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POSITIVE ION COMPOSITION FROM A ROCKET-BORNE MASS SPECTROMETER *

R. A. GOLDBERG

L. J. BLUMLE

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CONTENTS

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by

R. A. Goldberg
L. J. Blumle*
Laboratory for Space Sciences
NASA, Goddard Space Flight Center
Greenbelt, Maryland

ABSTRACT

On March 15, 1968, at 14:11 LMT, a Nike Tomahawk was launched from Wallops Island, Virginia, carrying a quadrupole mass spectrometer for measurement of positive ion composition in the ionosphere. The instrument was enclosed in a vacuum system principally controlled by a titanium getter pump, and marks the first attempt to use this technique for rocket experiments. A CW radio propagation experiment plus ground based ionosonde results enabled the reduction of the data to absolute values. On upleg, the spectrometer was opened at 98 km and provided well resolved spectra in the ram direction up to an apogee of 303 km, slightly above the F2 peak. The sweep range from 13 to 49 AMU enabled all major constituents to be determined and the high sensitivity of the spectrometer allowed many minor constituents, having densities as low as 2 to 5/cm^3. to be detected. On downleg, measurements were restricted to wake composition but values obtained at higher altitudes were similar to those seen on upleg. Upon entry into the D region, the vacuum system permitted spectra to be obtained to 68 km. D region results between 80 and 68 km show a predominance of 19$^+$ and 37$^+$ ions, possibly due to complex water clusters, marking the first independent observation in agreement with the earlier results of others.

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*Now at Biomedical Systems Dept., Hamilton Standard, Farmington, Conn.

INTRODUCTION

The quadrupole mass spectrometer is an instrument which has received growing attention in recent years as a device for ionospheric ion and neutral composition measurements in the upper atmosphere. The light, compact size of the quadrupole mass spectrometer accompanied by its insensitivity to ion velocity are two major features which make this instrument well suited for rocket and satellite applications.

The primary objective of the work reported herein has been to develop a technique for measuring ion composition in the D region of the ionosphere. Below 90 km, the ambient pressure of the atmosphere is sufficiently large to cause a mean free path of length less than the dimensions of the instrument. Since proper operation of the spectrometer can only be achieved in regions where the mean free path exceeds the dimensions of the instrument, this requires that the instrument be enclosed in a low pressure region less than 10^{-3} torr.

The vacuum system used to maintain this pressure must be able to override the tremendous influx of atmospheric particles permeating the system through the entrance orfice of the spectrometer system, especially at low altitudes in the ram direction of a fast moving rocket. This capability has been achieved with development of a vacuum system combining a titanum getter pump with a triode ion pump.

Finally, the above system has been tested aboard the flight of a Nike-Tomahawk rocket, NASA 18.10. Results of this flight are shown and discussed.

EXPERIMENTAL DESCRIPTION OF NASA 18.10

As previously mentioned, NASA 18.10 designates the Nike Tomahawk sounding rocket launched at 14:11:00 LMT on March 15, 1968 from Wallops Island, Virginia. The payload reached an apogee of 303 km and provided some 410 seconds of data. The payload included a quadrupole mass spectrometer contained in an enclosure evacuated by means of a titanium getter pump plus a triode ion pump. Other experiments aboard included a CW radio propagation measurement for determination of local ambient electron density plus a two axis magnetometer and a solar aspect sensor for rocket attitude determination. To minimize the effects of contamination, payload separation from the rocket occured prior to exposure of the spectrometer experiment to the ambient atmosphere.

A. The Quadrupole Mass Spectrometer Experiment

Specifications for the quadrupole mass spectrometer used in Flight 18.10 are listed below:

Rod Length	3.0 in.
Entrance Aperature Diameter	0.030 in.
Mass Sweep Range, Spectrometer Mode	13-49 AMU
Duration of Mass Sweep	1.24 sec.,
Mass Range, Highpass Filter Mode	>38 AMU
R.F. Frequency	2.120 MHz

The entrance aperature and rods were biased to -10^{v} in order to attract and accelerate positive ions to sufficient energy to counteract the effects of the fringing field encountered near the entrance to the spectrometer. The output current from the spectrometer was amplified approximately 10^5 times with an ITT #FW 141 electron multiplier. The final current output was measured by means of a logarithmic electrometer capable of detecting currents from 2×10^{-12} to 5×10^{-5} amps with a response time of less than 20 milliseconds over the lowest decade.

