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Space Physics Research Laboratory

Technical Report

REVIEW OF ATMOSPHERIC COMPOSITION MEASUREMENT TECHNIQUES

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ORA Project 05911

under contract with:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
CONTRACT NO. NAS 8-11054
HUNTSVILLE, ALABAMA

administered through:

OFFICE OF RESEARCH ADMINISTRATION ANN ARBOR

May 1964

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1. INTRODUCTION

The purpose of this report is to give a concise review of the present state of the art in atmospheric composition measurement techniques. A portion of the report deals with basic problems associated with composition measurements, and in the discussion of the different available techniques, their relative advantages and disadvantages are pointed out.

The terrestrial atmosphere can be divided into two regions in terms of composition characteristics. The homosphere is the region characterized by complete mixing, which means that the abundance ratios of the principal constituents remain constant. The region extends from ground level to an altitude of about 85 km. Above this level the composition changes with height, due mainly to dissociation and diffusion, and the region is called the heterosphere. Since the problems associated with the measurement of composition in these two regions are very different, they will be discussed under separate sections.

The number density of charged particles in the upper atmosphere—below a few thousand kilometers—is extremely small compared to the neutral gas density, but these particles are nevertheless very important. The electrons control the propagation characteristics of electromagnetic waves, and the ion composition must be known to fully understand such significant processes in the atmosphere as ionization, recombination, dissociation, etc. Simultaneous measurements of neutral and ion composition minimizes the assumptions necessary to interpret the data and therefore makes the results more significant. Section 5 is, therefore, devoted to a discussion of ion composition measurement techniques.

2. HIGH ALTITUDE NEUTRAL PARTICLE COMPOSITION MEASUREMENT TECHNIQUES

2.1. GENERAL REMARKS REGARDING DIRECT MEASUREMENTS

The transport of a mass spectrometer by the spacecraft to the region of interest was among the first direct composition measurement techniques employed. This approach provides in situ direct measurements. The general problems associated with this direct technique will be discussed first, before giving a short review of the different instruments which may be used for such measurements.

The transition between the homosphere and the heterosphere corresponds to an altitude where the mean free path of the neutral gas particles is of the order of 1 cm. Since all commonly used mass spectrometers require that the particle mean free path be at least of the same order as the dimension of the instrument, this technique is limited to measurements above about 90 km, unless some special steps are taken (see Section 3).

One of the major problems connected with direct measurements at high altitudes is the contamination of the ambient environment by gases originating from the spacecraft which carries the instrument. The payload is often separated from the rocket itself to minimize this problem. It is necessary to keep the separated payload vacuum sealed and its surface clean. The openings to the mass spectrometer should be placed such that a particle leaving the payload, either due to direct leakage or surface desorption, cannot reach the opening unless it has undergone at least one collision. Since the probability of a particle entering the spectrometer opening via collisions is small, if all these precautions are taken, the major problem will be the outgassing from surfaces in the vicinity of the ion source. The outgassing near the ion source can be minimized considerably if the ion source and spectrometer are kept under a good vacuum before and during the launch period. This has been done successfully on a number of occasions. 2-5 In these cases the spectrometers were evacuated, and sealed to pressures down to 10^{-5} - 10^{-8} torr, and opened by remote control, to the ambient atmosphere only at high altitudes. Of all the presealed spectrometers flown to date, one on Explorer XVII2 appears to have attained the lowest background pressure, which was in the order of lx10-10 torr. Ionization gages flown on the same satellite also achieved a background pressure in the order of 10-10 torr, indicating that improvements are necessary to permit direct measurements at altitudes where the ambient pressure is less than this value.

The uncertainty in the relation between the particles which finally get analyzed in the spectrometer and the ambient population causes further difficulties in the interpretation of direct composition measurements. This is especially a problem for highly reactive gases such as atomic oxygen. basic principle of operation of all spectrometers is to first ionize the gas particles and then analyze the resulting ions according to their e/m ratio. The number of collisions that the particles will have with the surface of the spacecraft before reaching the ionizing region, should be kept as low as possible, in order to minimize the possibility of chemical reactions (e.g., recombination). An unobstructed ionizing electron beam outside the spacecraft, in front of the opening to the mass spectrometer, is the ideal arrangement since the particles reaching the ionizing region will have had one collision at the most. There are of course practical problems which make such simple, ideal arrangements difficult to achieve. One problem, for example, stems from the fact that ambient positive ions must be kept out, since by reaching the collector they would cause erroneous results. This can be done by placing a positive grid around the opening, however, this causes further difficulties which are mainly due to possible ionization by the accelerated electrons.

