Helium Cleanup	3
2.3 Trial Analysis	4
3. KNOWN HELIUM SOURCE	5
4. COLD-CLOSURE BOTTLES	6
5. FUTURE PLANS	7
6. ACKNOWLEDGMENT	7



ABSTRACT

Progress in the construction and operation of a selective adsorption analyzer for upper-air samples is described. Progress in the development and use of a new bottle closure device is noted.

OBJECTIVE

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."



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## 1. INTRODUCTION

This is the third 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. NEW ANALYZER

A description of the construction of the analyzer appears in a previous report. During the quarter, a new fractionating column was constructed to replace the original unit. To expedite construction and to assure charcoal traps of minimum volume and correct quantities of charcoal, the charcoal traps were constructed and tested in our laboratory. The remainder of the unit was constructed in the Physics Department glass shop. The completed unit was attached to the system, and mercury was redistilled into the control pots. The damaged 800-cc toepler pump, the storage toepler pump, and the extraction line to the bottle were not replaced. Initial operational checks are planned, using ground air from a small glass vial attached to the Y mercury cutoff. An Alpert gage was attached to the system for measuring helium pressures resulting from fractionation of the sample.

### 2.1 ION GAGE TESTS

While the work on the fractionating column proceeded, numerous tests of the Alpert ionization gage were made. The results were not as satisfactory as had been desired. Five gage tubes were used. The first was discarded after



one of its filaments burned out, and the second arced between the filament and the grid electrodes inside the tube. One filament electrode was burned off inside the tube at the seal, and the glass at the press seal partly melted. The third and fourth tubes gave some results, and the fifth was attached to the output of the analyzer as noted above.

All gages were very difficult to "start." Rather high filament temperatures were used with a-c outgassing voltages of 800 to 1250. Even under these conditions no emission current was drawn until the tube had been operated for several hours.

The final procedure for making background readings on the tubes was as follows:

- (1) Pump a vacuum of  $10^{-5}$  mm Hg or better.
- (2) Use two cold traps to isolate the gage from the pump and mercury cutoff.
- (3) Bake the ion gage and nearest trap overnight at  $400^{\circ}\text{C}$ .
- (4) Place liquid nitrogen on the cold trap farthest from the gage.
- (5) Outgas the gage with 1250 volts of ac between the filament and parallel-connected grid and collector. The current required to sustain a red heat on the grid and collector is 40 to 60 ma. This temperature is maintained for about one hour.
- (6) While the tube is hot, the mercury cutoff is closed.
- (7) The a-c voltage on the grid and collector is turned off.
- (8) Liquid nitrogen is placed on the cold trap nearest the gage.
- (9) After allowing about 10 minutes for the gage to stabilize, the electrometer is attached to the tube.
- (10) An ionizing current is turned on and the collector current read.
- (11) Pressure is further reduced by overnight ion pumping.
- (12) Final background pressures are read.

All tubes blackened very rapidly, and low background pressures were not as frequent as expected. The best vacuum was obtained after allowing the gage tube to clean up by ion pumping overnight. Vacuums of  $4.6 \times 10^{-10}$ ,  $5 \times 10^{-10}$ , and  $5 \times 10^{-9}$  mm Hg were obtained. It has been calculated that a background vacuum of  $5 \times 10^{-10}$  mm Hg is desirable if samples 0.03 cc NTP are to be analyzed. The calculated helium pressure in the gage from a ground-air sample of this size would be of the order of  $5 \times 10^{-7}$  mm Hg. The gage is less sensitive to helium than to nitrogen so the lowest possible background pressure is desired.

I.(1) The sensitivity of the ionization to various gases is shown in Table

(1) D. Alpert, J. Appl. Phys., 24, 860 (1953).



TABLE I

<u>Gas</u>	<u>Dushman and Young</u> <sup>(2)</sup>	<u>Wagener and Johnson</u> <sup>(3)</sup>
H <sub>2</sub>	0.47	0.53
He	0.16	
Ne	0.24	
N <sub>2</sub>	1.00	1.00
A	1.19	
CO		1.07
CO <sub>2</sub>		1.37
H <sub>2</sub> O		0.89
O <sub>2</sub>		0.85
Kr	1.9	
Xe	2.7	
Hg	3.4	

Alpert reports routine vacuums in the Alpert gage of  $10^{-10}$  mm Hg or better. The system used, however, differs from the gas analyzer in several respects which make outgassing and pumping easier.

