ENGINEERING RESEARCH INSTITUTE
UNIVERSITY OF MICHIGAN

ATMOSPHERIC PHENOMENA AT HIGH ALTITUDES

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Submitted for the project by:

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Department of Aeronautical Engineering

1. INTRODUCTION

This is the ninth in a series of quarterly reports on Contract No. DA-36-039 SC-15443 describing high-altitude meteorological experiments being carried out by the University of Michigan for the Meteorological Branch of the Signal Corps. This program is a continuation of one which was carried out between July 1946 and August 1950 on Contract No. W-36-039 sc-32307 and from August 1950 to December 1951 on Contract No. DA-36-039 sc-125. For background material the reader is referred to the final reports on these contracts and the previous ones of this series.

2. SUMMARY

The work during the quarter was devoted primarily to the planning and designing of a new, high sensitivity gas analyzer. Work on adsorption in sample bottles and the design of sample bottles for flight was continued. An investigation of a quartz crystal vacuum-pressure gage was started.

3. NEW ANALYZER

As noted in the previous report, a new charcoal adsorption analyzer is to be constructed at Michigan. The new equipment will be used to continue the investigation of the separation which was detected in control bottles and to analyze the contents of the subsonic peak-sampling Aerobee SC-34. This rocket will be flown upon completion of the control bottle work. Its purpose is to obtain samples in which the effects of possible supersonic flow phenomena on composition have been eliminated.

3.1 Literature Survey

A systematic review of the extensive literature in the fields of chemical and physical gas analysis was carried out. It was determined that in the fields of mass spectrometry, infra-red spectrometry and chromatography (except charcoal adsorption) present techniques either do not apply or lack

the sensitivity or precision for analyzing upper-air samples. Thus it was confirmed that the new analyzer should be of the charcoal adsorption type. Several laboratory techniques and devices of possible use in the new analyzer were noted. One such device, the omegatron,[1] may have application in resolving helium and neon at low pressures.

3.2 Design

The basic requirement of the new analyzer is to measure the amounts of N_2, O_2, He, Ne, and A in air samples of the order of 0.03 cc NTP with a precision such that: (1) the mean deviations in ratios of the latter four gases to N_2 will be about ± 0.5 per cent and (2) these ratios in ground air will agree with accepted values to within the same amount. A second desideratum is that the time of analysis be substantially reduced. At present, a single complete analysis requires about 12 hours.

Three major steps to permit handling air samples of 0.03 cc NTP (the first Michigan analyzer requires 1.0 cc NTP) are contemplated. These are: a smaller O_2 cell (in which the original quantity of gas is measured), a more sensitive and accurate final gage, and a smaller system to reduce residual pressure errors. Three major steps in achieving faster analyses are planned. These are: a small system which can be completely enclosed by an oven for outgassing, a faster method of transferring samples to the analyzer, and a faster final pressure gage. Several secondary items of design to be considered for the new analyzer are: charcoal adsorption characteristics, type of glass, type of valves and fractionating column cells, automatic mercury controls, and a He and Ne recycling system.

Consideration of all of the above items is under way. Construction and test of components radically different from those previously used will be carried out. The current status of each item is as follows:

3.21 O_2 Cell

The conventional copper spiral O_2 cell will be used. A single spiral mounted in a cell of 5.0 cc volume in which the pressure can be read to ± 0.01 mm Hg is proposed.

3.22 Final Gage

The final pressure gage which measures the amount of He or Ne coming from the fractionating column is extremely important. In the Michigan and Durham analyzers Pirani gages operating at liquid N_2 temperatures have been used. The chief objections to the Pirani are the low temperature operation and the necessity for "before and after" calibrations on known amounts of gas to calibrate out hysteresis effects. An ionization gage does not have these drawbacks but has the disadvantage of "cleaning up" small samples. Cleanup of chemically active gases by the hot filament is so large as to preclude the

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[1] D. Alpert and R. S. Buritz, "Ultra High Vacuum (II) Limiting Factors on the Attainment of Very Low Pressures," J. App. Phys., 25 (Feb. 1954), p. 202.

use of an ionization gage on the input side of the analyzer. Cleanup of the noble gases is small so that the gage may be used on the output side. Typical values for He are shown below. The volume is 250 cc, and the amount of air is 0.03 cc NTP.