This "open source" arrangement although minimizing some of the difficulties mentioned earlier, creates a new problem which is as follows. Considering a spectrometer with an ideal focusing arrangement, the density measured corresponds to the density at the ionizing source, and the relation between the density there and the ambient density is extremely complex. A detailed knowledge of surface accommodation coefficients, reflection characteristics, etc., is needed to obtain such a relation. Some theoretical work has already been done towards obtaining a solution to this problem^{5,7} but still a lot more has to be done before a completely satisfactory solution is found. In interpreting the results of experiments carried out to date, rather crude assumptions have been employed. However, the errors thus introduced are probably still less than the error due to the uncertainties in the absolute calibration of the instrument. Experimental techniques for obtaining such a relationship have been considered and one is planned for the near future. In this experiment two spectrometers will be flown, one placed behind a knife edge orifice and one with an open source arrangement. The relation between the ambient and chamber densities for a knife edge opening is well known. The measurement of a relatively stable gas such as N_2 or He by both a spectrometer behind such an orifice and by a spectrometer with an open source will provide a calibration for the latter. The extension of such a calibration to less stable gases is not straightforward, since again a number of simplifying assumptions are necessary. Laboratory experiments have also been carried out, 9 but the extension of these results to actual spacecraft applications is also questionable.

It should also be mentioned that the measurement of molecular oxygen abundance by a spectrometer in a chamber behind a knife edge orifice is being considered as a technique for the determination of the ambient atomic oxygen concentration at high altitudes. The reasoning for such an experiment is based on the assumption that "all" the atomic oxygen changes to molecular oxygen due to the very large number of collisions of the particles with the chamber walls and therefore at altitudes where the ambient molecular oxygen density is small, this technique can provide atomic oxygen information.

A further important consideration in the selection of an open source spectrometer for sounding rocket or satellite measurements is the fact that due to the vehicle velocity the effective energy of the arriving particles may be as high as 15 eV. This means that the ion source must have extremely good focusing characteristics and the spectrometer must not be very sensitive to initial velocities.

2.2 REVIEW OF PRESENTLY AVAILABLE INSTRUMENTS FOR DIRECT MEASUREMENTS

2.2.1. The rf (Bennett) Spectrometer

One of the first spectrometers to make successful measurements on a rocket flight, was a specially modified Bennett radio-frequency instrument. $^{10-12}$ In such an instrument, shown in Fig. 1, the particles, after having been ionized,

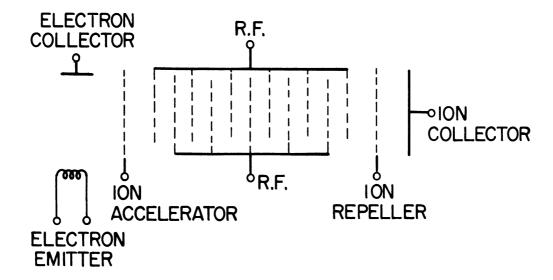


Fig. 1. Schematic diagram of the rf spectrometer.

are sent through a series of grids which have an rf voltage applied to them. The transit time of the ions between grids depends on their e/m ratio, so, for a given ion, there is a frequency which will accelerate them as they pass through successive grids, whereas the other species will be out of phase, and gain no net energy.

This spectrometer has been widely used both here in the U.S. and the U.S.S.R. One of the important relative advantages of this instrument is that for its operation it does not need a magnetic field, as some other spectrometers do. This is a significant factor in making it compatible with other experiments to be carried on the same spacecraft. A problem which is associated with this spectrometer is its tendency to produce ghost signals.