An example of the raw data obtained during flight is illustrated in Figure 1 where heights refer to the mean value during each sweep. Such spectra were taken every 1.27 sec. during the flight beginning at 99 km on upleg. The spectra shown were obtained at cap opening (99 km), 159 km, and apogee (303 km). Since no doubly ionized constituents should occur in the region of data aquisition, all peaks are identified with singly ionized masses. It should be remembered that the high peaks illustrated are several decades greater in magnitude than the minor peaks because of the logarithmic response of the electrometer. It is apparent that large peaks tend to mask adjacent minor peaks, but a mass separation of two or more is always discernible. In the illustrated examples, mass 14 is partially masked by 16 and mass 28 by 30. In cases such as these, the change of slope marking 14 and 28 is adequate to evaluate the magnitudes of currents generated by such ions; at least within the error limits introduced when attempting to read heights of peaks presented on a logarithmic scale. For adjacent minor constituents, no problem occurs in discerning peaks having mass separations of $\Delta m \geq \frac{1}{2}$ to 1 AMU.

At the right end of each spectra is a flat spectral peak representing the sum current of all ions having mass > 38 AMU. This is obtained by operating the spectrometer in the high pass filter mode. It provides information about the existence of heavy ions in the medium, although the actual mass values of such heavy ions is not supplied.

Finally, the voltage sweep applied t the spectrometer rods is also telemetered and the high linearty of this sweep (as illustrated in Figure 1) enables rapid reduction of the resulting data.

B. The Vacuum System

The quadrupole mass spectrometer was flown housed in a chamber evacuated by a titanium getter pump plus a triode ion pump (General Electric Model #22TP050) rated at 0.5 liters/sec. for air. The ion pump was added to pump inert gases such as argon, since the getter pump cannot effectively remove these gases.

The getter pump system has been developed because of its high pumping capacity and its relative simplicity of operation. Because of the relatively short duty cycle available for the getter pump (order of seconds in the lowest part of the D region) it is essential to prepare the getter pump in the following manner.

Prior to launch, the quadrupole is assembled within its housing and is maintained between 10^{-6} - 10^{-7} torr by a ground based vacuum system. This system uses titanium sublimation and ion pumping. At one to four hours prior to launch, the payload titanium filaments (see Figure 2) are activated by a large current and vaporize deposit a fresh coating of titanium on the inner walls of the evacuated housing chamber. The spectrometer and other critical components are protected from vapor deposition by a series of baffles. The freshly deposited titanium is retained in an active pumping state because of the low pressure ($<10^{-6}$ torr) present during the vapor deposition process.

In flight, this inoperative mode is maintained until the desired altitude for ionospheric measurement is reached. At such time, the front aperture to the housing and spectrometer is opened by removal of a cover plate, and the pump begins to operate on the incoming neutral gas and ion flow.

A laboratory experiment was designed to test the pumping capability within the flight housing for an aperture opened in the D region. In this experiment, a titanium coated chamber was exposed to a controlled external pressure at a specific time and pressure rise as a function of time noted. To compensate for the effect of ram pressure in an actual flight, the chamber was exposed to external pressures much larger than the expected ambient pressure. The results suggested that the pump should be able to maintain pressures needed to maintain a mean free path longer than the dimensions of the instrument, 10^{-3} torr, during an upleg flight commencing at 75 km.

It is impossible to appraise the uniformity of the coating applied in the above manner. Furthermore, gradual losses of effective titanium pumping area due to residual outgassing and leaking are difficult to estimate. As a result of these and other considerations, cap opening on NASA 18.10 did not occur until 98 km on upleg, above those heights where pumping is absolutely necessary. Nevertheless, on downleg, the pump proved to operate effectively, i.e. spectra were obtained down to 68 km. It must be noted that the downleg measurements were of wake exposure and do not indicate the effects of ram on the vacuum pumping ability. This effect will be analyzed on future rockets by opening the system at lower altitudes on upleg.

C. Support Experiments

In addition to the quadrupole mass spectrometer, other experiments were included to determine local electron density and rocket attitude.

The electron density experiment was of the continuous wave dispersive Doppler type introduced by Seddon (1953) and described in greater detail by Bauer and Jackson (1962). The frequencies transmitted for analysis were 73.6 and 24.53 MHz.

The rocket attitude was determined by two Heliflux magnetic aspect sensors (Schoensted Type Ram-5C) and a solar aspect sensor.

RESULTS OF NASA 18.10

A. Trajectory and Attitude

NASA 18.10 was fired at a launch elevation angle of 80°; the azimuth was 144° measured from North. Apogee occurred at approximately 303 km enabling data to be obtained from lower E region (98 km) through the F region maximum (285 km) on upleg, and down to the lower D region (68 km) on downleg. The horizontal range of the flight was less than 185 km allowing up and downleg passes to occur within close proximity of one another.