Ion Current (μ amp)	Grid Current (ma)	Degassing Speed (cc/sec)	Time for Loss of He = 20% of Total (secs)
.047	10	20	0.25
.000047	0.01	0.02	250

An improved ion gage[2] whose low pressure range has been extended may reduce cleanup because of the possibility of operating at lower grid currents in our pressure range. A further possibility is to record ion current as a function of time and extrapolate to zero time for the true value. Three Westinghouse type WL 5966 ionization gages (Fig. 1) were purchased for testing. A power supply to operate the gages was built. The power supply is shown in Fig. 2 and the circuit diagram in Fig. 3.
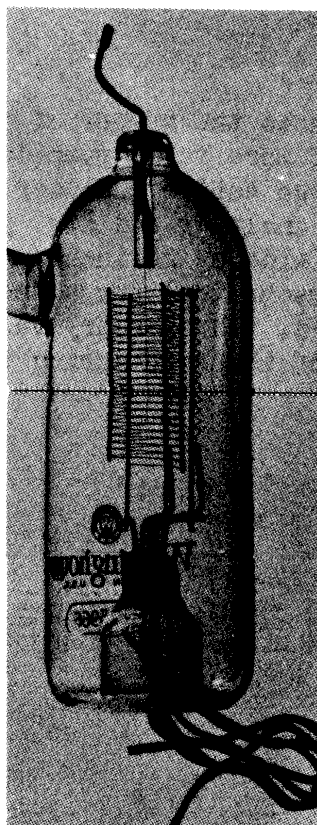

Fig. 1. Westinghouse Ionization Gage.

[2] D. Alpert, "New Developments in the Production and Measurement of Ultra High Vacuum," J. App. Phys., 24 (July 1953), p. 860.
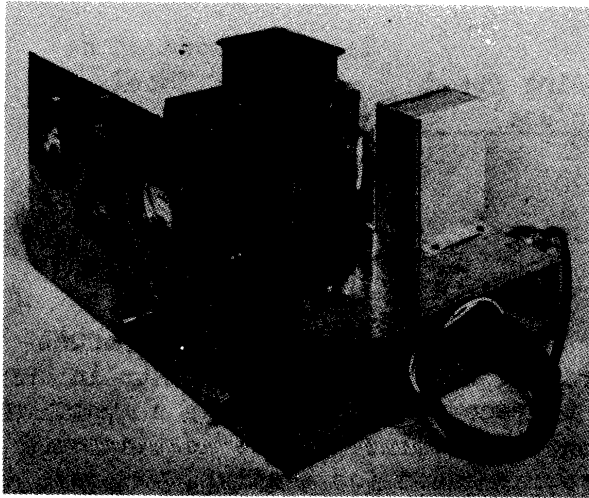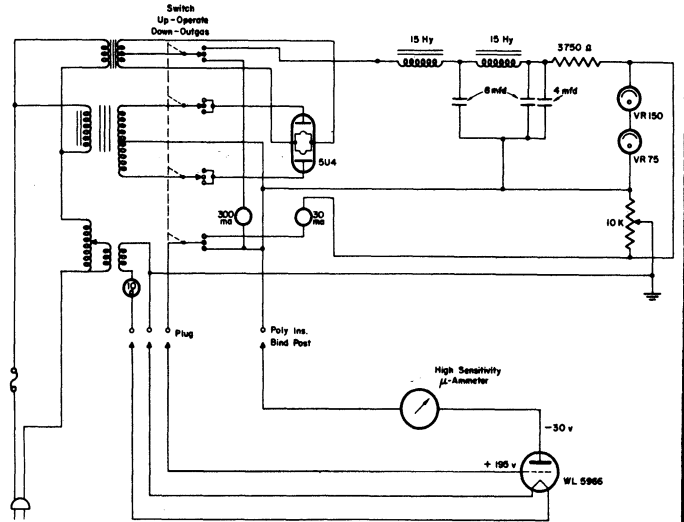
Fig. 2.
Power Supply for Ionization Gage.

Fig. 3
Circuit Diagram of Power Supply.