2.2.2. Magnetic Deflection Spectrometers

There are a large variety of instruments in existence which use the deflection caused by a magnetic field to separate the ionized gas particles of different e/m ratios. 13 Sometimes a combination of electrostatic and magnetic deflection is employed 14 , 15 (see Fig. 2). Recently a number of experiments using such instruments have been successfully carried out, 2 , 4 To keep the instrument size reasonable, the Larmor radii of the gases analyzed have to be reasonably small, which in turn means that a strong magnetic field is needed (e.g., the Larmor radius of N_2 in a field of 2000 gauss is 5.4 cm for a typical ion energy of 200 eV).

This kind of instrument is capable of providing excellent resolution characteristics. Another important advantage, as far as upper atmosphere measurements are concerned, is that it can measure simultaneously the abundance of numerous constituents. This can be of great advantage in interpreting the results.

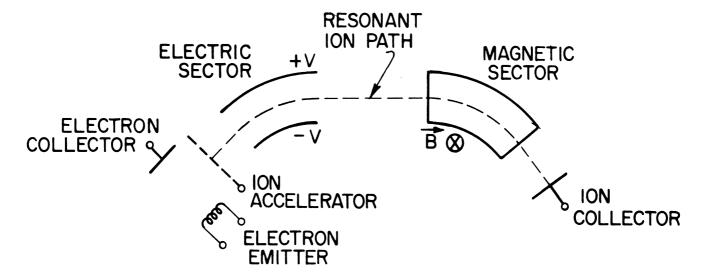


Fig. 2. Schematic diagram of a double focusing (electric and magnetic) mass spectrometer.

2.2.3. Quadrupole Mass Filter

The quadrupole mass filter 16 is a relatively recent development. It is an electrostatic instrument in which the ions are injected along the axis of the poles, as is shown in Fig. 3. The trajectory of most of the ions becomes unstable under the influence of the transverse, combined rf and dc fields applied to the rods. Due to the increasing amplitude of their transverse motion the ions collide with the rods and are thus removed. Only ions within a small range of e/m value have stable trajectories and reach the collector which is at the far end of the rods.

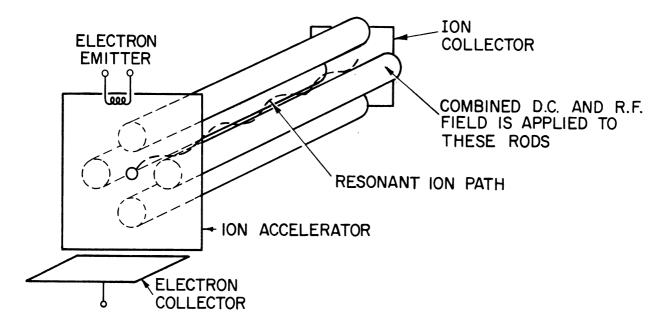


Fig. 3. Schematic diagram of the quadrupole mass filter.

This is a spectrometer which does not need a magnetic field and in which the ionized particles reach the collector without having to pass through a number of grids, thus overcoming some of the disadvantages of the previously discussed instruments. Since the mass separation is due to transverse instabilities, this instrument can accept a relatively large range of velocities, which can be quite a significant advantage especially for satellite application as was pointed out in Section 2.1. It can also operate up to a relatively high pressure ($\approx 10^{-3}$ torr). One drawback which is associated with this spectrometer at the present is that the power requirements are relatively high.

2.2.4. The Time-Of-Flight Spectrometer

The time-of-flight spectrometer 17 is also an electrostatic instrument. In this instrument bunches of ions are first accelerated by a dc voltage and then allowed to drift through a field free region as indicated in Fig. 4.

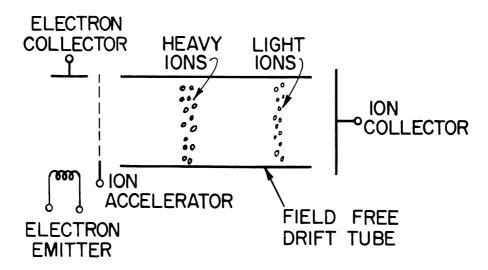


Fig. 4. Schematic diagram of the time-of-flight spectrometer.

