

GCA Technical Report No. 65-28-N

MEASUREMENT OF UPPER ATMOSPHERE HELIUM PROFILE

A. E. Barrington

FINAL REPORT

Contract No. NAS5-9211

22 November 1965

GCA CORPORATION  
GCA TECHNOLOGY DIVISION  
Bedford, Massachusetts

Prepared for  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
Headquarters  
Washington 25, D. C.

## TABLE OF CONTENTS

SUMMARY	1
INTRODUCTION	1
COMPATIBILITY OF TANDEM MASS SPECTROMETER WITH JAVELIN SPECIFICATIONS	5
PROBLEMS ASSOCIATED WITH A MASS SPECTROMETER JAVELIN PAYLOAD	7
FEASIBILITY OF ALTERNATE HELIUM AND HYDROGEN MEASUREMENTS	9
BAKEABLE HELIUM-HYDROGEN TANDEM MASS SPECTRO- METER	11
RECOMMENDATIONS FOR FURTHER WORK	17
APPENDIX A - PARTICLE NUMBER DENSITIES TO 10,000 KM ALTITUDES ABOVE THE EARTH'S SURFACE	19
APPENDIX B - SUPPRESSION OF EXCITED SPECIES FROM SPUTTER-ION PUMP BY A ZEOLITE TRAP	25
APPENDIX C - HELIUM MEMORY EFFECTS OBSERVED WITH TANDEM MASS SPECTROMETER	27

## LIST OF ILLUSTRATIONS

<u>Figure</u>	<u>Title</u>	<u>Page</u>
1	Aerobee Tandem Helium Mass Spectrometer	2
2	Javelin Tandem Mass Spectrometer Design	12
3	Javelin Tandem Mass Spectrometer	13
4	Mass Spectrometer Electronics and Hydrogen-Helium Command Unit	14
5	Number Densities at 1000 <sup>o</sup> K.	21
6	Number Densities at 2000 <sup>o</sup> K.	22
7	Neutral Hydrogen Number Densities at 1000 and 2000 <sup>o</sup> K.	23

## MEASUREMENT OF UPPER ATMOSPHERE HELIUM PROFILE

A. E. Barrington

### SUMMARY

2 0078

The present report describes work performed during a period of four months to upgrade a tandem mass spectrometer, designed originally for an Aerobee helium experiment, to measure the helium and hydrogen profiles between 125 and 1500 kilometers with a Javelin rocket. Several electrical and mechanical components required only minor modifications; however, to facilitate a meaningful hydrogen measurement, an ultra-high vacuum bakeable stainless-steel analyzer was designed, fabricated and tested. Because of the limited time available, the tests were confined to an unbaked vacuum system.

In order to measure the helium and hydrogen number densities, predicted by theory as outlined in Appendix A, a minimum detectable signal capability of  $10^{-12}A$  at the multiplier output is desirable. Although the original multiplier had adequate gain and a dark current well below this value, it was mechanically incompatible with Javelin specifications. Two commercial ruggedized multipliers were investigated - one in a sealed-off envelope, the other incorporated in the new stainless-steel mass spectrometer. This new mass spectrometer has adequate resolution and sensitivity for the proposed measurements. However, a firm choice of the multiplier will have to await tests under ultra-high vacuum conditions following adequate bake-out and oil-free pumping. Recommendations regarding the attainment of this objective form the concluding section of the report.

*Quesada*

### INTRODUCTION

Two helium tandem mass spectrometers, designed by G. Sauermann and R. F. K. Herzog for an Aerobee flight over the altitude range from 100 to 200 kilometers, were constructed by the GCA Corporation in 1961 under Contract No. NAS5-270 (Figure 1). Details of these instruments were documented in GCA Report 61-8-N. A meaningful helium measurement

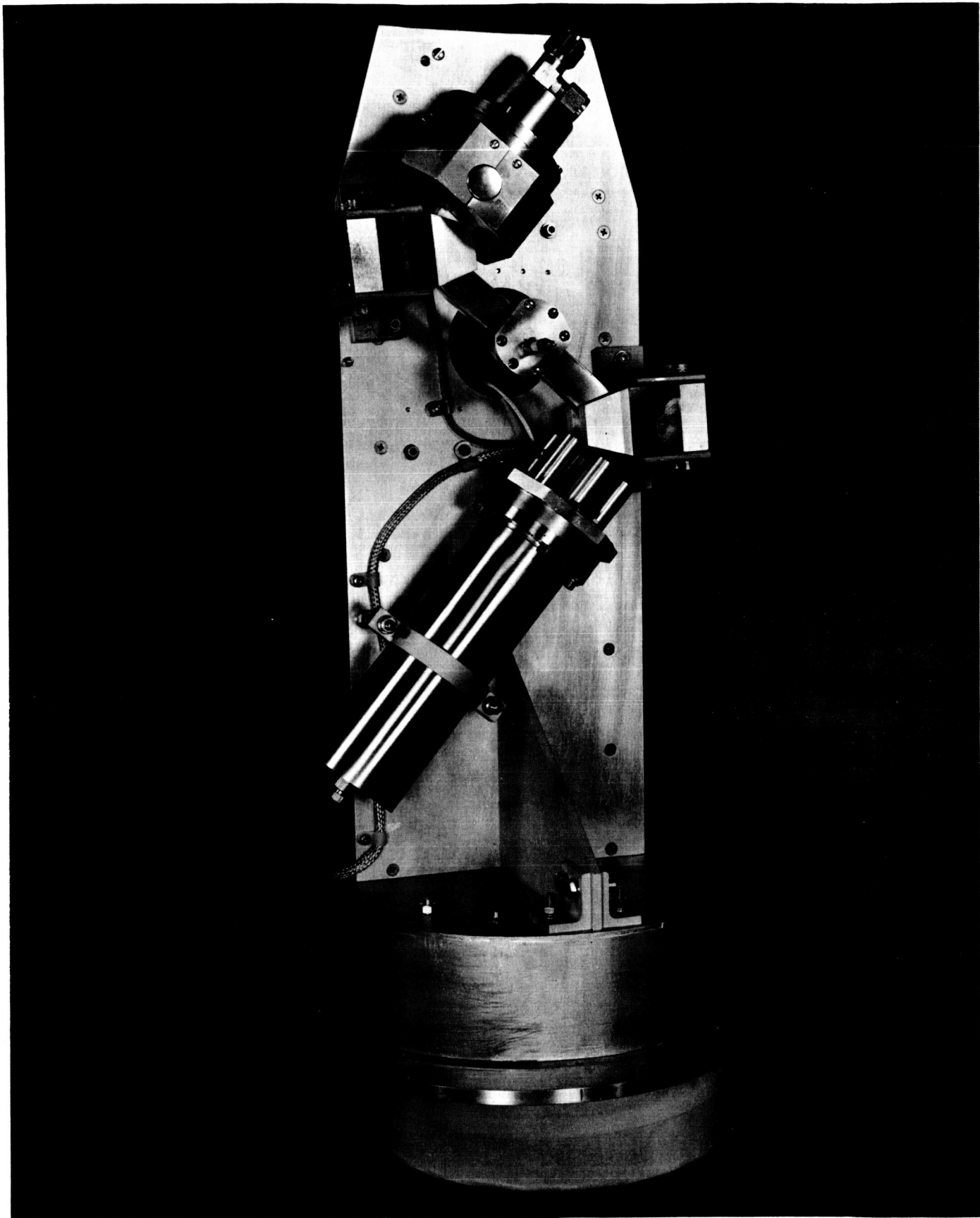


Figure 1. Aerobee tandem helium mass spectrometer.

at these altitudes requires a mass spectrometer with a sensitivity of better than 5 parts per million of helium in air at an ambient air pressure as high as  $10^{-4}$  Torr down to a helium partial pressure of less than  $10^{-11}$  Torr.