At the time and date of firing, solar zenith angle was determined to be 48.8^{o}, solar azimuth was 42.3^{o} measured from South. Calculations for rocket attitude were made at 51 seconds (83 km) after lift-off. This time (altitude) was chosen because a study of the magnetic and solar aspect data showed no further change in attitude throughout that part of the flight during which data were obtained until the rocket had dropped below 69 km on downleg. Magnetic parameters used at 83 km for the calculation of the rocket-magnetic field angle were obtained by means of a computer program incorporating the 1965 magnetic field, time dependent coefficients (Cain et al, 1965) adjusted for the date of firing.

The results of attitude analysis demonstrate that after the ejection of the nose cone plus the separation of the payload from the second stage, the rocket attitude stabilized to an elevation angle of 58^{o} and an azimuth of 145^{o}, only 1^{o} out of the plane of the trajectory. This was quite fortunate since it enabled the aperture of the spectrometer to practically face the forward ram direction during most of the upleg flight.

The attitude data also indicate a small coning effect with an angle of approximately ±2.5° and a period of 4 seconds. The attitude discussed above represents the center position of the rocket within this cone.

B. Electron Density Analysis

The raw data obtained and illustrated in Figure 1 were reduced to absolute values of ion composition by normalization to an electron density profile obtained during the flight. The electron density data came from two sources, a ground based ionosonde and a rocket based CW propagation experiment. Unfortunately, the CW experiment malfunctioned above 95 km on upleg, preventing absolute determination of the electron density from 95 to 150 km.

The combined profile from both experiments is illustrated in Figure 3 on the curve marked N_e. For the sake of comparision, the identical curve is duplicated on both up and downleg. The analysis of the ionogram does not permit unique determination of electron density in the height region 110 to 150 km. The approach used in the true height reduction is that of Jackson (1956), whereby valleys of different depths are assumed and applied to the reduction of the ordinary propagation mode trace on an ionogram. The various profiles obtained are reconverted into extraordinary propagation mode traces, and selection of the correct profile is based on the best fit with the actual extraordinary trace. The approach is somewhat error prone, but nevertheless indicated an assumed valley of .25 MHz as the most probable best fit. Below 110 km, the profile is considerably more reliable because of the ionogram fit to the CW propagation data. Below 85 km, the electron density curve was linearly extrapolated. More recent results with Gerdien condenser and Faraday rotation techniques tend to justify such an extrapolation, at least down to 70-75 km. The presentation of absolute ion composition below 75 km may be somewhat in error because of the above assumption.

The ionogram provided data only up to the F2 peak (285 km). The profile was extrapolated to 303 km by assuming the topside to be symmetric to the bottomside within ±20 km of the F2 peak.

C. Reduction of Spectrometer Data

Assuming charge neutrality over the entire range of flight, the raw data, as sampled in Figure 1, has been normalized to the electron density at the height of each spectral sweep. The up and downleg electron density profiles have been assumed identical in this analysis since ionsonde data indicated no apparent change during flight. In applying the normalization, mass weighting corrections attributable to free molecular flow theory (Tsien, 1946) have been applied to account for modifications due to variable rocket attitude and velocity above 100 km. Below this height, the theory breaks down because of increasing collision frequencies. The $v_m F(s)$ function, as this correction factor is often termed, relates to neutral pressure effects and is justified in this instance of positive ion analysis because of the improved agreement it provides between up and downleg results during the rocket flight. Typical shifts in mass magnitudes relative to normalized values without the $v_m F(s)$ correction are of order 20% or less on upleg. The effect tends toward larger values on downleg because the entrance aperture moves away from incoming particles in this region. In the realm below 250 km on downleg this effect becomes extremely large and can modify the density of certain constituents by factors of order 10 or more.

The quantity "s" is the ratio of the rocket velocity component along the spectrometer axis to the mean thermal velocity (v_m) of the ion species in question. As a result, $v_m F(s)$ is a function of the angle of attack determined from trajectory and attitude data, and the temperature and mass of the ion species in question. The temperature model used for this correction is that obtained from the 1965 CIRA Tables. On each spectral sweep, the current for each species is weighted by the v_m F(s) function before normalizing the total of the currents to the electron density at that height. This mass weighting technique obviously causes little shifting of those masses centered near the mean of all masses on the sweep, but enhances heavier mass contributions while reducing the relative contribution of lighter masses.