Measurements of the linearity of collector current as a function of ionization current were made. Reproducible results were not obtained, and the investigation of this aspect of gage operation will be continued.

## 2.2 HELIUM CLEANUP

Gases in an ionization gage are cleaned up either by chemical or physical adsorption or by ion pumping. The data of Alpert<sup>(1)</sup> indicate that the major loss of gas is due to ion pumping in the case of the noble gases. Therefore, in the following discussion only ion pumping will be considered.

Using cleanup data given by Alpert,<sup>(1)</sup> the number of molecules removed per second at two ion-gage pressures was computed. These were compared with the number of molecules permeating an ion gage whose area is 196 sq cm as well as with the total number of molecules of helium in a ground-air sample of 0.03 cc NTP. Permeation of He is from Norton<sup>(4)</sup> for Pyrex 7740.

(2) S. Dushman and A. H. Young, Phys. Rev., 68, 278 (1945).

(3) S. Wagener and C. B. Johnson, J. Sci. Instr., 28, 278 (1951).

(4) F. J. Norton, J. Am. Ceram. Soc., 36, 90 (1953).



TABLE II

Molecules of He in 0.03 cc NTP Air	He Molecules Degassed/Sec		Molecules of He Permeating from Air/Sec
	P = 10 <sup>-7</sup> mm Hg, Ion- izing Current 10 ma	P = 10 <sup>-9</sup> mm Hg, Ion- izing Current 0.1 ma	
4.2 x 10 <sup>12</sup>	1 x 10 <sup>10</sup>	1 x 10 <sup>6</sup>	1.9 x 10 <sup>7</sup> at 25°C 1.7 x 10 <sup>8</sup> at 100°C

Assuming a 10-second reading time and approximately linear response of the gage to changing ionizing current, the following is established:

- (1) Helium permeating the gage is negligible.
- (2) Ionizing currents of 10 ma are not practical.
- (3) To keep cleanup of the sample to 1 percent or less, the ionizing current should be a maximum of 4 ma.

### 2.3 TRIAL ANALYSIS

The controls of the analyzer were all tested. A roof-air sample of about 0.5 cc NTP was taken in a glass vial. A special dosing device was constructed of soft glass for the purpose.

The oven was placed on the analyzer, and the system was baked at 340°C overnight. A small amount of liquid air was added to the single cold trap. (The two-trap system was not settled at this time.) The ionization gage was outgassed according to the above schedule. The system was now in condition to admit a sample. Mercury in the Y cutoff valve was operated to prevent loss of sample when the vial was opened. The septum of the vial was broken. A sample of about 70 cc at 1.05-cm pressure appeared. It is probable that the stopcock of the "doser" system leaked, causing the increase in the quantity of gas over that originally taken. The system was then allowed to stand overnight.

The next day an initial test sample of 0.65 cm Hg, measured in the oxygen cell at 28.5°C, was admitted to the oxygen cell from the vial. Oxygen was removed from the sample by oxidation of a copper filament, heated electrically to approximately 700°C. The pressure in the oxygen cell was again measured, and it was found that the sample had been reduced by 19.7 percent. This operation was repeated, but no further volume reduction appeared.

The ion gage was prepared for operation. This time, the gage failed to operate properly. The tube became badly blackened in the outgassing opera-





tion. The best vacuum obtainable was  $5 \times 10^{-8}$  mm Hg.

The routine fractionation was now performed at the end of the 11th fraction (before the first sample arrives at the final pipette); the pipette was opened to the gage to measure the pressure developed by the residual gas in the column and charcoal traps. The gage pressure before opening was  $6.4 \times 10^{-9}$  mm Hg. After opening the pipette, the pressure increased to  $7 \times 10^{-9}$ . If this gas were all nitrogen, this would represent a background pressure of  $6 \times 10^{-10}$ . Even with the smallest sample planned, this would be satisfactory.