According to the performance tests described in the above GCA report, the helium sensitivity of the tandem mass spectrometers was 80A/Torr; since the minimum detectable multiplier output current was  $10^{-10}$ A, the minimum detectable helium partial pressure was approximately  $10^{-12}$  Torr, entirely adequate for the proposed application.

The present investigation was undertaken in order to determine the feasibility of utilizing the helium tandem mass spectrometers for a measurement of both the helium and hydrogen profiles over a considerably wider range of altitudes with a Javelin rocket.

The scope of work to be performed was as follows:

(1) Examine and test all subassemblies of the two tandem helium mass spectrometers. Provided these are in working order, examine and test one set of subassemblies to determine their performance capability for a Javelin payload;

(2) Examine theoretically and experimentally the problem areas associated with using these helium mass spectrometers to measure the atmospheric helium concentration from a Javelin rocket over the altitude range from 125 kilometers to approximately 1500 kilometers;

(3) Determine the feasibility of using these spectrometers to alternately measure both the atmospheric helium and hydrogen concentration;

(4) Modify one spectrometer so that the instrument is repeatedly bakeable (approximately 10 times) to  $400^{\circ}\text{C}$  for a duration of 8 hours each time. This to include among other things:

- (i) Fabrication of a stainless steel envelope.
- (ii) Mounting to the spectrometer a Johnston Laboratory bakeable electron multiplier.

(5) The modified instrument to have the capability of measuring  $m/e$  of 2, requiring among other things modification of the system electronics.

(6) Deliverable end items:

- (i) The tested but unmodified mass spectrometer system.
- (ii) The modified and vacuum-tested mass spectrometer system.

COMPATIBILITY OF TANDEM MASS SPECTROMETERS  
WITH JAVELIN SPECIFICATIONS

Electronic Subassemblies

Before any vibration testing of the existing instruments was performed, the following subassemblies were examined:

(1) High and Low Voltage Power Supplies:

One power supply met all original specifications. The other required replacement of a reference precision Zener diode.

(2) Electrometer Amplifiers:

These were tested regarding drift and noise level and met the original specifications.

The thermally-controlled voltage regulator, considered a critical component under high acceleration and vibration was then tested up to 56 g. Its performance was not impaired by the test. Both High and Low Voltage Power Supplies were again tested with the thermal regulator and met the original specifications.

As regards the electrometer amplifiers, their compatibility with Javelin specifications was considered questionable. Furthermore, because of the higher altitude of the Javelin flight, their range was marginal. Recommendations regarding the use of compatible electrometers will be found at the conclusion of this report.

Mechanical Subassemblies

(1) Ion Source

The flexible leads joining the feedthroughs to the electrodes were replaced by rigid connections and insulating spacers were inserted in the feedthrough openings to minimize vibration.



The ion source was tested prior to and following acceleration and vibration tests and performed satisfactorily.

(2) The existing twenty-stage electron multiplier did not survive even partial vibration and acceleration tests without serious mechanical misalignment. This result was anticipated and only confirmed by the test.

The above acceleration and vibration tests were performed and certified by Associated Testing Laboratories, Inc., Burlington, Massachusetts.

PROBLEM AREAS ASSOCIATED WITH A MASS SPECTROMETER  
JAVELIN PAYLOAD

As mentioned previously, the electrometer amplifiers and the electron multiplier presented major difficulties which required solution. Since it was understood that compatible electrometer amplifiers were in existence at the Goddard Space Flight Center, no further work in this area was undertaken. Instead, efforts were concentrated on the electron multiplier problem.

According to test data of the helium mass spectrometers prior to delivery to Goddard Space Flight Center in 1961, the helium sensitivity with a multiplier gain of  $2 \times 10^6$  was 80A/Torr. As shown in the Appendix, the most pessimistic figure for partial particle density of hydrogen and helium at 1000 km is not likely to be less than  $10^4 \text{cm}^{-3}$ . This corresponds to a partial pressure of  $3 \times 10^{-13}$  Torr and an output current from the multiplier of not less than  $2 \times 10^{-11}$ A. For a meaningful current measurement, the multiplier dark current should be at least a factor of 10 below this value.

Two ruggedized multiplier types available commercially warranted serious consideration for the present application, namely Model FW-305 windowless multiplier manufactured by ITT Industrial Laboratories, Fort Wayne, Indiana, and Model MM-1 focused mesh multiplier produced by Johnston Laboratories, Inc., Baltimore, Maryland.

A laboratory model of the ITT multiplier was obtained on consignment. This was shipped to GCA in an evacuated sealed-off enclosure. It was not, at any time, exposed to air at atmospheric pressures.

The characteristics of this unit following vibration tests according to the manufacturer were as follows:

MEASURED CHARACTERISTICS  
F4020 #036502

VOLTAGE V	GAIN	DARK CURRENT A
2000	$7 \times 10^4$	
2200	$1.5 \times 10^5$	$1.5 \times 10^{-12}$
2400	$3.1 \times 10^5$	
2600	$6.2 \times 10^5$	
2800	$1.2 \times 10^6$	$2.5 \times 10^{-10}$
3000	$2.2 \times 10^6$	

When the ITT multiplier was tested by GCA, the dark current was approximately a factor of 10 higher than was indicated by ITT. Intermittent internal discharges cleared up only after several hours of aging. Eventually, after 8 hours, the dark current decreased to  $2 \times 10^{-12}$  A at 3000 V. It was confirmed by ITT that this was the optimum value for this multiplier type.

According to discussions with the manufacturer, the dark current of the MM-1 Johnston multiplier is less than  $10^{-15}$  A, with a gain of  $10^6$  at 4 kV. Since it was not possible to obtain a unit for testing on consignment, the ITT unit was returned and a Johnston MM-1 multiplier, custom-mounted for use in the bakeable version of the tandem mass spectrometer (described in a subsequent section), was purchased. This unit was supplied in a housing evacuated and backfilled with an argon atmosphere.

It should be pointed out that, to-date, this unit has not been baked. The test data given below are therefore not directly comparable with those of the ITT multiplier.

VOLTAGE V	GAIN (ARBITRARY UNITS)	DARK CURRENT A
3000	1	$2 \times 10^{-13}$
3500	10	$1 \times 10^{-12}$
4000	77	$1 \times 10^{-11}$
4200	133	$3 \times 10^{-11}$

Testing was performed at an ambient pressure of  $10^{-6}$  Torr with the multiplier pumped by a zeolite-trapped oil diffusion pump (DC-704 oil). Past experience has shown that multiplier performance is greatly improved in an oil-free system. For example, although the gain of the multipliers supplied with the existing Aerobee tandem mass spectrometers was approximately 2 orders of magnitude less than the figure of  $2 \times 10^6$  quoted for full activation (this is, of course, due to the four-year exposure to atmospheric air during storage), the dark current of a similar multiplier in current use in an unbaked ion-pumped system is less than  $10^{-14}$  A with a gain of  $10^5$ .

