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A Mass Spectrometer Probe for Composition and Structure Analysis of the Middle Atmosphere Plasma and Neutral Gas

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Abstract. An instrument for composition and structure analysis of the atmospheric plasma and neutral gas at altitudes below 130 km is described. The probe consists of a quadrupole mass filter which can be operated in three modes allowing composition analysis of atmospheric positive ions, negative ions, and neutral gases. The pressure in the spectrometer is kept low by a liquid helium-cooled cryopump. An electrostatic probe for the measurement of total positive ion densities and an ionization gauge for the measurement of the total gas density and thereby also the gas temperature are also integrated in the instrument. Some results obtained by two flights of the instrument during the Western Europe Winter Anomaly Campaign 1975/76 are presented. The measurements indicate drastic enhancements of plasma densities in the D-region as well as characteristic changes compared to normal conditions of the positive ion composition namely low fractional abundances of cluster ions and high fractional abundances of molecular and atomic ions.

Key words: Mass spectrometer — Ion composition — Neutral composition — D-region — Mesosphere — Middle atmosphere — Winter anomaly.

Introduction

Our current understanding of the physical and chemical processes governing the neutral and ionized gas in the middle atmosphere (<100 km) is still very limited mainly due to the relatively high gas densities which cause complex photochemical and dynamical processes and which complicate in situ measurements. Associated with the complex nature of atmospheric processes is a pronounced variability of the plasma and the neutral trace gases. A striking example for the complexity of these phenomena is the variability of plasma densities in the ionospheric D- and lower E-regions. From ground-based observations it was
found that the mid-latitude $D$-region exhibits anomalously high radio wave absorption during winter (Appleton, 1937). This phenomenon, termed winter anomaly in the $D$-region was attributed to enhanced electron densities at altitudes below 90 km (e.g. Dieminger, 1952). Further observations revealed a strong short-term variability of the absorption characterized by time scales of days (e.g. Lange-Hesse, 1953; Schwentek, 1974). Because of the complexity and temporal as well as spatial variability of the winter anomaly it is important to measure as many as possible relevant atmospheric parameters at the same time and location. These observations have to be complemented by synoptic measurements to study the spatial extent and temporal development of the event. A detailed description of the campaign strategy is given by Offermann (1977). The scientific objective within the campaign of the instrumentation to be discussed in this paper has been to measure composition and structure of the plasma and the neutral atmosphere in order to help identifying the causes for the anomalous enhancement of the plasma densities in the $D$- and lower $E$-region. The instrumentation has evolved from many years of research in the middle atmosphere. It comprises an instrument package deliberately designed for use on small rockets delivering measurements of composition and density of ionized and major neutral species as well as densities of several neutral trace gas and atmospheric temperature.