3.23 Small System

A small analyzer increases the number of analyses that can be performed in a given time by permitting more rapid pump-down. This may be further speeded by enclosing nearly the entire analyzer in an oven during pumping. Fig. 4 is a schematic of the analyzer and Fig. 5 a plan view. The final size is estimated to be 44 inches wide by 24 inches deep by 78 inches high. About half the height consists of mechanical pumps, mercury pots, and other accessories which will not be baked. The mercury valves will be baked, but the mercury will be lowered out of the oven during this period. The oven will be operated at 350° C.
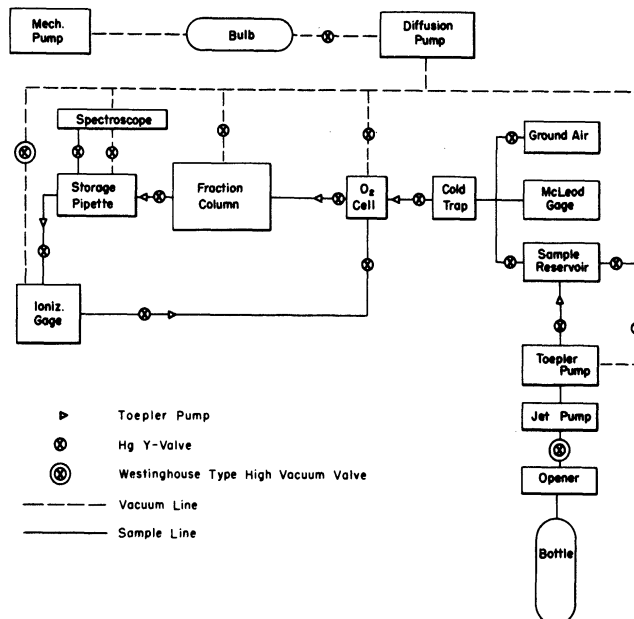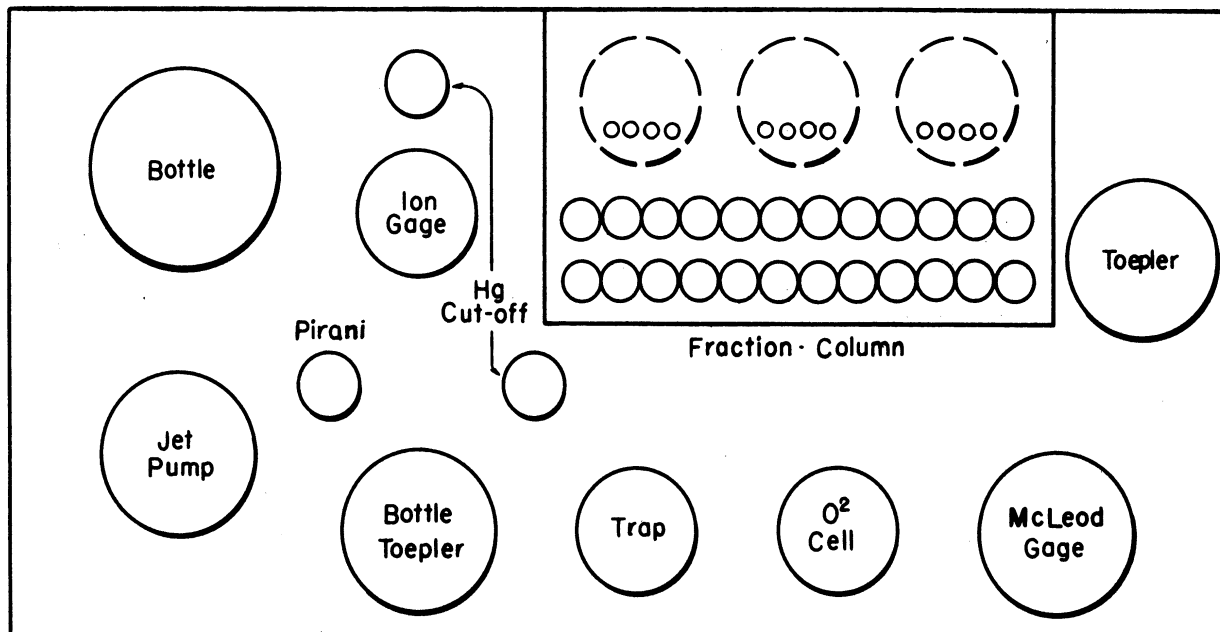

Fig. 4. Schematic of Analyzer.

Fig. 5. Plan View of Analyzer.