In consequence, no firm conclusions regarding the merits of the MM-1 focused-mesh multiplier can be drawn at this time. Further and more detailed tests will have to be made.

FEASIBILITY OF ALTERNATE HELIUM AND  
HYDROGEN MEASUREMENTS

Although, as mentioned previously, the sensitivity of the existing Aerobee mass spectrometers was reduced by approximately 100, it was readily established that  $m/e = 2$  can be indicated by changing the following ion source potentials:

Electrode	Potential V	
	For $m/e = 4$	For $m/e = 2$
Ion Acceleration	285	525
Repeller	290	0
Ion Focus	118	395

All other potentials are the same as are required to indicate  $m/e = 4$ . Accordingly, an attachment to the present mass-spectrometer power supply was designed which, on command, can switch alternatively into the helium mode or into the hydrogen mode.

To accomplish this, the following modifications of the existing unit were necessary:

- (1) Provide an additional 525 volts with at least a plus or minus 5 volt adjustment for the ion source acceleration potential in the hydrogen mode.
- (2) Provide an additional 395 volts with at least a plus or minus 5 volt adjustment for the ion source ion-focus potential in the hydrogen mode.
- (3) Provide an additional emission current control for the hydrogen mode of operation.
- (4) Provide and incorporate a latching relay for command switching of the electrode potentials and bias levels in order to switch between helium and hydrogen modes.
- (5) Provide command input terminations and monitor terminations at the input and output connectors.

(6) Modify high voltage supply to be capable of driving a 20 megohm load at 4 kV.

(7) Modify input and output connectors to make above changes compatible with existing system.

(8) Modify output cable as is required.

The 525 volt and 395 volt ion-acceleration and ion-focus potentials were derived from ten-turn pots across a thermally-stabilized, Zenored 600-volt source, followed by an L-type filter.

The hydrogen mode emission control employs a variable resistor with a self-bias technique. The emission current can be adjusted to the optimum value.

The latching relay is a TL 17DA, selected because of its higher voltage characteristics and its proven shock and vibration capabilities in flight applications. The TL 17DA is a 4-pole latching type relay. The electrode potentials and bias levels are switched simultaneously, as a command function. A short duration 28-volt pulse on Pin E of the input connector will place the latching relay in the helium mode. A similar pulse to Pin G will place the latching relay in the hydrogen mode.

The function switching of the latching relay can be monitored at the output connector. A closure of the relay in the helium mode is indicated by continuity between Pin R and Pin A of the output connector. A closure of the relay in the hydrogen mode is indicated by continuity between Pin R and Pin B of the output connector.

Pin R of the output connector is connected to an arm of the latching relay. Pin A is connected to a contact on the latching relay which is in contact with Arm Pin R when the system is in the helium mode. Pin B is connected to a contact which is in contact with Arm Pin R when the system is in the hydrogen mode.

## BAKEABLE HELIUM-HYDROGEN TANDEM MASS SPECTROMETER

The design, fabrication and testing of the bakeable tandem mass spectrometer was the major and final task of the present program.

The design involved the elimination of all materials incompatible with ultra-high vacuum practice and a bake-out temperature of 450°C.

The body was fabricated of type 304 stainless steel, replacing the original copper envelope; Micalex and Teflon insulators were replaced by alumina ceramic. In particular, the filament mount was redesigned by fusing two chrome-iron stainless steel filament supports directly into an alumina body with Pyrocera Cement #95. The filament and filament connections were spot-welded directly to the filament supports. In this manner, uncontrolled rotation of the filament supports, a weakness of the original design, was eliminated, whereas replacement of the filament was greatly facilitated.

The number of demountable joints was minimized. Where necessary, flanges with ConFlat profiles and OHFC copper gaskets were utilized. All permanent joints were argon-arc welded.

The symmetrical geometry of the tandem mass spectrometer analyzer was preserved, as were the dimensions of the ion source, center slit, and suppressor slit. To reduce costs, many original components were utilized, including electrodes and deflection magnets.

The instrument was mounted on a baseplate compatible with Javelin dimensions.

In order to facilitate storage, the instrument can be pumped via a non-bakeable one-inch isolation valve and back-filled with argon, to prevent deterioration of the multiplier. However, it can also be mounted directly via a stainless-steel ConFlat flange to an ultra-high vacuum system for bake-out.

A layout of the instrument is shown in Figure 2, photographs showing the complete instrument and various details are shown in Figures 3 and 4.

The mass spectrometer was first leak-checked on a helium leak detector and all welded and flanged joints were found to be tight. Next, the instrument was evacuated with a zeolite-trapped oil dif-