The spectrometer operation in the high pass filter mode (masses > 38 AMU), illustrated by the flat peak at the right side of each spectral sweep in Figure 1, occurs with one to two decades more sensitivity than operation in the mass filter mode. Unfortunately, it was difficult to determine the exact amount of increased sensitivity in the high pass filter mode during the flight of NASA 18.10, and therefore difficult to ascertain the contribution of ions with mass greater than 50 AMU (the upper limit of the filter mode) to the current sum. However, the heavy ion total peak over most of the flight, even if taken at equivalent sensitivity to the individual mass peaks, never exceeds 5 to 10% of the sum of all individual currents up to mass 50 except in the downleg D region, and thereby can be neglected when determining the normalization constants at each spectral sweep altitude. Although the high pass filter mode has not contributed to the normalization process, it does show a qualitative behavior which will be discussed in a later section.

Figure 3 shows the up and downleg data from NASA 18.10 for all major constituents and some minor constituents when corrected and normalized to the accompanying electron density profile. This data has been somewhat smoothed to eliminate a certain amount of structuring, probably introduced by experimental and reading errors in the data. In the downleg portion of Figure 3, the mass 32^+ curve is illustrated by a lighter weight line between two x's to indicate marginally readable data points in that region.

DISCUSSION OF RESULTS

A. General Properties and Probable Contaminants

The data in Figure 3 is plotted with the abscissa linear in height and the ordinate logarithmic in density. The markings on the scale above the figure indicate time after lift off. Constituents plus their probable identification are listed in Table 1.

The apogee line marks the separation between up and downleg data. Most constituents for which there are both up and downleg data (14^+, 16^+, 28^+, 30^+, 32^+) show marked symmetry about apogee, in spite of the fact that downleg results are obtained in wake conditions where depletion of particles results in ion currents as much as 10^5 times smaller than those obtained in upleg ram conditions. The normalization factor to convert current into electron density was obtained by dividing the electron density measured at the height of a given spectral sweep by the sum of the individual mass currents measured during the sweep and mass weighted by $v_m F(s)$. This weighting procedure assumes that the normalization factor does not depend on any other strongly mass dependent processes, an assumption which must be established by means of further laboratory studies but which appears reasonably sound from the agreement it provides between up and downleg results.

The extreme mass sensitivity provided by the weighting function during the downleg portion of the flight between 250 and 120 km bears some discussion at this time. During the upleg portion of a flight, the entrance aperture lies in a ram configuration such that ions are scooped into the aperture at velocities well in excess of their mean thermal velocities. Analysis of the weighting function shows that as the angle of attack approaches 90° near apogee, the maximum mass dependence ($\sim m^{-\frac{1}{2}}$) for ram configuration occurs. Once the angle of attack exceeds 90° (which occured on 18.10 at 300 km on downleg) the payload aperture begins to depart from ions to be sampled at an ever increasing rate which soon exceeds the mean thermal velocity of such ions. This implies that only those ions lying in the high energy tail of the Boltzmann distribution and having velocities greater than the payload velocity will be able to reach the aperture for sampling. A study of the weighting function for these conditions indicates a large and ever increasing mass dependence in the normalization weighting factor as sampling tends toward ions of higher velocity.

The fact that free molecular flow theory alone brings upleg and downleg data into good agreement indicates that the attractive electric field applied at the aperture in this experiment did not have sufficient power to override this effect under wake conditions above 120 km. This implies that the electric field did not have particularly deep penetration into and beyond the wake.

It is evident that the parallel curves for 18^+ and 19^+ do not exhibit the symmetry about apogee found for other constituents shown. Instead, these constituents appear to follow the time dependent curve, as is further evidenced by the increased slope of both curves near apogee where the time scale becomes exceedingly contracted. The origin of these constituents can thereby be attributed to contamination, with 18^+ and 19^+ possibly being produced by charge exchange processes above 160 km. It is also interesting to note that both 18^+ and 19^+ disappear between 180 and 150 km on downleg indicating that they are wake dependent and thereby originate from a source external to the spectrometer aperture.

Since 18^+ and 19^+ are contaminants, they should not be included in the normalization procedures previously discussed. Both of these constituents are sufficiently minor to prevent any shifting of the curves illustrated in Figure 3 by their neglect, at least within the experimental limits of error introduced by other causes.

Other minor constituents, mainly between 40 and 50 AMU, were seen on 18.10 but ignored, primarily because they exhibited contamination behavior similar to that discussed for 18^+ and 19^+.