3.24 Transfer Pump

The current method (Durham) of transferring samples from bottles to the analyzer is a large toepler pump. An 800-cc toepler takes two hours to transfer 99.9 per cent of a sample to the storage flask. In the case of small samples it is often difficult to transfer the tiny slugs of air through the toepler valve. A mercury diffusion pump was considered for this job previously but was not used because of the possibility of losing the oxygen from the sample by reaction with the mercury. This possibility is no longer of significance since most of the O_2 is still lost in the sample bottles in spite of the use of cold-weld sealers. A combination diffusion-toepler pump with inverted jets was designed and built but did not operate satisfactorily. When a decision to use soft glass instead of Pyrex was made, the combination pump was abandoned because it was required to withstand large, rapid temperature changes. Construction of a soft glass diffusion pump which will work into a 150-cc toepler was then started.

3.25 Fractionating Column

Calculations of the number of stages, the size of cell, and amount of charcoal to be used in the fractionating column were made. The results are summarized below:

Number of Stages	Percentage of Neon in Helium Fraction*
12.4	0.5
10.6	1.0
6.5	5.0

* 0.5% = 99.5% He, 0.5% Ne.

Sample Size (cc)	Charcoal (gms)	Column Volume (cc)	Column Area (cm^2)
2.0	2.5	88	100
0.2	.25	8.8	25

It has been decided to use 12 stages consisting of 8.8-cc cells with the traps containing 0.25 grams of charcoal, thus permitting separation of He from Ne to 0.5 per cent in samples up to 0.2 cc NTP.

3.26 Charcoal

The adsorption characteristics of charcoal were reviewed for two reasons. One was to permit calculation of the number of fractionations required. The second was to permit investigation of the possibility of making a single fractionation of helium and then, by means of the omegatron,[1] measuring the amounts of helium and neon in the first fraction. From the known adsorption characteristics of the charcoal, the amounts of the two gases in the entire sample would be calculated. This method will not be pursued further now, however, because the necessity for calibrating the charcoal is a distinct disadvantage. In the fractionating column the separation is the same within limits for two charcoals if the adsorption characteristics are not too different. Typical characteristics are seen in Fig. 6.
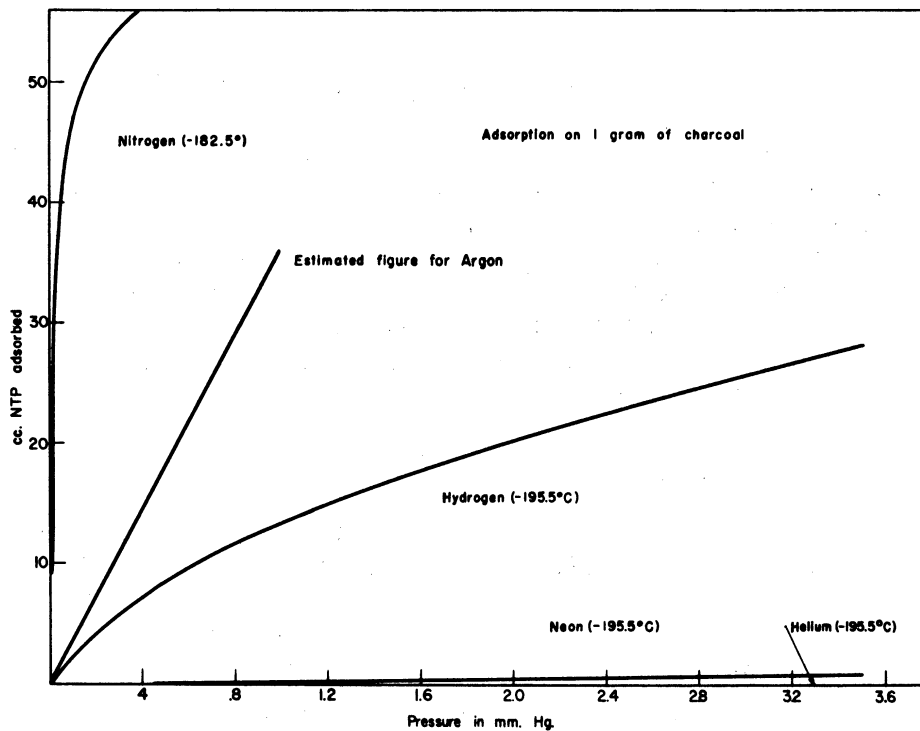

Fig. 6. Typical Adsorption Characteristics of Charcoal.

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[1] Alpert and Buritz, op. cit., p. 202.