Since all the ions are accelerated by the same potential, the time taken by the ions to reach the collector at the other end of the drift space is only a function of their e/m ratio. Analysis is therefore achieved by counting only those particles which arrive within a given time interval. This instrument again has the advantage of no magnetic field; however, it requires a somewhat more complex ion source since the ions have to be accelerated into the drift space in bunches. Being a pulsed instrument, only a portion of the available particles reach the collector, resulting in short duration current pulses. This means that a current detector having very high sensitivity and a fast response is needed. An electron multiplier satisfies these requirements but also creates some further problems which are discussed in Section 2.3. Extremely fast spectral sweeps are inherent to the time-of-flight spectrometer, since a complete sweep is achieved in a time which corresponds to the drift time, in the analyzing tube, of the heaviest particle to be measured.

2.2.5. The Omegatron

The omegatron 18 which has been the workhorse of vacuum research has also found considerable use in atmospheric measurements. The omegatron is in effect a miniature cyclotron (Fig. 5). Under the influence of crossed radio frequency electric and static magnetic field the resonant ions describe an Archimedes spiral and are then collected. The problems arising out of the need for a strong magnetic field have been pointed out earlier. An internal ion source is inherent for the operation of this instrument. This means that it should only be used for the measurement of such relatively stable gases as N2, He, etc., and then the internal source is not a disadvantage since if used behind a knife edge orifice the uncertainties associated with an open source are eliminated (see Section 2.1). The simplicity and reliability of the omegatron makes it attractive for such measurements.

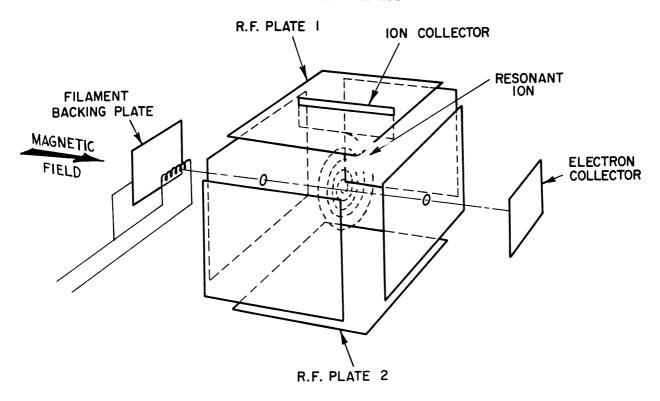


Fig. 5. Schematic diagram of the omegatron.

2.3. MEASUREMENT OF EXTREMELY LOW DENSITIES

Assuming that the problems associated with outgassing can be overcome, the instruments listed in Section 2.2 are limited at very low densities by the detection threshold of their ion current. Electron multipliers do significantly increase the detection capability of these instruments; however there are numerous problems associated with them. Some of the more important ones are:

(a) difficulty in obtaining constant gain characteristics over the large operating ranges usually encountered;

- (b) difficulty in ensuring good gain stability;
- (c) the high voltages requirements.

Whenever the gain of the multiplier permits the use of pulse counting techniques problems (a) and (b) are eliminated.

Another approach towards increasing the minimum measurable density is the use of integration methods. Ideally it is possible to "integrate" either the output current from the spectrometer or the number of particles available for measurement. There are no practical systems presently available which would permit significant imporvement from electronic integration. A technique which may provide a possible means of "particle integration" is a modified form of the desorption spectrometer technique 19,20 to be described next.

The desorption spectrometer is a device which collects particles during a given time interval and then analyzes them. In such a spectrometer a clean refractory metal surface chemisorbs gas molecules impinging on it. The rate of adsorption is related directly to the rate of impingment and is therefore a function of the ambient density. The gas particles absorbed are held to the surface by binding energies that are a function principally of the metalgas combination. When such a surface is heated the adsorbed particles are driven off at temperature corresponding to their binding energies. Thus, if the heating is carried out at a known rate and in a confined volume, ideally, it is possible to obtain a spectrum, since the desorption of each gaseous species is indicated by a sharp increase in pressure. Feasibility studies²¹ have shown reasonable agreement between theory and experiment for such gases as nitrogen, however, as soon as a more reactive gas such as hydrogen was used or simple binary mixture of gases were used the experimental behavior of the spectrometer did not follow simple theoretical predictions.