**Instrumentation**

*Mass Spectrometer: Ion Mode*

A mass spectrometer has to be operated at low pressure in order to avoid collisions of ions with neutral molecules or atoms. Taking a typical ion path length of 10 cm a pressure below $5 \times 10^{-4}$ torr is required. This value corresponds to an altitude of about 120 km in the atmosphere. Consequently, mass spectrometric in situ studies of the atmospheric plasma and neutral gas composition at heights below 120 km require differential pumping. This implies that the mass spectrometer has to be mounted inside a vacuum tank into which ions enter through a small inlet orifice. The neutral gas which also enters the tank has to be pumped by a high speed pumping system. In a first approximation the sensitivity of the instrument is mainly determined by the pumping speed, as this limits the diameter of the inlet orifice and thereby the flux of ambient species into the mass spectrometer. A schematic representation of the instrument is given in Figure 1. The mass analyzer is a quadrupole mass filter $MF$ of small length because then it can be operated at somewhat higher pressures than most other types of mass spectrometers. This is mainly due to the focusing effect of the quadrupole field which makes the mass filter less sensitive to changes in ion injection parameters and to ion scattering. Aside from the $MF$ the vacuum tank $VT$ also contains a liquid helium-cooled cryopump consisting of the helium dewar $HD$ and the cold pumping surfaces $CS$ which both are gold plated. In order to reduce the input of heat radiated from the warm vacuum tank to the dewar, the latter is surrounded by a radiation shield $RS$ which is cooled by cold
helium gas evaporating from the dewar. On top of the vacuum tank a sampling cone $SC$ is mounted bearing the knife-edged orifice $IO$ with 1 mm diameter at its center. The purpose of the $SC$ is to attach the shock wave which forms upstream the instrument when it traverses the atmosphere at supersonic speeds. The ejectable cap $EC$ covering the sampling cone and released at 55 km on the upleg, seals the probe after bake-out and calibration in the laboratory. The inside pressure is maintained below $10^{-6}$ torr by a small ion getter pump $IP$ with a pumping speed of $0.51 \text{s}^{-1}$. On most of the ascent part of the rocket trajectory the flow approaches the instrument under an angle of attack $\alpha$ which is close to zero. By attaching the shock to the edges of the orifice atmospheric constituents can be sampled without being disturbed by flow effects. It should be noted, however, that an attached shock only forms under conditions of laminar flow below 65 km and that a modestly detached shock has to be expected in the transition flow regime between 65 km to 90 km. Here the effect of the sampling cone is to considerably decrease the distance between the shock wave and the orifice. Thus possible disturbances of the sampling efficiency and of the sample's composition are minimized. Downstream the inlet orifice ions, either ambient or formed from neutrals in the ion source, are extracted from the gas beam and
focussed onto the entrance hole of the mass filter by use of a simple ion optical system. It consists of one cylindrical and two planar grid electrodes which are located above and below the planar part of the cold surface. The latter freezes out most of the gas beam. Only neutral particles contained in the central part of the beam reach the interior of the mass analyzer where they are scattered by warm walls and finally pumped by the cylindrical part of the cold surface.

In order to investigate the efficiency of the pumping system, the pressure distribution along the beam axis was measured by use of a miniaturized ionization gauge under stationary conditions in the laboratory. As seen from Figure 2 the pressure decreases steeply from its value upstream the inlet orifice towards the planar cold surface and continues to decrease further downstream to a constant value. When compared with a theoretical profile considering no obstacle downstream the SC which is also shown in Figure 2, the data points agree reasonable well. When the cold surface is kept at room temperature and the $VT$ is pumped by a high speed oil diffusion pump, a region of enhanced pressure forms upstream the planar cold surface due to particle reflections and scattering. At an outside pressure of 0.5 torr the region of enhanced pressure extends over a distance of 2.5 cm upstream the CS. Under conditions of a rocket flight the gas beam is more concentrated towards the axis as a result of the relative velocity between the gas and the instrument which is superimposed on the Maxwell-Boltzmann velocity distribution of the gas particles. Detailed computations for typical flight conditions at 90 km altitude indicate an increase of the axial pressure by about a factor of 3 when compared with the stationary case. This implies that the curve for 0.5 torr roughly corresponds to a flight situation at about 60 km altitude. Here collisions of ions with neutral particles in the beam are only important on the first centimeter downstream the orifice and beyond this point effective extraction and focusing is possible. After passing through the mass filter $MF$ the mass analyzed ion current is detected by a
channel multiplier $CM$ operated in a pulse counting mode. A mass range from 1 amu to 250 amu is swept in about 2s. Mass spectra of positive and negative ions and of neutrals are measured sequentially (see Table 1). For this purpose the electrical potentials applied for extraction, focussing, mass analysis, and ion detection are changed between subsequent mass scans. A summary of characteristic parameters of the instrument is given in Table 1.

The fragmentation of fragile cluster ions found in the $D$-region during sampling is a problem that deserved special attention in the design of the present instrument. Dissociation energies of cluster ions which are held together by weak dipole interaction forces can be as low as a tenth of one eV. Such complexes may become dissociated upon collisions with neutral molecules. As soon as the relative kinetic energy of the collision partners becomes enhanced with respect to the value in the undisturbed atmosphere, a depletion of fragile ions may occur which, of course, is associated with an increase of fragment ions. The kinetic energy may be increased either due to high temperatures associated with the shock wave or by acceleration of cluster ions in electric fields. Since collisions are not very important downstream the inlet orifice collisional dissociation, if at all, only occurs upstream the inlet orifice. If the shock is attached to the $SC$, no shock-induced composition of cluster ions has to be expected. Electric field-induced decomposition of cluster ions may occur upstream the sampling cone which is kept at a bias potential in order to extract ions from the plasma. Previously typical potential of 10 V were applied (c.f. Narcisi, 1970; Aikin et al., 1977). Laboratory studies which we have carried out (Marien and Arnold, 1973), however, revealed severe decomposition of