B. E and F Region Results

A study of Figure 3 shows that 32^+ and 30^+ remain major constituents until approximately 155 km, where 16^+ crosses over and becomes dominant. The ratio $[30^+]/[32^+]$ remains approximately 2 to 1 over most of the flight until regions below 150 km on downleg, where inaccuracies due to sensitivity loss can probably account for deviations from this value. There is a tendency for this ratio to approach 1 at heights just below 100 km on both up and downleg, but downleg data indicate that 30^+ becomes dominant again below this region. This result differs from some earlier midday results (Holmes et al., 1965, Johnson, 1966, Narcisi, 1968) which show regions where $[30^+]/[32^+]$ becomes less than one but agrees with others not exhibiting this behavior (Taylor and Brinton, 1961, Meadows-Reed and Smith, 1964, Arnold et al, 1969). The other molecular constituent, 28^+, remains minor and nearly constant throughout the entire flight. A small peak for 28^+ between 110 and 120 km may be attributable to enhancement by the silicon metallic ion (Istomin, 1963, Narcisi, 1968). All three molecular constituents appear to peak between 120 and 160 km before dropping off at a linear logarithmic rate with altitude, [redacted]. The other major atomic constituent, 14^+, becomes second most abundant above 200 km but easily remains less than 1% of the total. Finally the symmetry of 14^+ about apogee seems to be less pronounced than for other constituents, indicating a possible difference between up and downleg concentrations for this constituent.

The minor constituents 23^+ and 24^+ are seen to be dropping from a peak near the height of cap opening (98 km). These constituents are identified as sodium and magnesium ions and may represent the metallic belt reported by others earlier (Istomin, 1963, Narcisi and Bailey, 1965, Narcisi, 1968). Of interest is the result that these ions remain in trace quantities of 2 to 10 cm^{-3} up to altitudes in excess of 200 km. Unfortunately, depletion of ions within the wake prevented detection of these ions on downleg.

The curve 34^+ is shown and identified as an 0_2^+ isotope. The expected abundance of this constituent is $[34^+]/[32^+]=1/499$. The measured 34^+ constituent is found to be parallel to the 32^+ curve and of order 1 to 500 over the entire height range in which it is detectable.

The total measured in the high pass filter mode, representing the sum of all masses > 38 AMU, is not shown because of sensitivity arguments previously stated. In addition, it was found to exhibit similar behavior to that of 18^+ in the region above 130 km, indicating that it was primarily composed of contaminants in this region. This does not follow in the D region, as will be discussed in the next section.

C. D Region Results

As previously stated, precautionary action on upleg prevented the acquisition of D region data in the ram configuration. However, comparisons between up and downleg data already discussed indicate a depletion of ions on a relatively mass independent basis in the wake, thereby justifying the presentation of data obtained for D region in the downleg portion of the flight.

Between 100 and 85 km, 30^+ and 32^+ are the only observable constituents, with 30^+ being dominant in the entire range. Below 85 km, 37^+ and 19^+ make their appearance in concurrence with earlier results (Narcisi and Bailey, 1965, and Narcisi, 1967). Figure 4 illustrates the raw data spectral sweeps obtained in this altitude region. The ratio $[37^+]/[19^+]$ is seen to be greater than one above 76 km and less than one below 76 km. Below 68 km, no further mass peaks were obtained.

Narcisi and Bailey (1965) first tentatively identified 19^+ and 37^+ as complex water clusters. Recently, Fehsenfeld and Ferguson (1969) have provided a theoretical analysis to justify this description. Figure 4 also illustrates the total contribution of all particles > 38 AMU, as obtained in the more sensitive high pass filter mode. Heavier water clusters such as 55^+ ($H_7O_3{}^+$) and 73^+ ($H_9O_4{}^+$) may possibly contribute to the larger values of this quantity observed between 85 and 76 km. Some leakage of 37^+ particles into this mode may also occur, and is suggested by the complimentary behavior of the two peaks.

The results noted above do not completely fall within the

Fehsenfeld and Ferguson scheme. The domination of 19^+ below 76 km and the possible presence of heavier water clusters are not expected in this theory. However, the existence of a possible contamination source from an external water cloud accompanying the payload, as evidenced by E and F region results, plus the wake sampling used to obtain these results might account for some of the discrepancies.

SUMMARY AND CONCLUSIONS

The results discussed in this paper have demonstrated the feasibility of housing a quardupole mass spectrometer in a titanium getter, ion pumped vacuum system for measurement of D region positive ion composition. The D region results, although obtained under downleg wake conditions, show a predominence of 19^+ and 37^+ below 85 km, in concurrence with the earlier results of Narcisi and Bailey (1965) and Narcisi (1967). The origin of these water clusters is still open to question. A recent theory (Fehsenfeld and Ferguson, 1969) supports the concept of natural origin in the local ambient ionosphere, but data presented here clearly indicate that 18^+ and 19^+ originate from an external "vapor cloud" accompanying the payload at higher altitudes, and this contamination cloud may provide a partial source for water clusters seen at D region levels.