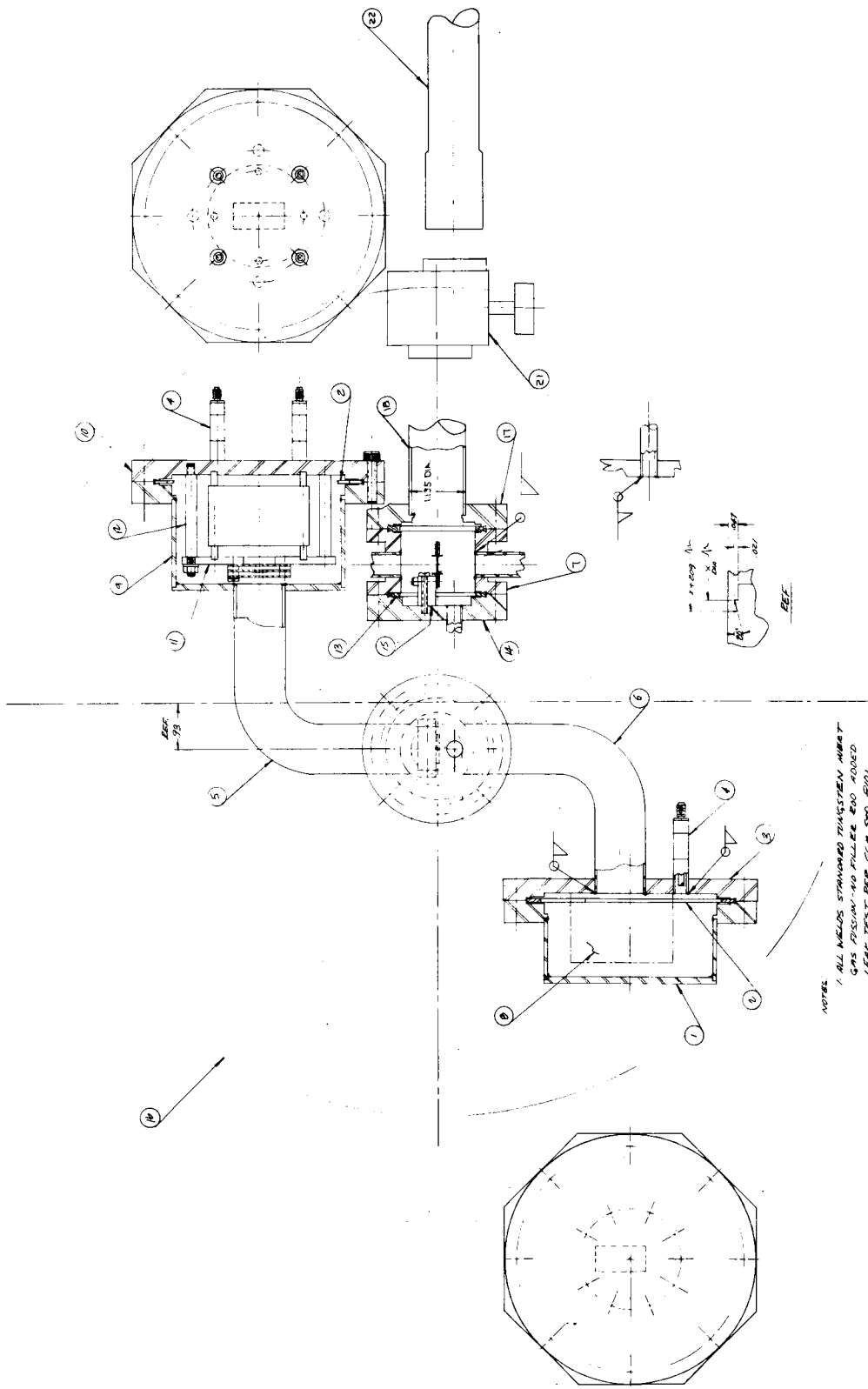


Figure 2. Javelin tandem mass spectrometer design.

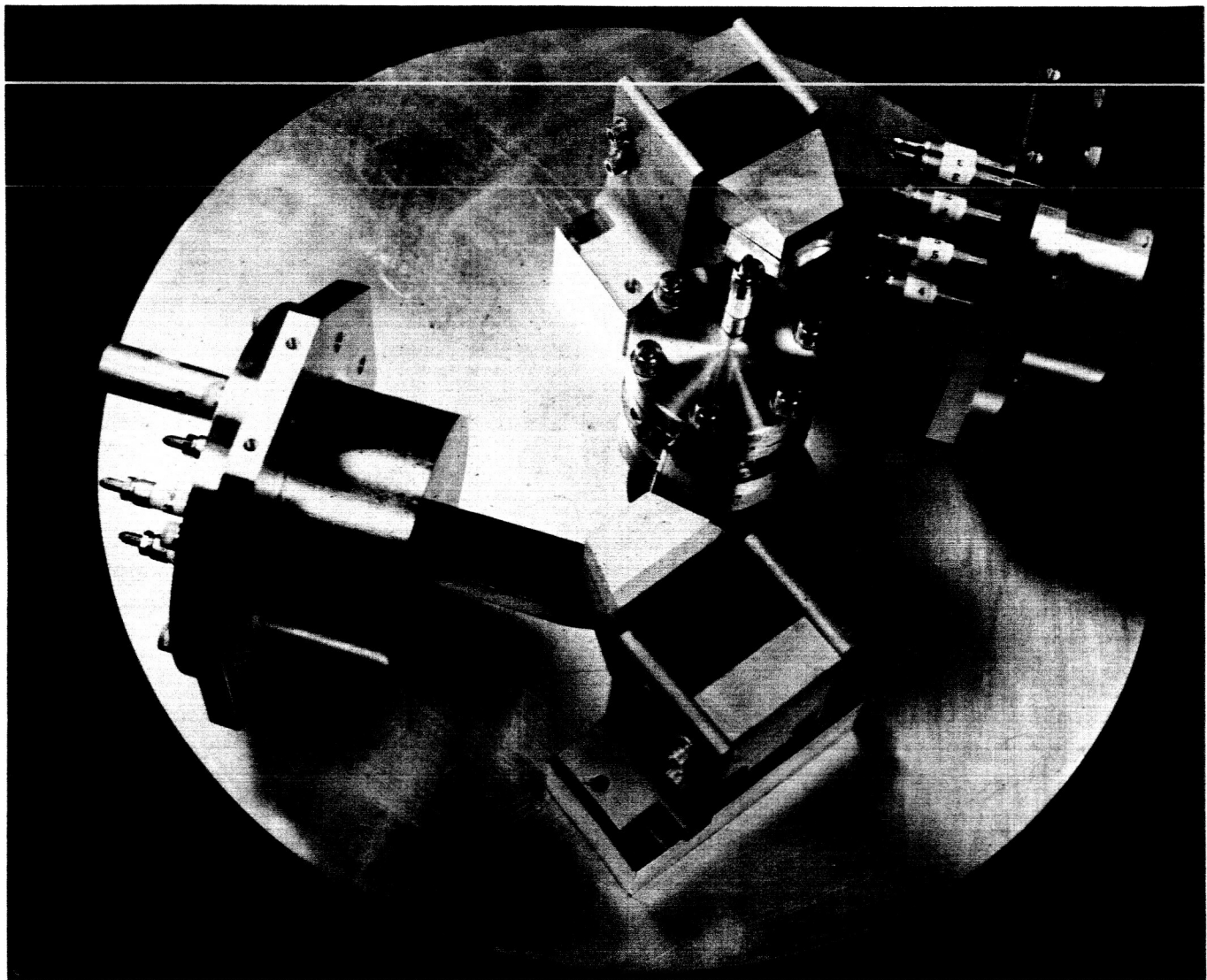


Figure 3. Javelin tandem mass spectrometer.