3.27 Glass

A review of the characteristics of various glasses was made. In the original Michigan analyzer Pyrex 7740 was used in spite of its known permeance to helium because the samples were large (1 to 10 cc NTP) and the design accuracy $\pm$ 5 per cent. The calculated error due to the effusion of helium in the new analyzer would be marginal with Pyrex 7740, and it was decided to try Corning 1720 combustion tubing. Considerable difficulty was experienced in working this glass, and the decision was made to use soft lime glass.

3.28 Miscellaneous

U-tube fractionating cells will be used instead of the concentric type because of the ease of construction and the fact that the gas must pass over most of the charcoal surface.

Automatic control of the mercury columns will not be provided at first. The safe range of movement will be made as large as possible to make manual, and subsequently automatic, manipulation easier.

With two exceptions, mercury Y-valves will be used throughout, the mercury being lowered below the level of the oven during baking. Metal mechanical high-vacuum valves of Westinghouse design will be used between the bottle and the extraction pump and between the ionization gage and the vacuum line.

Provision will be made to reintroduce the separated helium (or neon) to the fractionating column for recheck. See Fig. 4.

4. CONTROL AND SAMPLE BOTTLES

4.1 Adsorption in Control Bottles

Two control bottles containing Durham ground air have been in preparation at Michigan for analysis at Durham. In view of the termination of the air analysis program at Durham, these bottles will be held for the completion of the Michigan analyzer. Each bottle was tested for nitrogen adsorption before insertion of the ground-air vial and evacuation. The measurement of negligible adsorption of N_2 in one bottle (now designated as B-25-P) was described in Section 3.3 of the previous report. The second bottle, B-20-P, showed appreciable separation in ground air when first analyzed at Durham. It was returned to Michigan for re-evacuation and insertion of ground air but leaked upon opening the glass vial. It was decided then to run a nitrogen adsorption test on B-20-P before refilling the bottle with ground air for analysis. It is interesting to note that a measurable adsorption of pure N_2 was detected in this bottle. See table on next page. B-20-P will now be re-evacuated and filled with Durham ground air.

First Run

Slug No.	McLeod Reading mm Hg	Δp Observed mm Hg	Δp Calculated mm Hg	p Calculated mm Hg	% Difference
1	5.24	5.24	5.87	5.87	10.1
2	10.8	5.56	5.83	11.70	7.7
3	16.35	5.55	5.79	17.49	6.5
4	21.70	5.35	5.75	23.24	6.6

Second Run

Slug No.	McLeod Reading mm Hg	Δp Observed mm Hg	Δp Calculated mm Hg	p Calculated mm Hg	% Difference
1	8.25	8.25	8.82	8.82	6.5
2	16.70	8.45	8.76	17.58	5.0

4.2 Flare-Fitting Vacuum Joint

A flare-fitting vacuum joint to be used in sample bottles was described in the previous report. Such a fitting was constructed (see Fig. 7) but has not been tested.
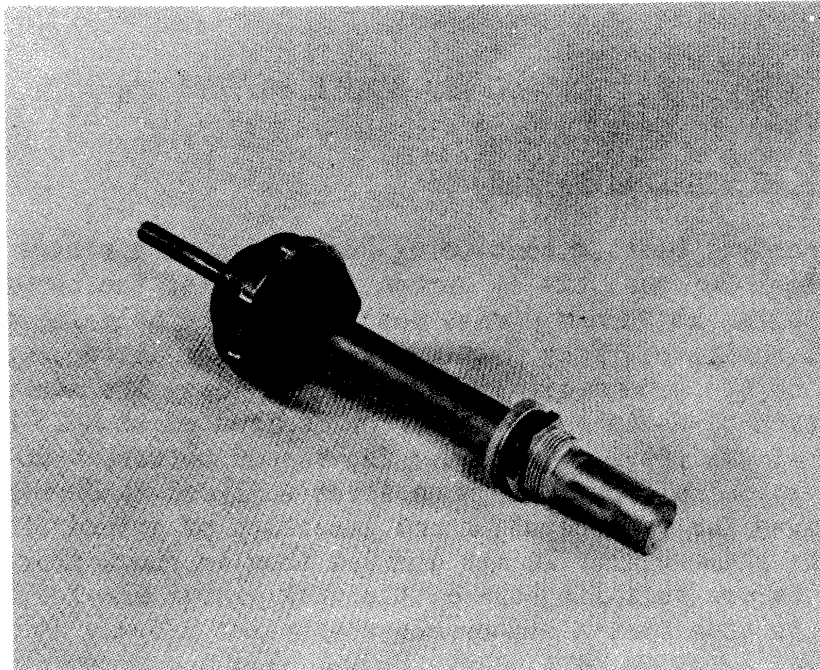

Fig. 7. Flare-Fitting Vacuum Joint.