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H^+(H_2O)_n n = 1, 2, 3, 4 cluster ions when 10 V are used (Fig. 3). Based upon these results, we use bias potentials of 2.5 V which are just sufficient to overcompensate a possible charge-up of the rocket. The benefits of a sampling cone and the use of low sampling potential became obvious when the instrument was flown for the first time (Arnold and Krankowsky, 1974) and a great number of weakly bound atmospheric cluster ion species was observed. The fact that from these measurements reaction cross sections for the formation of such species in the atmosphere could be quantitatively derived (Arnold and Krankowsky, 1977), which were confirmed by laboratory measurements afterwards, can be regarded as a strong argument in favour of the reliability of modern ion composition measurements in the lower ionosphere.

*Mass Spectrometer: Neutral Gas Mode*

For the mass analysis of neutral atmospheric gases an ion source mounted inside the sampling cone is used. Electrons are produced from a heated tungsten-rhenium filament Fi and injected into the region inside the cylindrical grid electrode. Here they are accelerated towards the inner walls of the front part of the sampling cone. The potential field between the upper planar grid electrode and the sampling cone is adjusted such that the electrons reach an energy of about 16 eV at a potential surface located 2 cm downstream the orifice and gain another 2 eV until they hit the sampling cone. As the ion production rate is proportional to the gas density and to the cross section for electron impact ionization, which strongly increases with energy around 16 eV for most gases of interest here, the downstream boundary of the ionization region is sharply defined. This is true because both the gas density and the electron energy decrease steeply with increasing distance downstream from the sampling cone orifice. As the probability for focussing ions into the mass filter strongly decreases with increasing radial distance from the sampling cone, the effective ionization region is a small volume rather closely confined to this axis. Thus it is
possible to keep the fractional abundance of particles which have undergone wall collisions smaller than 30% of the total number of particles populating the effective ionization volume. This arrangement largely avoids the depletion by wall-reactions of reactive gases, such as for example oxygen atoms, relative to unreactive gases. Another problem associated with mass spectrometric measurements of atomic oxygen below 100 km where O/O₂ is small comes from the production of O⁺ ions by dissociative ionization of O₂. As the cross section for the latter process decreases much faster with decreasing electron energy than does the ionization cross section for the other gases of interest, the problem can be tackled by using low electron energies. However, electron energies much lower than about 18 eV are hardly acceptable because of loss of sensitivity. Thus, atomic oxygen measurements are only possible at altitudes above about 80 km. Another limitation comes from the pressure inside the effective ionization volume which should be sufficiently low in order to avoid ion-neutral collisions. This height limit is about 60 km. Characteristic instrument parameters are given in the Table 1. A full duty cycle of the mass spectrometer includes three mass spectra, one for positive ions, one for negative ions, and one for neutrals. Between subsequent spectra the voltages are switched. No electrons are emitted from the filament in the ion modes.

Positive Ion Probe

The ion probe IP (Fig. 4) which measures the relative total positive ion density consists of a cylindrical grid and of an axial cylindrical collector with a diameter of 3 mm which is small compared to the 32 mm diameter of the cylindrical grid. While the grid is on rocket potential, a voltage of −10 V is applied to the ion collector via a fast floating electrometer. The probe is mounted on a small boom which is housed under the ejectable vacuum cap EC of the mass spectrometer. The probe is deployed upon cap ejection. Thus contamination of the probe prior to deployment is excluded. As the axis of the cylindrical grid is parallel to the rocket axis, the gas flow is assumed to be only weakly disturbed by the edges of the probe. Therefore the collector current I_C should be related to the total

![Fig. 4. Flight configuration of mass spectrometer sampling cone, ion probe (right), and ion gauge (left). 1 and 5 shielding grids, 2 ion collector grid, 3 filament, 4 electron collector, 6 ion collector, 7 booms, 8 sampling cone](image-url)
positive ion density $n_+$ by:

$$ I_C = D e_0 F_G $$

where $F_G$ = flux of positive ions through top circular area of the cylindrical grid
$e_0$ = unit charge
$D$ = coefficient describing the fraction of collected ions with respect to incoming ions.