All major ionic constituents expected at E and F region levels from 100 to 303 km have been observed. In addition metallic constituents (23^+, 24^+, 28^+) have been identified between 100 and 200 km. The sensitivity of the spectrometer system flown has enabled concentrations as low as 2 to 5 cm^{-3} to be detected, and illustrate a nearly constant low density profile of 23^+ and 24^+ to altitudes slightly exceeding 200 km. The minor constituent 34^+ has also been identified as an isotope of 32^+ by exhibiting the proper abundance relative to 32^+ in the region where it is detectable.

ACKNOWLEDGEMENT

Design, development and preparation of the flight package including the quadrupole mass spectrometer and the novel vacuum system were made possible through the efforts of Giles Spaid, Donald Silbert, Dennis Endres,and Robert Farmer. In addition, we thank John Jackson and Giles Spaid for assistance in the reduction of the electron density data and George Kowalski for development and interpretation of the solar aspect sensor. Finally, we thank Jackie Hard and Lloyd Purves for developing the computer routine to reduce the raw spectrometry data into absolute form.

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TABLE 1

PROBABLE IDENTIFICATION OF OBSERVED CONSTITUENTS

Mass to Charge Ratio	14^+	16^+	18^+	19^+	23^+	24^+	28^+	30^+	32^+	34^+	37^+
Probable Identification	N^+	O^+	H_2O^+	H_3O^+	Na^+	M_g^+	N_2^+, S_i^+	NO^+	O_2^+	O_2^+ (Isotope)	$H_5O_2^+$