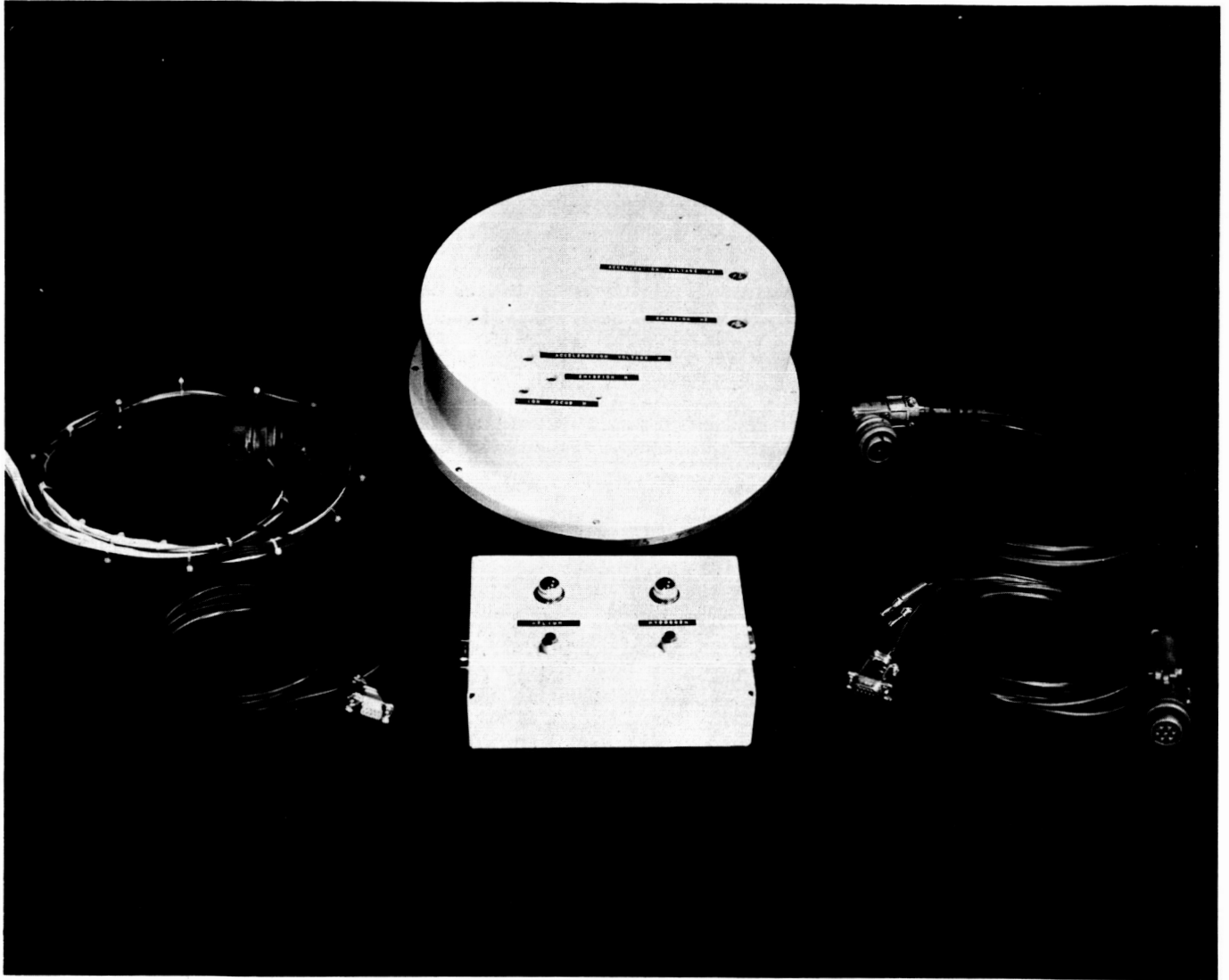


Figure 4. Mass spectrometer electronics and hydrogen-helium command unit.

fusion pumped system capable of a base pressure in the  $10^{-8}$ -Torr range. Because of the tight tolerances in manufacture, the optimization of ion transmission through the analyzer was straightforward. The instrument can be readily tuned to indicate  $m/e = 2$  and  $m/e = 4$ . The sensitivity appears to be of the same order as that of the original instruments; however, as mentioned previously, the minimum detectable signal cannot be determined to any degree of accuracy because of the excessive multiplier noise. A good indication of the source of noise is the rise in pressure when the multiplier is operated at high input levels. Presumably, the intensive electron bombardment of the unbaked dynode surfaces releases large quantities of adsorbed gas.

The coding of terminals is identical with the original Aerobee instrument, with the following exceptions: terminals 8-8 of the ion source are not in use; terminals 3 and 5 of the multiplier have been omitted.

## RECOMMENDATIONS FOR FURTHER WORK

In order to clarify the limits of performance of the MM-1 focused mesh multiplier, it will be necessary to pump the mass spectrometer in oil-free fashion and to bake it up to 450°C. This can be accomplished most readily with a sputter-ion pump and a bakeable isolation valve temporarily connected to a roughing pump, provided radiation and charged particles are excluded from the multiplier as shown in Appendix B.

Whereas the bakeable tandem instrument specifically meets the requirements of compatibility with Javelin specifications, it will have to be shown experimentally to what extent the residual hydrogen pressure can be reduced by a bake-out. However, since hydrogen is the principal residual gas in ion-pumped systems, a meaningful answer to this question can be obtained only with a well-trapped mercury-pumped ultra-high vacuum system.

As presently constituted, neither of the two multipliers tested is designed to produce a positive output signal, as is desirable for a logarithmic electrometer read-out and was provided in the original Aerobee design. This problem, as well as the type of electrometer amplifiers for ion-source and multiplier output monitoring will have to be looked into.

The new mass spectrometer will also lend itself to a more fundamental investigation regarding memory effects produced by ionically-pumped gases re-emitted under subsequent ion bombardment in the ion source. This phenomenon was recognized by GCA scientists, as shown in Appendix C, and constitutes a serious limitation of the ultimate helium sensitivity; undoubtedly, it also affects the limit of hydrogen detection. With a mercury diffusion-pumped bakeable system, the sources of such memory effects will be more readily identifiable and it may be possible to reduce and minimize them. It should be pointed out that at the low ambient pressures encountered in a Javelin experiment, errors due to memory effects are likely to be less serious than at lower altitudes where bombardment by gases other than hydrogen and helium is more severe and may release significant amounts of hydrogen and helium buried in the ion source electrodes during previous exposures to these two gases.

## APPENDIX A

### Particle Number Densities to 10,000 km Altitudes Above the Earth's Surface

R. A. Minzner

GCA Technology Division  
Technical Note

15 July 1965

Recognizing that the problem of estimating particle number densities at distances of the order of one earth radius above the earth's surface is beset with numerous uncertainties and unknowns, not the least of which is the charge condition of the particles, it is nevertheless desirable, for want of a better approach, to calculate such number densities on the basis of some very simple assumptions given below. These assumptions are certainly applicable to 500 km altitude and appear to apply at 1000 km altitude. Whether they continue to apply at 10,000 km altitude is unknown. It is likely that at 10,000 km the solar atmosphere, which is not considered in the calculations, dominates the region, and the computed values of number density may be too low.

The calculation involves the assumption of constant temperature and the use of the equation of state and the hydrostatic equation in terms of geopotential. Consequently the variation of the acceleration of gravity is eliminated from the differential equations. The transformation of geopotential to geometric altitude, however, does involve the assumption of the validity of the inverse square law at these altitudes. This relationship may be degraded somewhat at these altitudes, but it ought to be as good, or better, than the other assumptions. A further assumption (reasonably well substantiated) is that the neutral particles are primarily hydrogen and helium at altitudes above 2000 km but the ratio of neutral to ionized hydrogen at these altitudes is unknown.

The relationships are:

$$\frac{n}{n_b} = \exp \left[ \frac{-GM}{RT} (h - h_b) \right]$$

$$\text{and } Z = \frac{r h}{\left( \frac{g_o}{G} \right) r - h}$$

where

- n = particle density at altitude h
- $n_b$  = particle density at altitude  $h_b$
- $G = 9.80665 \text{ m}^2 \text{ sec}^{-2} (\text{m}')^{-1}$
- R = universal gas constant
- M = molecular weight of particular gas
- T = kinetic temperature
- Z = geometric altitude
- r = effective earth's radius (6,356.766 m)
- $g_o = 9.80665 \text{ m sec}^{-2}$

As observed by Reber (1964) and Reber and Nicolet (1965),  $n_b$  for helium is  $8 \times 10^5 \text{ cm}^{-3}$  at 300 km altitude,  $n_b$  for neutral hydrogen is  $1 \times 10^5 \text{ cm}^{-3}$  at the same altitude as computed by L. Miller (1956). In the calculations, constant temperatures of  $1000^\circ\text{K}$  and  $2000^\circ\text{K}$  represented two conditions of solar activity.

The calculated profiles of number densities versus altitudes are shown in the attached figures. Figure 5 shows number densities for helium and hydrogen based on a  $1000^\circ\text{K}$  isothermal atmosphere above 300 km. Figure 6 shows number densities of helium and hydrogen based on an isothermal atmosphere at  $2000^\circ\text{K}$ . Figure 7 shows the influence of the 2 to 1 change in temperature on hydrogen number density assuming a constant value at 300 km.