Under molecular flow conditions $D = 1$ and

$$ F_G = \frac{A_{\text{eff}} \bar{V} F(S) n_+}{4} $$

where $A_{\text{eff}}$ = effective top circular sampling area of the cylindrical grid.

$$ \bar{V} = \left( \frac{8 K T}{\pi m} \right)^{\frac{1}{2}} $$

$$ F(S) = \exp(-S^2) + S \pi^{\frac{1}{2}} (1 + \text{erf}(S)) $$

$V_R$ = rocket velocity
$S$ = $V_R \cos \alpha/(2 K T/m)^{\frac{1}{2}}$
$\text{erf}$ = error function
$\alpha$ = angle of attack
$K$ = Boltzmann’s constant
$m$ = ion mass.

In an intermittent mode of operation the potential of the ion collector is varied from +15 to -15 V in 2.5 V steps. Thereby a probe characteristic is obtained which allows to judge whether $D$ is one. The measurements indicate that $D$ becomes smaller than one at altitudes below about 60 km. Here the time required for an ion to drift from the grid to the axial collector becomes larger than the residence time of the ion inside the cylindrical grid which is $\tau_{\text{res}} = L/V_R$ where $L$ is the length of the cylindrical grid and $V_R$ is the rocket velocity. At these altitudes the $IP$ is not considered to provide meaningful data.

**Ionization Gauge**

The ionization gauge $IG$ (Fig. 4) measures the absolute total gas density. It consists of a tungsten-rhenium filament from which electrons are emitted and accelerated to a circular wire electrode at +70 V. Positive ions produced by electron bombardment ionization are collected by a cylindrical grid electrode to which a potential of $-10$ V is applied via a fast floating electrometer. Another cylindrical grid electrode which is at rocket potential is used in order to shield the sampling area of the mass spectrometer from the ion gauge potentials. The geometry of the ionization gauge is designed in such a way that the ambient gas flow reaching the ionizing volume is likely almost undisturbed.
The dimensions of the ion gauge are small so that collisions of ions produced and neutral particles become important only at pressures above about \(10^{-2}\) torr. However, laboratory measurements (Hettmannsperger, 1976; Arnold et al., 1975) indicated that the collected ion current remains almost proportional to the gas density even up to pressures of 0.1 torr. This corresponds to an altitude of about 63 km. The upper height limit for obtaining useful measurements is determined by the density of ambient positive ions which also contribute to total positive ion current measured by the ion gauge. This contribution can be determined when the filament of the ion gauge is switched off. This occurs during the ion modes of the mass spectrometer. Under quiet daytime conditions ionospheric ions contribute \(50\%\) to the total collector current at an altitude of about 100 km. The absolute sensitivity of the ion gauge is calibrated in laboratory. The gauge is mounted on a small boom which before cap ejection is housed under the vacuum tight cap of the mass spectrometer. The boom is deployed upon cap ejection. Thus contamination of the ion gauge is avoided. Absolute number densities of the atmospheric gas are obtained by use of the laboratory calibration. The contribution of ionospheric positive ions to the collector current determined as described above is corrected for up to altitudes where it becomes \(50\%\). Above this height the method is not considered to be applicable anymore. As the sampling of ions produced in the ionisation region becomes severely affected by collisions below about 60 km, the method cannot provide relative data below this altitude.