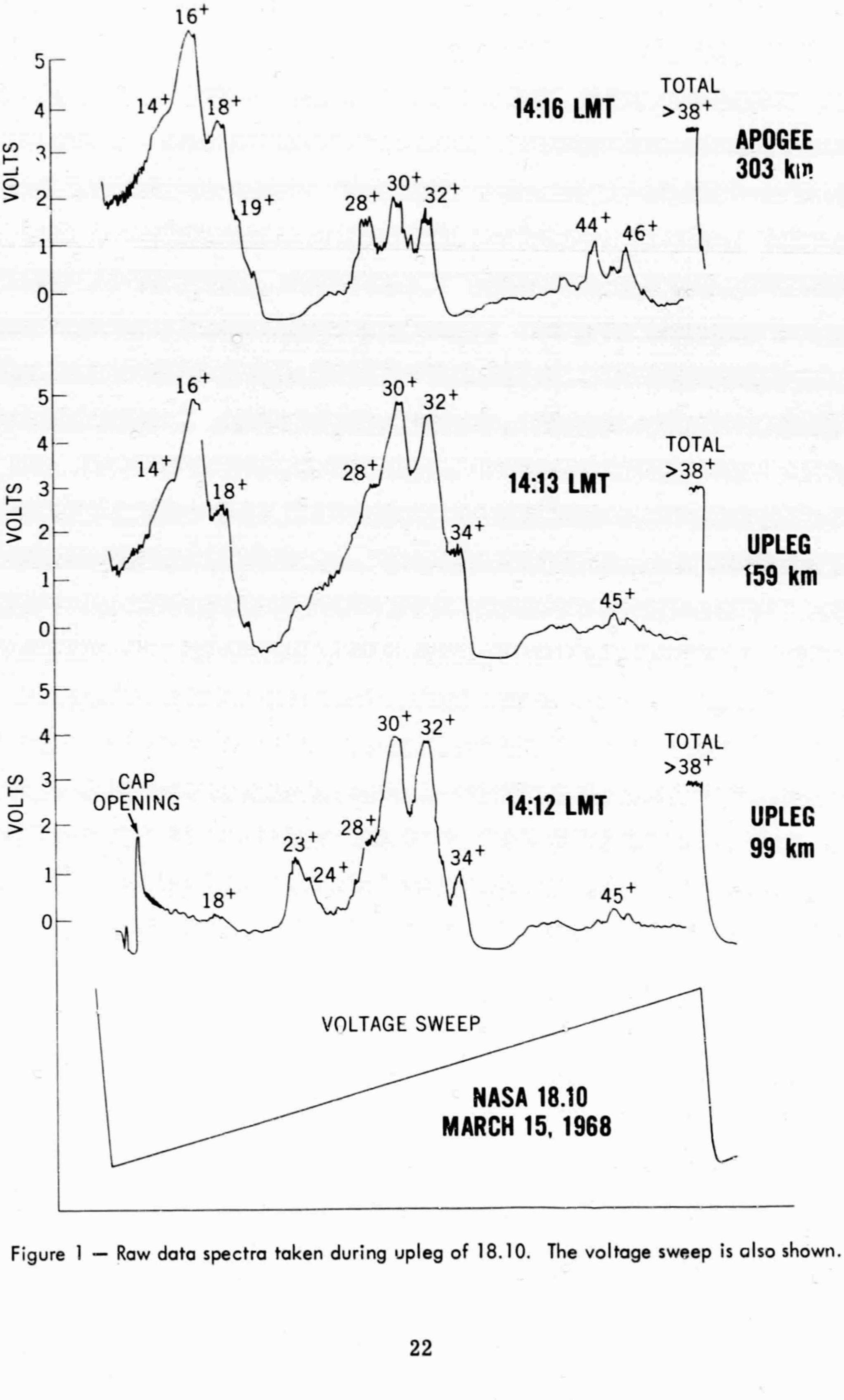

Figure 1 — Raw data spectra taken during upleg of 18.10. The voltage sweep is also shown.

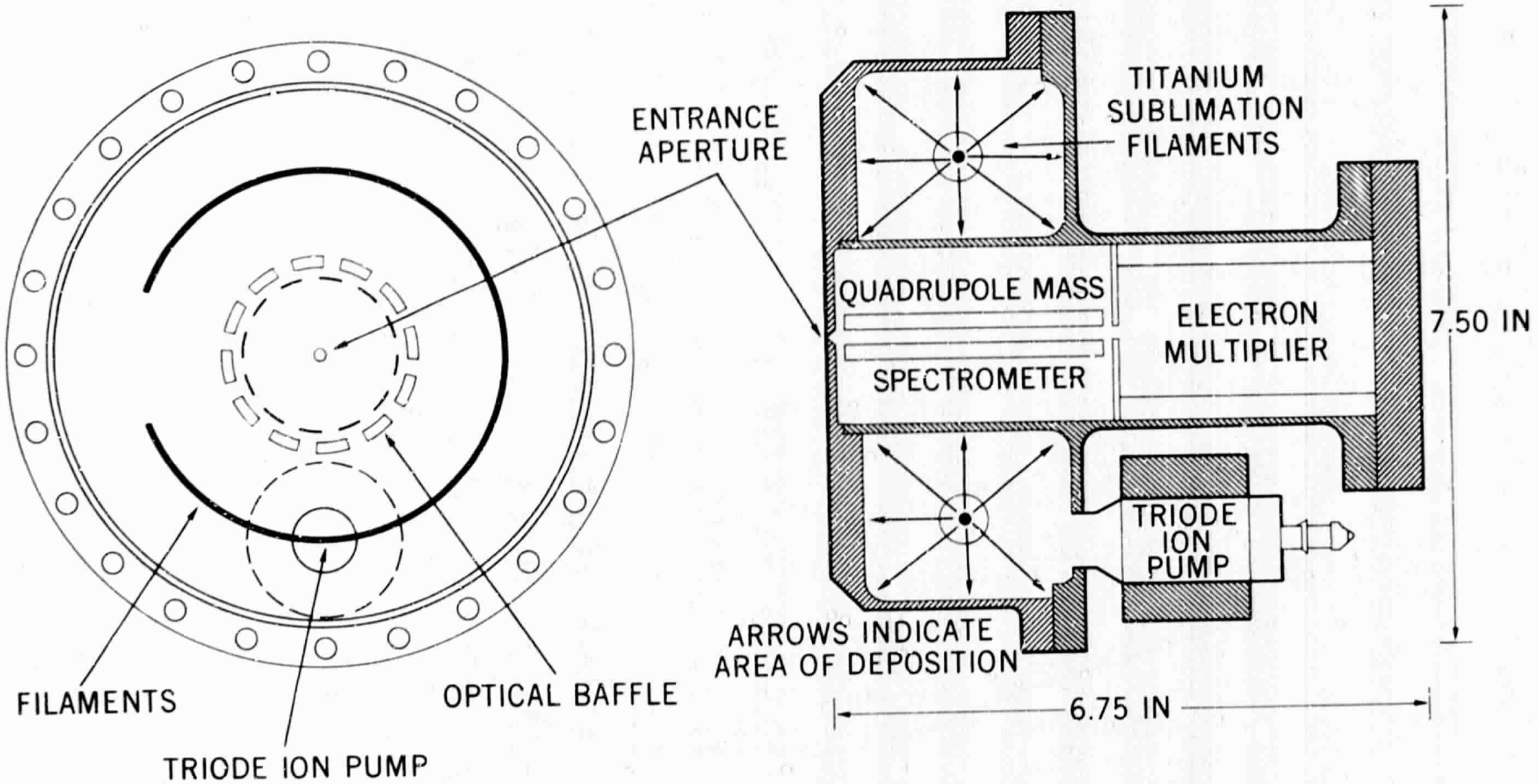

Figure 2 — Schematic of the basic spectrometer plus the vacuum housing and pumps.