The extreme complexity of the sorption phenomena, even for simple gases, resulting in a multitude of binding states, indicates that more work has to be done before this approach can be used in practical applications. It seems that instead of using a total pressure indicator to measure the increase in pressure caused by the desorbing gases, the use of a partial pressure sensor would make the technique more feasible since the problems due to the uncertainty in binding energies would be eliminated. The problem of chemical changes during the adsorption and desorption process would, however, still be present.

2.4. INDIRECT COMPOSITION MEASUREMENT TECHNIQUES

In this section a brief discussion will be given of composition measurements by a number of indirect methods. In these techniques, the actual particles are not collected and analyzed by a device on the spacecraft, but the composition of the surrounding ambient particles is obtained by observing either an artificial or natural phenomena.

2.4.1. Optical Absorption Measurements

The spectrum of the sun's radiation reaching the earth is strongly altered by absorption processes taking place in the upper and lower atmosphere. A detailed knowledge of the attenuation of the sun's ultraviolet radiation as a function of altitude can be used to derive the composition of the atmosphere, since the rate of absorption is directly related to the ambient gas particle densities and their absorption cross sections at the frequencies under consideration. Since our present day knowledge of absorption cross sections is very limited, one needs to study the attenuation of many different wavelengths to be able to determine the ambient particle composition.

Since the end of World War II, when the V-2 rockets first became available for upper atmosphere research, spectrographs have been flown to study the sun's ultraviolet spectra. 22 Techniques have improved significantly since those early flights, permitting more detailed and accurate measurements. Recently a sophisticated grazing incidence grating monochromator was flown, 23 which measured the wavelength range from 250Å to 1300Å with a spectral resolution of 15Å to 30Å. Using the measured intensities as a function of altitude of the different wavelengths, the ambient number density of molecular nitrogen and oxygen as well as atomic oxygen was derived for the altitude region 170-210 km.

The main advantage in using this absorption technique for composition measurement is that it is relatively insensitive to such environmental problems as outgassing, which are important in direct measurements, and since no collection is involved, the problem of chemical reactions by gases such as atomic oxygen is eliminated. There are, however, also numerous disadvantages associated with this approach, the more important ones are as follows:

- (a) the measurement can be made only in the daytime;
- (b) in general the measurements require relatively large and expensive equipment and/or special controls are necessary to maintain proper orientation with respect to the sun;
- (c) the interpretation of the results is difficult because of very limited knowledge of absorption cross sections;
- (d) the technique does not provide fine structure details in the altitude profiles, since it is an integrating method.

Considering the relative advantages and disadvantages of this method, its greatest value lies in the measurement of "difficult" gases such as atomic oxygen. To overcome some of the disadvantages listed earlier, the possibility of flying an ultraviolet source on the same spacecraft with a spectroscope is being studied at this laboratory. In such an experiment, the light beam will travel through the neighboring ambient atmosphere and either its absorption or

the resultant reradiation will be measured and interpreted in terms of composition.

2.4.2. Ultraviolet Dayglow Measurements

The solar radiation incident upon the atmosphere is modified by several physical processes. Atomic absorption, of the incident radiation at certain frequencies is followed by reemission of the absorbed energy by resonance reradiation. The molecules will also absorb certain wavelengths of incident radiation and then reemit it in fluorescence. Rayleigh scattering is significant in the lower atmosphere. In the earth's atmosphere, the principal atomic lines that occur in the ultraviolet dayglow, which is the result of the processes named above, are the Lyman alpha 1216Å line of atomic hydrogen and the 1300Å resonance line of atomic oxygen, 3p-3s.

The measurement of the radiation at a given frequency, corresponding to the resonance reradiation of a given atom, at an altitude where the atmosphere is optically thin for this frequency, can be used for composition measurement. ²⁴ As an example, the measurement of the intensity of the 1300Å oxygen line as a function of altitude starting at about 300 km can be used to obtain an atomic oxygen profile, since the intensity is proportional to the integrated density. This technique holds great promise, especially for upper atmosphere and planetary fly by experiments, however, it is still in its infancy—no successful experiments using this technique have yet been reported—and thus much more work is necessary before it can be properly evaluated.