**Mass Spectrometer Data Analysis**

**Ion Measurements**

Relative partial ion densities are obtained from the measured partial count rates. The count rate \(C(X)\) of an ion species \(X\) is related to the flux through the inlet orifice \(F_0(X)\) by

\[
C(X) = T(X) F_0(X)
\]

where \(T(X)\) is the transmission factor of the ion optics and the mass analyzer. \(T(X)\) is calibrated in the laboratory for various ion species including \(\text{He}^+, \text{CH}_4^+, \text{H}_2\text{O}^+, \text{Ne}^+, \text{N}_2^+, \text{O}_2^+, \text{A}^+, \text{CO}_2^+, \text{Kr}^+,\) and \(\text{Xe}^+\). Under flight conditions \(F_0(X)\) is related to the number density \([X]\) by

\[
F_0(X) = \frac{1}{4} A_{\text{eff}} \bar{V} F(S) [X]
\]

where \(A_{\text{eff}}\) = effective orifice area.

The other quantities are the same as in Equation (2). The relation applies only for molecular flow conditions (>90 km) and assumes that ions are sampled like neutral particles except for an enrichment upstream the orifice due to the application of a draw-in potential at the sampling cone. This leads to an effective orifice area. If we assume \(A_{\text{eff}}\) to be mass independent, it does not enter into the evaluation of relative partial densities. It may also be noted that for a
small $\alpha$ and $V_R \gg \tilde{V}$ which occurs below 90 km on ascent, the expression simplifies to

$$F_0 \approx A_{\text{eff}} V_R [X]. \tag{5}$$

In a first approximation no mass dependance of $F_0$ is expected at these altitudes. Provided all ions can be extracted from the expanding gas beam and focussed into the mass filter, mass discrimination due to flow effects downstream the sampling orifice should not be important. Absolute partial ion densities are obtained by normalizing the sum of the relative partial densities to the total ion density at one altitude. Usually the total ion density is obtained by independent methods such as electrostatic probes, Faraday rotation and ionosonds. The latter two methods give the electron density which above about 70 km should equal to the total positive ion density. At altitudes below about 60 km $F_0(X)$ and $T(X)$ become pressure- and thereby altitude-dependant and the normalization has to be done for each data point.

**Neutral Gas Measurements**

The channeltron count rate $C(X)$ of a species $X$ is related to the rate of production of ions $X^+$ in the effective ionization region $P(X^+)$ by:

$$C(X) = P(X^+) T_N(X) \tag{6}$$

where $T_N(X^+)$ is a transmission factor for ion optics and the mass filter. $P(X^+)$ is determined by the ionization cross section $\sigma(X)$, the flux of ionizing electrons $F_E$ and the density of $X$ which all depend on the location inside the effective ionization volume. In the laboratory $C(X)$ is determined as a function of $[X]$ for stationary conditions ($V_R = 0$) and for various gases including CH$_4$, H$_2$O, N$_2$, O$_2$, A, CO$_2$ and Kr. The mass discrimination due to flow effects occurring under flight conditions ($V_R \neq 0$) is computed as a function of $V_R$ and atmospheric gas temperature.

For atomic oxygen which was not calibrated in the laboratory $P(O^+)$ was determined from measures $P(X^+)$ values by

$$P(O^+) = \frac{\sigma(O^+)}{\sigma(X^+)} P(X^+). \tag{7}$$

The transmission factor $T_N(X)$ which is essentially a function of mass was determined for O from the measured relation $T_N(M_X)$. Absolute partial densities are determined by normalization of the sum of relative partial densities to the absolute total gas density measured by the ionization gauge. Regarding a possible depletion of reactive gases as for example atomic oxygen the computations indicate that less than 30% of the incoming oxygen atoms undergo wall collisions upstream the effective ionization volume. This method of analyzing neutral gas data as well as the experimental approach outlined above has been checked against a different experimental technique for atomic oxygen.
which certainly is the constituent most difficult to measure by mass spectrometer. Atomic oxygen number densities obtained from a mass spectrometer identical to the instrument described here and a resonance fluorescence and absorption experiment (Dickinson, private communication) flown about 30 min after the mass spectrometer showed an excellent agreement of the relative number densities within an altitude region between 83 and 113 km altitude. The average ratio of the resonance fluorescence to the mass spectrometer number densities compared for 26 data points in the height range indicated above was 2.1 with a standard deviation of 0.5.