If, at altitudes of about 1 earth's radius, there is a significant amount of gas in some way rather directly associated with the sun, the temperature of this mass of gas would most probably be much greater than  $2000^\circ\text{K}$ . Because of the large mean free paths at these altitudes however, there would be little interaction between the sun-associated gas and the earth-associated gas. Thus, the two masses would exist

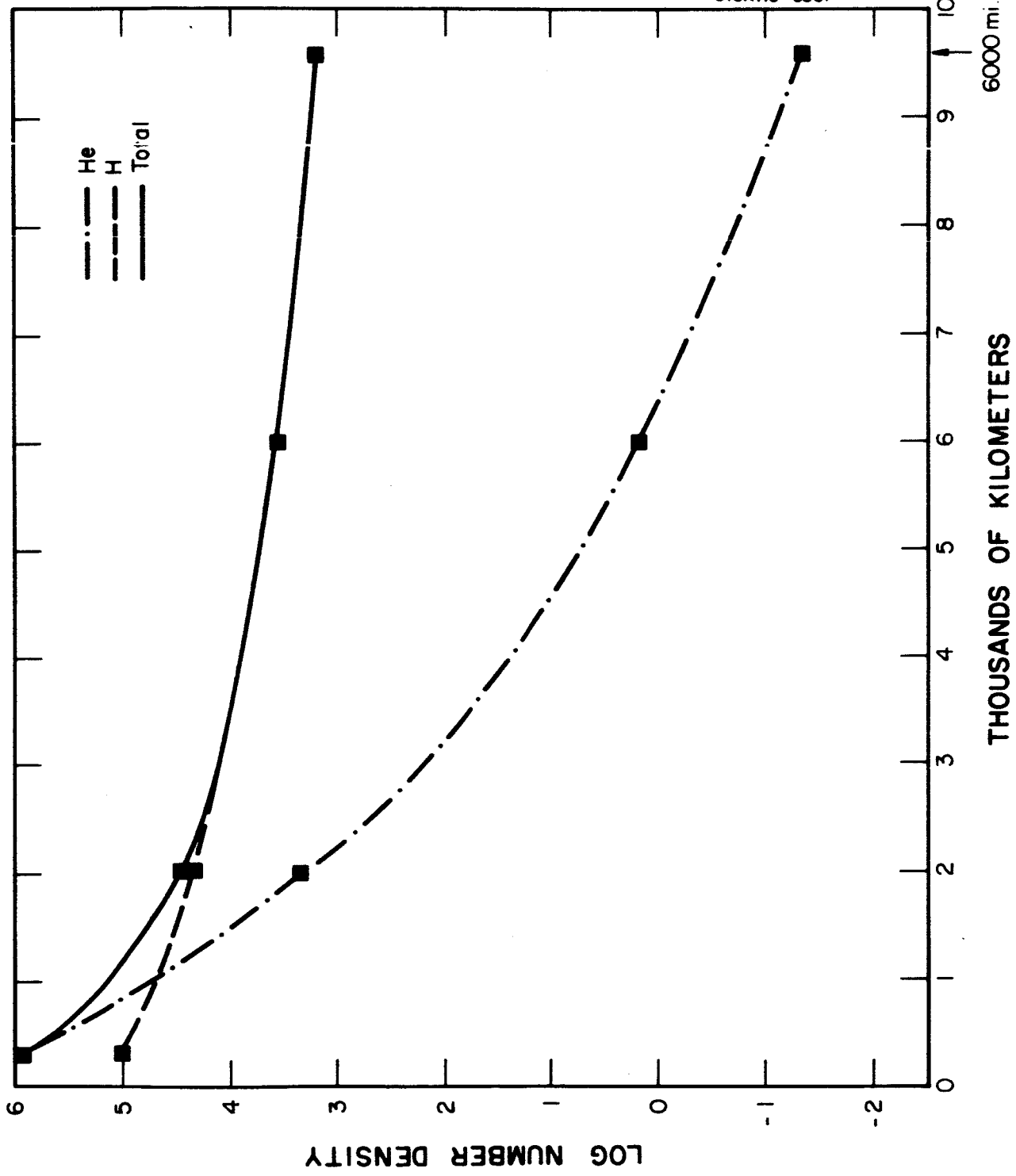


Figure 5. Number densities computed on the basis of an isothermal atmosphere at 1000° K.

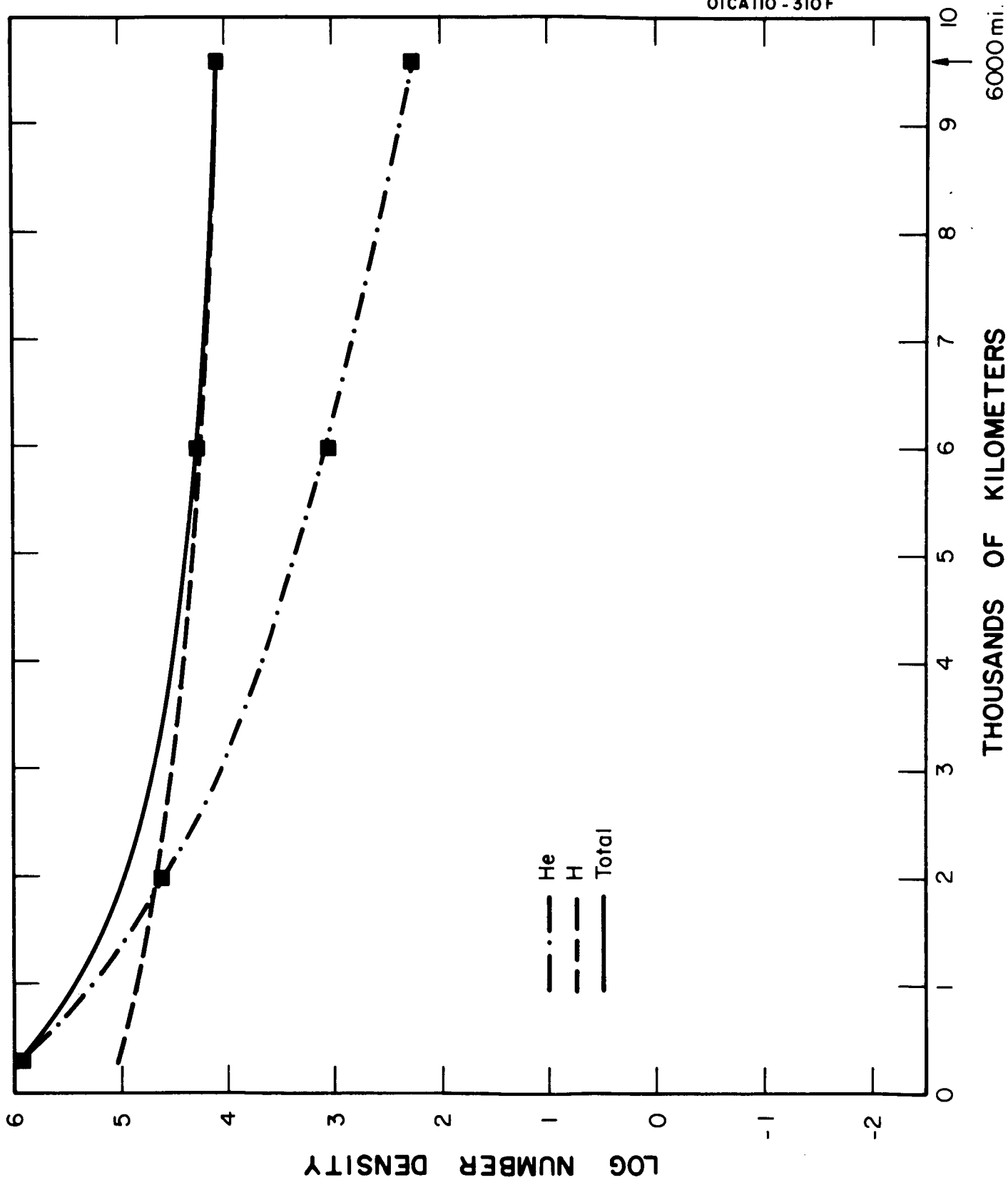


Figure 6. Number densities computed on the basis of an isothermal atmosphere at 2000°K.