Auroras are caused by charged particle bombardment of the upper atmosphere. The study of the spectrum of the aurora emission can be used to investigate atmospheric composition, but due to its geographic limitation it cannot be considered a general technique.

3. LOW ALTITUDE NEUTRAL PARTICLE COMPOSITION MEASUREMENT TECHNIQUES

In the homosphere, where mixing is complete, the relative composition of the principal constituents remains constant. The minor constituents which are highly variable are, however, of great significance. For example ozone, which absorbs a portion of the UV radiation, is of great importance in the establishment of the heat balance in the lower atmosphere. Another example of the importance of certain minor constituent is the question of NO abundance in the 70-85 km region. 25 An NO concentration of only 10-10 times that of the total concentration is needed, to make X-rays unimportant in explaining the formation of the D-region under quiet solar conditions, a question which is still highly debated. The altitude where mixing ends and diffusive separation

starts can be determined by observing the change in the concentration ratio of noble gases. A detailed knowledge of the concentration of the minor constituents is therefore clearly needed for an understanding of the physics of these lower regions.

The principle of operation of the spectrometers described in the previous section is such that the particle mean free path has to be at least of the order of the instrument. This means that these spectrometers cannot be used directly at the lower altitudes. The use of simple vacuum pumps to reduce the pressure to a value necessary for these instruments has been proposed. A system consisting of a zeolite absorption pump surrounded by liquid nitrogen, built for this purpose, to be used with a quadrupole ion mass spectrometer, has been flown. 26,27 The system seems to provide the low pressure necessary during the ascent of a typical rocket flight, however, the question of how selective the pumping is has not been answered, therefore, there are difficulties present in interpreting the measured composition in terms of the ambient conditions.

Another direct composition measurement technique which was popular a few years ago, is the so called "sample bottle" approach. 28,29 In these experiments steel bottles, which were cleaned, evacuated and sealed on the ground, were carried by a rocket to a predetermined altitude, where the intakes were opened and then sealed off again a few seconds later. In later experiments to avoid possible uncertainties due to separation in a high speed flow, the rockets were ballasted to reach peak at the desired sampling altitude. Parachutes were used to recover the bottles.

In the earlier experiments the bottles were closed by both squeezing and heat soldering the inlet tube. This technique has a tendency of removing oxygen and it may have also caused considerable contamination of the sample. To overcome these difficulties, in later flights, sealing of the bottles was achieved by a pyrotechnically operated vice, which made a vacuum tight cold weld in the copper intake tube. In the early experiments it was difficult to be certain that the sample collected did not contain air originating from the rocket, and therefore in the later experiments the sample bottle assembly was separated from the rocket shortly after burnout, to minimize this difficult On a number of occasions radioactive CO₂ was released within the capsule to provide means for detecting, in the bottle, the presence of non-ambient gases. These tests indicated that the bottles did not contain any detectable amount of gas originating from the capsule.

This "sample bottle" approach is limited to relatively low altitude measurements where the densities are still relatively high. The technological problems associated with this technique (e.g., obtaining a good clean cold weld) could probably be eliminated but there are also major difficulties, which are inherent to the technique, namely the outgassing from the walls of the bottle, the uncertainty in the absorption coefficients and the chemical

reactions inside the bottle. Some of these problems are also present in the instantaneous direct measurement techniques, but it should be remembered that since a relatively long time elapses between the collection and the analysis of the bottle, these effects are integrated over the elapsed time. All these factors make this technique unattractive today, except for perhaps some special low altitude measurement of inert gases.

The use of optical techniques for these low altitude measurements holds great promise. A modified form of the absorption measurement technique was used recently 30 to obtain the ozone concentration profile up to an altitude of 85 km. In this arrangement a radiometer—whose spectral passband was centered at 2600\AA —is looking out from an attitude controlled satellite at a zenith angle of 90° . The efficiency of the radiometer system is independent of the azimuth and essentially independent of elevation over the $\pm 10^{\circ}$ encountered during the experiment. The variation in the absorption path in this case is obtained not by vertical motion, but it is due to the orbital motion of the satellite, which causes the radiometer to observe effective sunrises and sunsets. From the measurement of the variation of the measured radiation during a sunrise or a sunset, the concentration of the absorbing particles as a function of altitude can be calculated. The main difficulties associated with this technique are the uncertainty of the satellite position and the errors in the measured radiance values.