**Results and Discussions**

Two instruments B4-1 and B4-2 were flown within the campaign (cf. Offermann, 1977) on January 4 and January 21, 1976 at 1431 UT and 1433 UT, respectively. Both days were characterized by high radio wave absorption in the D-region.

The instruments performed as expected and data were obtained on the upleg part of the trajectory from 60 km to apogee at 130 km. On the downleg portion useful measurements were made down to 60 km and 35 km for B4-1 and B4-2, respectively.

The N$_2$ and O$_2$ number densities measured on both days are shown in Figure 5 along with the total gas density measured by the ionization gauge on January 21. When compared with N$_2$ and O$_2$ profiles of the CIRA 1972 reference atmosphere deviations up to about ±50% are observed. Total positive ion densities as measured by the ion probes with relative probe densities normalized to ionosonde values (Rose and Widdel, private communication) at 109 km are shown in Figure 6. When compared with each other both profiles are very similar. They are characterized by pronounced relative maxima around 88 km and around 79 km. When compared with average winter conditions at

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**Fig. 5.** Height profiles of N$_2$ and O$_2$ number densities. Lines represent CIRA 1972 model densities.
37°N the plasma densities are enhanced between about 75 km and 90 km by factors up to 4.

The enhancement is strongest at the heights of the conspicuous structures mentioned above. The fractional abundance of positively charged molecular ions, atomic metal ions and cluster ions as measured by the positive ion mode of the mass spectrometer is shown in Figure 7. Again the data sets of both days reveal a high degree of similarity when compared with each other. Molecular ions dominate above 76 km (B4-1) and 77 km (B4-2) and cluster ions below.
Atomic metal ions reach high fractional abundance up to 40% in the altitude range 85–90 km. This altitude range coincides with the upper structure shown by the total positive ion density profiles (Fig. 6). Two extraordinary features are obvious when compared to a normal midlatitude situation (Narcisi, 1976).

First, a lowering of the height by about 3 to 4 km is observed at which molecular ions and cluster ions become equally abundant. Second, atomic metal ions appear in high relative abundance below 90 km. When compared with the two previous positive ion composition measurements carried out under winter anomaly conditions (Zbinden et al., 1975; Arnold and Krankowsky, 1977) the present results are rather similar to those reported by Zbinden et al., 1975. But they are much different from those obtained at high middle latitudes (Arnold and Krankowsky, 1977). The preliminary results presented here indicate at least three causes of the winter anomalous plasma density enhancements: (1) an increase in the production of molecular ions; (2) a decrease of the bulk recombination coefficient for positive ions and electrons due to low fractional abundances of rapidly recombining cluster ions above 76 km and 77 km, respectively; (3) a decrease of the bulk recombination coefficient for positive ions and electrons due to high fractional abundances of very slowly recombining atomic metal ions around 85–90 km.

Summary

A mass spectrometer probe experiment for use on small rockets has been developed for studying the plasma and neutral gas in the middle atmosphere. The mass spectrometric measurements are supplemented by an electrostatic ion probe and an ion gauge which both are integral parts of the mass spectrometer. Thus mass spectrometric composition data can be converted to absolute number densities of ions and neutral gas species. In addition, from the data of the ion gauge neutral atmospheric temperatures are derived.

When compared to previous instruments utilized below 100 km the, mass spectrometer sampling technique has been vastly improved. Shock effects and disturbances of the flow field are largely avoided. The absence of shock effects and the use of low draw-in potentials enables the measurement of fragile cluster ions with low bond energies. Reactive neutral gases as e.g. atomic oxygen can be measured without severe depletion due to an appropriate design of the ion source. Therefore, the measured ion and neutral gas composition closely resembles the atmospheric composition.

The mass spectrometer probe delivers ion and neutral gas number density profiles independent from additional observations. To some extent ion and neutral gas data complement each other and allow a check of the internal consistency. Neutral gas temperature derived from the ion gauge data can be compared to the temperature obtained from the ion composition considering the temperature dependent ion-neutral reaction rate coefficients. Number densities of certain neutral gases like NO, H$_2$O, H$_2$O$_2$, and O can indirectly be determined from the ion composition and complement the direct neutral measurements.
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