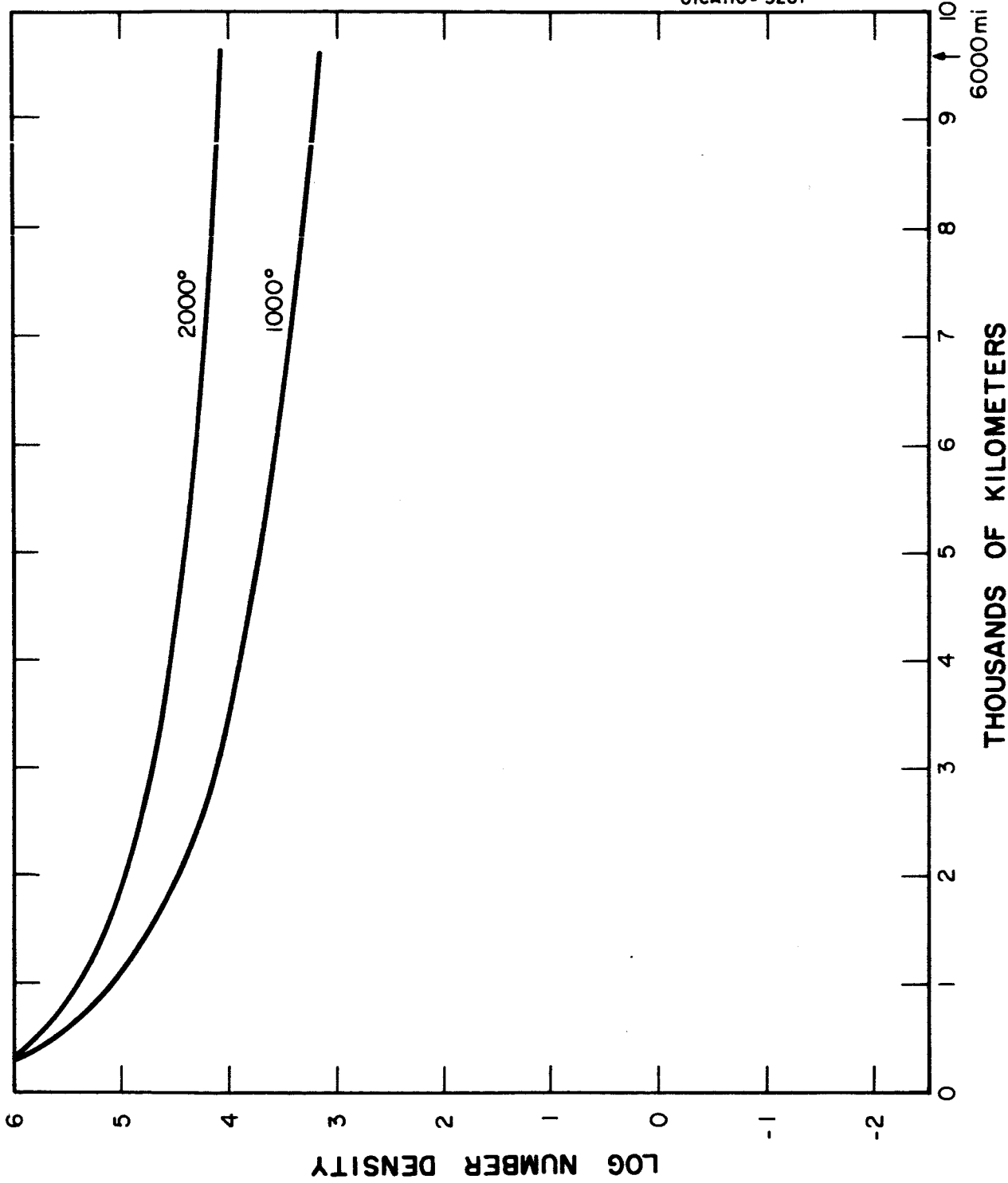


Figure 7. Neutral hydrogen number densities computed on the basis of isothermal atmosphere at 1000 and 2000°K.



within the same volume at two widely different temperatures without either affecting the other, except perhaps at altitudes below 200 km. It would be interesting to be able to measure the kinetic energy of individual particles in suitable samples of particles swept out by a probe on a vertical sounding rocket during its flight to great altitudes. Such a measurement might determine whether two distinct energy maxima exist, and thereby support or refute the concept of two masses of gas each at different temperature.

#### References

- Reber, C. A., "Data from Explorer 17 on Composition of the Upper Atmosphere", J. Geophys. Res. 69, 4681-4685 (Nov. 1, 1964).
- Reber, C. A. and M. Nicolet, "Investigation of the Major Constituents of the April-May 1963 Heterosphere by the Explorer XVII Satellite", NASA X-650-65-64, Goddard Space Flight Center, Greenbelt, Md., (Feb. 1965).
- Miller, L. E., "Molecular Weight of Air at High Altitudes", J. Geophys. Res., 62, 351-365 (Sept. 1957).

Reprinted from THE REVIEW OF SCIENTIFIC INSTRUMENTS, Vol. 36, No. 4, 549, April 1965  
Printed in U. S. A.

### Suppression of Excited Species from Sputter-Ion Pump by a Zeolite Trap\*

A. E. BARRINGTON

*Physics Research Division, Geophysics Corporation of  
America, Bedford, Massachusetts*

(Received 23 September 1964; and in final form, 3 December 1964)

IN a current mass-spectrometer development,<sup>1</sup> the performance of a twenty-stage electron-multiplier ion collector was seriously degraded by a background signal whose source was traced to a 15 liter/sec sputter-ion pump in the magnetic analyzer section. Charged particles and radiation from the pump which operated at  $10^{-7}$  Torr were the major contributors to the signal, but these could be excluded from the multiplier by an appropriate baffle arrangement. However, there remained a small residual multiplier signal corresponding to about one count per second (the multiplier readily detects single ions or photons). It was demonstrated conclusively that the signal was produced only when the sputter-ion pump was operating but that it was not caused by charged particles or radiation from the pump discharge. Since argon was the principal residual gas in the system, the effect was attributed to photons radiated by metastable argon atoms which were able to migrate from the pump into the multiplier section.