There is also a possibility of using the intensity of back-scattered solar radiation, measured by looking down on the atmosphere, for making low altitude composition measurements. A number of satellite flights are planned in which spectrometers covering the range from 1000-3000Å will look down on the atmosphere, and the results from these experiments will tell whether such interpretation of the dayglow is feasible.

As mentioned in Section 2.4.1 the use of a rocket borne optical source along with the measurement of either the absorption of the intensity of the emitted light in the neighboring ambient atmosphere or the resultant reradiation, seems feasible for low altitude measurements, however detailed feasibility studies of this technique are still needed.

4. ION COMPOSITION MEASUREMENT TECHNIQUES

The mass spectrometers discussed in Section 2.2—with their ion sources removed or turned off—can be used for ion composition measurements. The problem of ion sources is of course eliminated, however, the measurement of charged particles introduces a further difficulty. Any body immersed in a partially ionized gas, such as the ionosphere, will take up a potential with respect to the plasma, such that ion and electron currents reaching its sur-

face are equal. When photo-emission is negligible this potential is in the order of -1.0V. A positive ion sheath, of the order of a few Debye lengths, surrounds such a body and the result is that ions are accelerated towards the opening. This in general means that a knowledge of the shape and dimension of the sheath as well as the potential of the spacecraft is necessary to interpret the ion density measured by the device, in terms of the ambient density. Simplified theory predicts that the number of charged particles reaching a planar surface is independent of the sheath thickness and the potential across the sheath (as long as it is accelerating). Attempts to take advantage of this, have so far not been successful, probably because it is difficult to achieve an ideal planar configuration, but still this approach seems to be a most promising one.

Satellites, which travel with velocities in the order of 10 km/sec permit ion composition measurements by a completely different technique. Assuming that the ion thermal velocity is small compared to the satellite velocity, the kinetic energy of an ion in the reference frame moving with the satellite is directly proportional to its mass, therefore the measurement of the arrival energy is in effect a measurement of the mass. Of course this is also true for a neutral gas particle, however, using electrostatic means it is relatively easy to measure charged particle energies, whereas with present day technology the neutral measurement is not practical. Experiments based on this principle have been used successfully on a number of occasions. 32,33 It should be remembered that these measurements are restricted to very high velocity vehicles, and that the problems due to the uncertainties in sheath thickness and wall potential still exists and as a matter of fact are enhanced since the change of ion energy by its travel through the sheath can be significant and thus introduce uncertainties, especially for the light atoms, where the arrival energy is of the order of the potential drop across the sheath.

5. DISCUSSION

A detailed knowledge of the chemical composition of the atmosphere is necessary to permit a basic understanding of its behavior. This has been known for many years, and yet, today the knowledge of atmospheric neutral gas and ion composition is very limited. This does not reflect a lack of effort or interest, but is rather an indication of the problems associated with these measurements. Recently the number of experiments to determine atmospheric composition have increased significantly. Along with some of these experiments simultaneous measurements of other related physical parameters are being made which will make the interpretation of the result in terms of the physics of the atmosphere even more meaningful. Simultaneous neutral particle and ion composition measurements, for example, would be highly desirable, and these

could be made even more significant by coupling it with the simultaneous measurement of other parameters (e.g., electron temperatures, energy sources, etc.)

Instrument packages, containing multiple experiments of the type discussed here, can be made relatively small, such that medium size sounding rockets (e.g., Sparrowbee, Exos, etc.) can carry them. The advantage of using such vehicles arises from the fact that the experimenter has complete control over all phases of the experiment, including launch conditions.

The availability of space on large booster rockets (e.g. Saturn) raises the question of the desirability of using these vehicles for carrying composition measurement experiments. This report has described many different techniques, however none of these can be especially recommended for use with large boosters without first establishing a more concrete region of interest and a firm objective.

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