5. CRYSTAL VACUUM-PRESSURE GAGE

In the course of considering various possibilities for the final gage of the new analyzer an investigation was made of a vibrating quartz crystal as a low-pressure gage. It was thought that if the crystal method were feasible, it could be incorporated in a gage of very small volume ideally suited for measuring the output of the fractionating column. The method makes use of the fact that the Q of a crystal is a function of pressure. Calibrations of the crystal gage were made in the range 760 mm to 5 mm Hg. Also, it was noted qualitatively that the gage was sensitive both above and below this interval. The limiting factor in the low-pressure direction was the inherent drift of the circuit. Improvements in this respect appear possible but have not been pursued since the Westinghouse ionization gage mentioned above was selected as being suitable for the final analyzer gage.

A 7.38-mc crystal was used in a Pierce oscillator circuit. The crystal housing was made vacuum-tight and fitted with an evacuation tube. Calibrations were made against Bourdon tube type vacuum gages. The pressure was read in terms of the d-c grid voltage of the oscillator. A bucking voltage permitted setting the voltmeter reading at zero for any desired pressure. Fig. 8 is a series of calibrations with plate voltage as a parameter. Fig. 9 is the oscillator (removed from its case). The crystal is in the rectangular case to the right of the vacuum tube with the evacuation tube extending through the top. Fig. 10 is a circuit diagram. A pentode oscillator with increased output and a balancing network in the plate circuit was used for some tests. The inherent stability was about the same as with the triode.
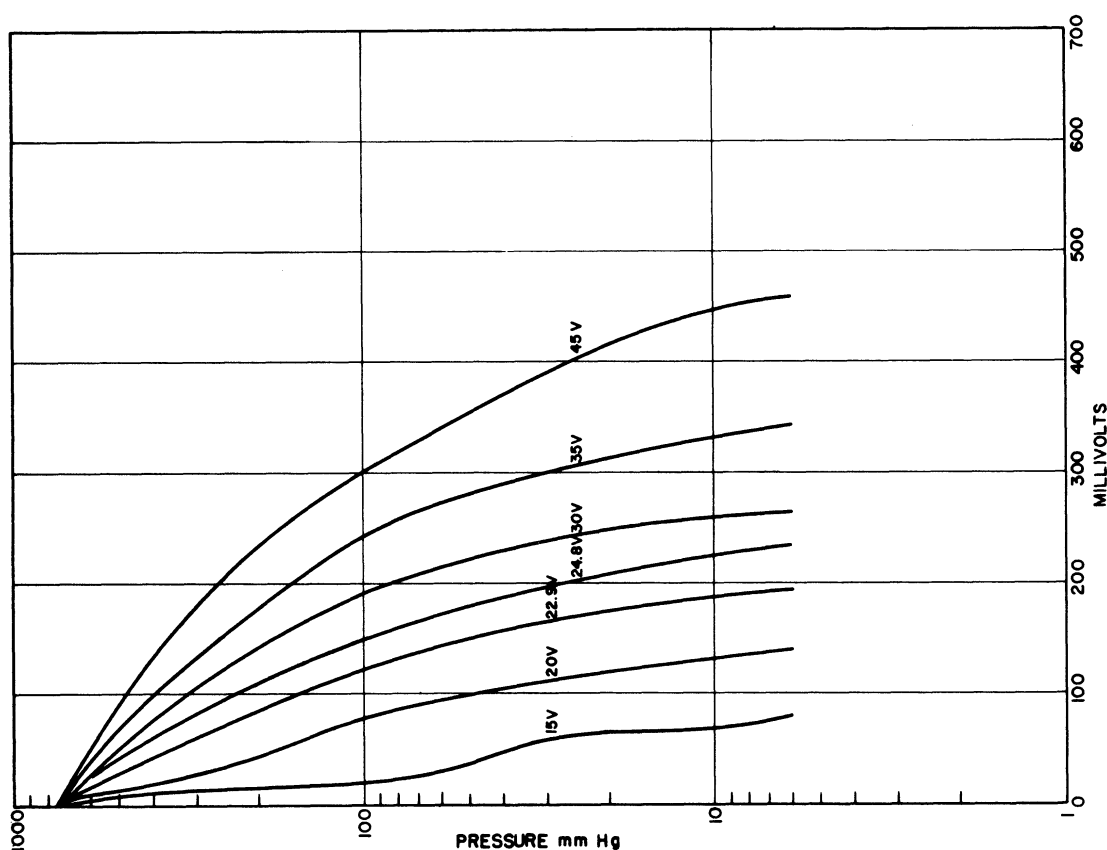