It was possible to prevent this migration by means of a zeolite trap (molecular flow conductance 15 liter/sec) placed in series with the sputter-ion pump. The trap

consists of a wire cage mounted on radial vanes which are welded onto the exterior of a hollow stainless steel cylinder. The cage is filled with 0.9 kg of Linde 5A molecular sieve. The cage assembly is contained in a 15 cm diam outer cylinder with two flanged 3.8 cm ports. A 3 mm annular space separates the cage from the outer cylinder. The inner cylinder can accommodate a 1.27 cm diam 150 W Inconel-sheathed cartridge heater; if required, it can also serve as an 8 h capacity liquid-nitrogen reservoir. Following a vacuum bake at 350°C during which the trap was pumped by a well-trapped mechanical pump, it has operated continuously without reactivation for several months. Complete trapping of excited species takes place at room temperature. When the trap is chilled by liquid nitrogen, it also enhances the available pumping speed for argon and prevents the occasional instabilities which are to be expected on prolonged argon exposure of a sputter-ion pump.<sup>2</sup>

\* This work was supported by the National Aeronautics and Space Administration under Contract No. NASw-839.

<sup>1</sup> H. J. Liebl and R. F. K. Herzog, *J. Appl. Phys.* **34**, 2893 (1963).

<sup>2</sup> A. E. Barrington, *High Vacuum Engineering* (Prentice-Hall, Inc., Englewood Cliffs, New Jersey, 1963), pp. 101-106.

## APPENDIX C

Reprinted from *The Journal of Vacuum Science and Technology*

Vol. 2, No. 2, March/April 1965

### **Helium Memory Effects Observed with Tandem Mass Spectrometer**

**A. E. Barrington, R. F. K. Herzog,  
and G. O. Sauermann**

*Physics Research Division,  
Geophysics Corporation of America,  
Bedford, Massachusetts*

(Received 29 September 1964)

It is customary to define the sensitivity of a helium mass spectrometer leak detector as the helium partial pressure which would produce the minimum detectable electrical signal on the most sensitive scale of the helium partial pressure indicator. Based on this definition, the minimum detectable helium partial pressure

is deduced by extrapolation from a helium partial pressure calibration at some convenient higher helium partial pressure.

This extrapolation procedure is acceptable down to helium partial pressure detection limits of  $10^{-11}$  Torr; however, it cannot be applied indiscriminately at lower pressures as, for example, in a recent publication.<sup>1</sup> Several unsuspected sources of helium may exist in a helium leak detector system which can contribute a helium background several orders of magnitude larger than the minimum detectable partial pressure predicted by extrapolation. In such a case, the minimum detectable helium partial pressure depends predominantly on the magnitude of the helium background and its stability, rather than on the minimum detectable electrical signal.

To demonstrate this, several observations with a stigmatic-focusing magnetic tandem helium mass spectrometer, equipped with a Nier-type ion source and an electron multiplier, will be reported. The instrument was constructed in this laboratory under contract with the National Aeronautics and Space Administration (contract NAS 5-270) for upper atmosphere rocket measurements. Before exposure to other gases the instrument was run repeatedly at pressures as high as  $10^{-4}$  Torr of pure oxygen, admitted through a commercially available<sup>2</sup> silver diffusion leak.<sup>3</sup> Over the electron acceleration voltage range between 100 and 200 V no measurable signal at mass 4 was observed. This is an indication that  $O^{+}$  ions (threshold potential 77:39 V)<sup>4</sup> which would occupy the same position on the atomic mass scale as  $He^{+}$  ions were not produced in any measurable quantity, although the electron energy was considerably higher than the threshold value.

However, a signal at the helium peak was registered invariably when a run on pure oxygen was performed immediately after a run on air at  $10^{-4}$  Torr. It was established that this signal was due to helium which originated in the ionization gauges used to monitor the system pressure. The effect was easily identified by installing a valve between gauge and mass spectrometer. On closing the valve, the signal diminished immediately. The quantity of helium produced by a Penning cold-cathode gauge exceeded that produced by a Bayard-Alpert glass gauge. Since the helium signal was proportional to the oxygen pressure, it was concluded that helium atoms were released in the gauges under oxygen ion bombardment. Even with the gauges switched off, some thermal release of helium was observed. This helium release was considerably smaller than the amount of helium produced under ion bombardment, but, in the case of the glass gauge, greater than the theoretical diffusion rate of atmospheric helium through the gauge wall.

The helium produced in the Bayard-Alpert gauge could be reduced below the partial pressure detection limit of the mass spectrometer (approximately  $10^{-13}$  Torr) by degassing the gauge for about one hour. Since the Penning gauge could not be degassed, it was removed from the system. Because of the much higher ion energy in its cold-cathode discharge, memory effects were more pronounced<sup>5, 6</sup> and could not be readily eradicated.

The tandem mass spectrometer itself did not exhibit a helium memory when it was operated only on air. However, serious residual effects occurred when it was operated on a standard helium leak as is customary for calibration and peak adjustment. In this case, bombardment-induced re-emission proportional to the

pressure of subsequently admitted oxygen was observed. The helium memory could only be eliminated by replacing those ion-source electrodes which were under intensive ion bombardment.

The above results are in line with recent observations on ion-bombardment-induced re-emission and thermal desorption of inert gases reported in the literature.<sup>7, 8</sup> They also point to a weakness of the current definition of the minimum helium partial pressure detectable by a particular ultrasensitive helium leak detector. When such a device is connected to a small leak-free volume, an apparently memory-free performance can readily be obtained by evacuating it to a sufficiently low pressure; the minimum detectable helium partial pressure will then indeed be close to the value obtained from a helium calibration extrapolated to the minimum detectable electrical signal. In practice, however, the system to be tested can rarely be evacuated to this same low pressure; the pressure may well be limited to a value several orders of magnitude higher by wall-outgassing and vapor-pressure effects, rather than by air leaks. In consequence, if a helium memory exists, the helium partial pressure will rise when ions of this residual gas bombard various critical surfaces. In particular, most leak detector systems include an ionization gauge which actuates protective over-pressure relays; as indicated above, this gauge can be the major source of helium contamination. It does not seem unreasonable, therefore, to specify a perhaps more stringent but certainly more realistic procedure whereby the minimum detectable helium partial pressure would be defined as the maximum fluctuation of the signal at the helium peak during a period when the mass spectrometer operates at its maximum rated pressure in a helium-free atmosphere. As demonstrated above, such a test is simple and requires only a relatively inexpensive commercial silver diffusion leak. By periodic tests of this kind, the condition of the leak detector can also readily be monitored over a period of prolonged use.

<sup>1</sup> F. Kirchner and A. Benninghoven, *Vakuum Tech.* **12**, 207 (1963).

<sup>2</sup> K-B Glass Apparatus Company, Schenectady, New York.

<sup>3</sup> J. R. Young and N. R. Whetten, *1961 Transactions of the Eighth National Vacuum Society Symposium* (Pergamon Press, New York, 1962), pp. 625-627.

<sup>4</sup> M. von Ardenne, *Tabellen der Elektronenphysik, Ionenphysik und Übermikroskopie* (VEB Deutscher Verlag der Wissenschaften, Berlin, 1956), p. 479.

<sup>5</sup> A. R. Hamilton, *1961 Transactions of the Eighth National Vacuum Society Symposium* (Pergamon Press, New York, 1962), pp. 388-399.

<sup>6</sup> A. E. Barrington, *High Vacuum Engineering* (Prentice-Hall, Inc., Englewood Cliffs, New Jersey, 1963), p. 162.

<sup>7</sup> L. H. James and G. Carter, *1962 Transactions of the Ninth National Vacuum Society Symposium* (The Macmillan Company, New York, 1962), pp. 502-509.

<sup>8</sup> P. G. Smeaton, G. Carter, and J. H. Leck in Ref. 7, pp. 491-496.