The pipette was closed, and fractionation continued through the 24th fraction. Before opening the pipette, the background pressure was  $3.8 \times 10^{-9}$  mm Hg as nitrogen. The gage was then opened to the column. The pressure increased, then dropped too rapidly to make satisfactory readings. The highest pressure recorded was  $1.4 \times 10^{-7}$  mm Hg. Considering the time necessary for adjustments to make the first reading and the rate of cleanup, it is estimated that the original pressure in the tube was about  $6 \times 10^{-7}$  mm Hg.

### 3. KNOWN HELIUM SOURCE

A source of helium yielding gas at a known rate would be useful in checking the leak detector, adding known quantities of helium to ground-air samples and checking the analyzer. Such a source may be made by filling a small Pyrex 7740 vial with helium which will diffuse at a known rate<sup>(5)</sup> using the relation

$$q = \frac{KA t(P_1 - P_2)}{d},$$

where

- q = quantity of helium permeated (cc NTP)
- K = permeation constant ( $\text{cm}^3$  gas NTP/sec/sq cm)/(unit pressure gradient in cm Hg per mm wall thickness)
- t = time (sec)
- $P_1$  = pressure on high side (cm)
- $P_2$  = pressure on low side (cm)
- d = thickness of the glass (mm)

Calculations were made for a bulb of diameter 0.45 inch, area 4 sq cm, wall thickness 1.0 mm, and internal pressure of He at 45 cm Hg. In a vac-

(5) W. A. Rogers, et al., "Diffusion Coefficient, Solubility and Permeability for Helium in Pyrex," Westinghouse Scientific Paper No. 1766. Calculation based on Norton's data.



uum system, this vial would provide  $1 \times 10^{-6}$  cc NTP of He in 10 minutes at 25°C. Using this as an additive to a ground-air sample of 0.1 cc NTP, it would provide an increase in the helium content of 20 percent.

A similar unit, of larger volume and filled with helium at atmospheric pressure, was enclosed in an appendage and attached to the helium leak detector. The leak detector was found to be relatively insensitive. Tuning procedures were followed, but only slight improvements were made. An appendage of this type will be attached to the analyzer after initial test runs.

#### 4. COLD-CLOSURE BOTTLES

Parts for several cold-closure bottles were machined. Three bottles were assembled for tests, and the following tests will be made:

- (1) Bottles will be pumped and tested on the helium leak detector.
- (2) They will be baked at 190°C until they show a pressure of less than  $10^{-6}$  mm Hg as read on a Philips gage.
- (3) The bottle will be sealed off the system in the standard manner.
- (4) The bottle will be set aside and aged for at least a month. In this time the pressure in the bottle shall not read more than  $10^{-5}$  mm Hg.
- (5) The bottle will be set up in a jig and sealed as in a standard rocket firing.
- (6) The bottles will be opened and attached to the helium leak detector, and all cold-closure seals tested for leakage due to stresses involved in the pyrotechnic sealing.

It is planned to test-fire two bottles with 2-inch intake tubes, and one with a 1-inch intake tube in the above manner.

One of the 2-inch bottles was completed, and the pyrotechnic test sealing made. No leak appeared in the bottle until several days had passed. It is believed that the small 1/4-inch pinchoff tube was inadvertently damaged. The leak detector is not in operating condition, so the final check must be postponed.

A second bottle with a 1-inch intake tube was prepared, baked, and sealed off. After a month of standing, the bottle showed no readable pressure on its Philips gage. A pressure of  $5 \times 10^{-6}$  mm Hg is detectable. This bottle may be reserved as a flight bottle.

A third bottle was prepared but failed to pump properly. The difficulty is believed to be in the screw of the cold closure. It is being rebuilt.

An attempt was made to simplify the Philips gage built into the bot-



tle. A trial gage was constructed of sheet metal with indentations for an external magnet. If it had been successful, it would have eliminated the complicated iron pole pieces now built into the bottle Philips gage, as well as the extra silver-soldered connections necessary to attach the pole pieces. At this time two models have been built, and neither has the required sensitivity which seems to be reduced by about one third.

#### 5. FUTURE PLANS

Construction and operation of the analyzer will continue.

#### 6. ACKNOWLEDGMENT

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