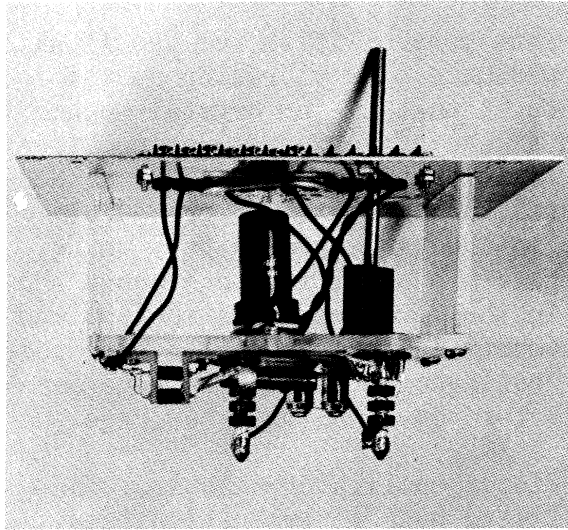Fig. 8. Calibration of Crystal Vacuum Gage.
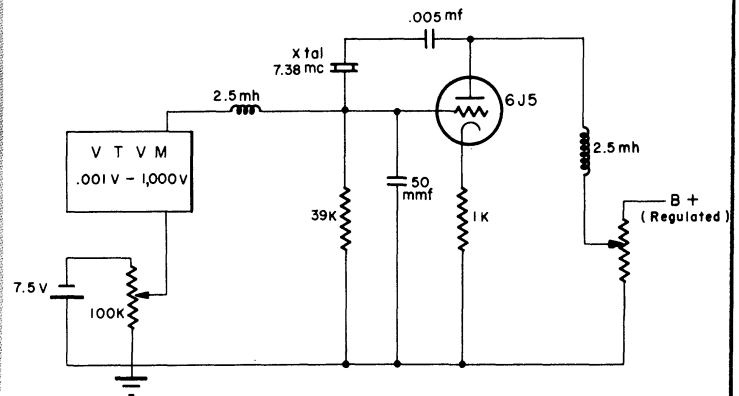
Fig. 9.
Crystal Vacuum Gage Oscillator.

Fig. 10.
Crystal Vacuum Gage Circuit.

6. PREVIOUS UPPER-AIR SAMPLES

Two communications discussing atmospheric composition and based in part on Michigan upper-air samples were received.

6.1 Oxygen and Nitrogen Isotopes

Dole et al[3] analyzed two samples from Aerobees SC-5 and SC-3 (mean altitudes: 46 km and 51.6 km, respectively) for oxygen content and oxygen and nitrogen isotope abundance. The nitrogen isotopic composition was found to be normal. The O^{18} percentage was found to be very slightly larger in the upper-air samples, but the increase is not thought to be significant in view of the magnitude of the corrections made for chemiosorption of O_2 in the bottles.

6.2 Partial Separation of Gases in the Upper Atmosphere

Wiborg[4] developed a theory supporting the possibility of partial separation of gases in the atmosphere between 55 and 70 kilometers. His

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[3] Dole, Rudd, Lane, Zarkelies, and Brown, The Height and Geographical Distribution of the Oxygen Isotopes, Final Report, Air Force Contracts AF 19(122)-157 and AF 19(604)-587, 1954.

[4] B. S. Wiborg, Ph.D. dissertation, University of Durham, To be published.

analysis which evaluates the relative importance of ionization flow, basic flow, gravitational flow, molecular flow, and flow due to turbulence yields values for the concentrations of helium, neon, and argon which are in good agreement with our rocket samples showing separation in this region.

7. FUTURE PROGRAM

Design and construction of the new analyzer will be continued.

8. ACKNOWLEDGMENT

Thanks are due the Meteorological Branch of the Signal Corps for continued cooperation and support.