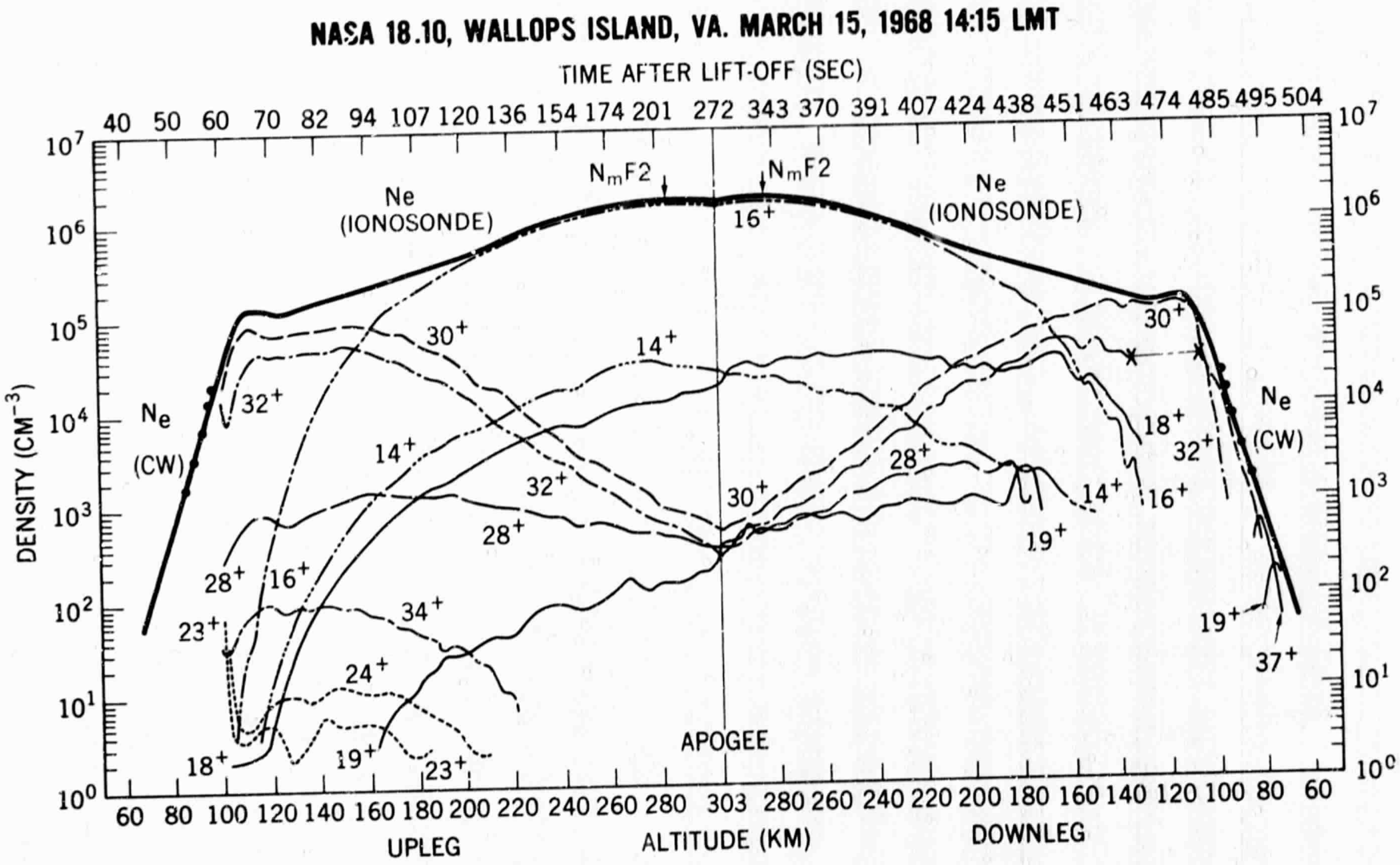

Figure 3 — Positive ion composition measured by NASA 18.10. Up and downleg results are shown as a function of altitude and time. The electron density profile used for normalization is also illustrated.

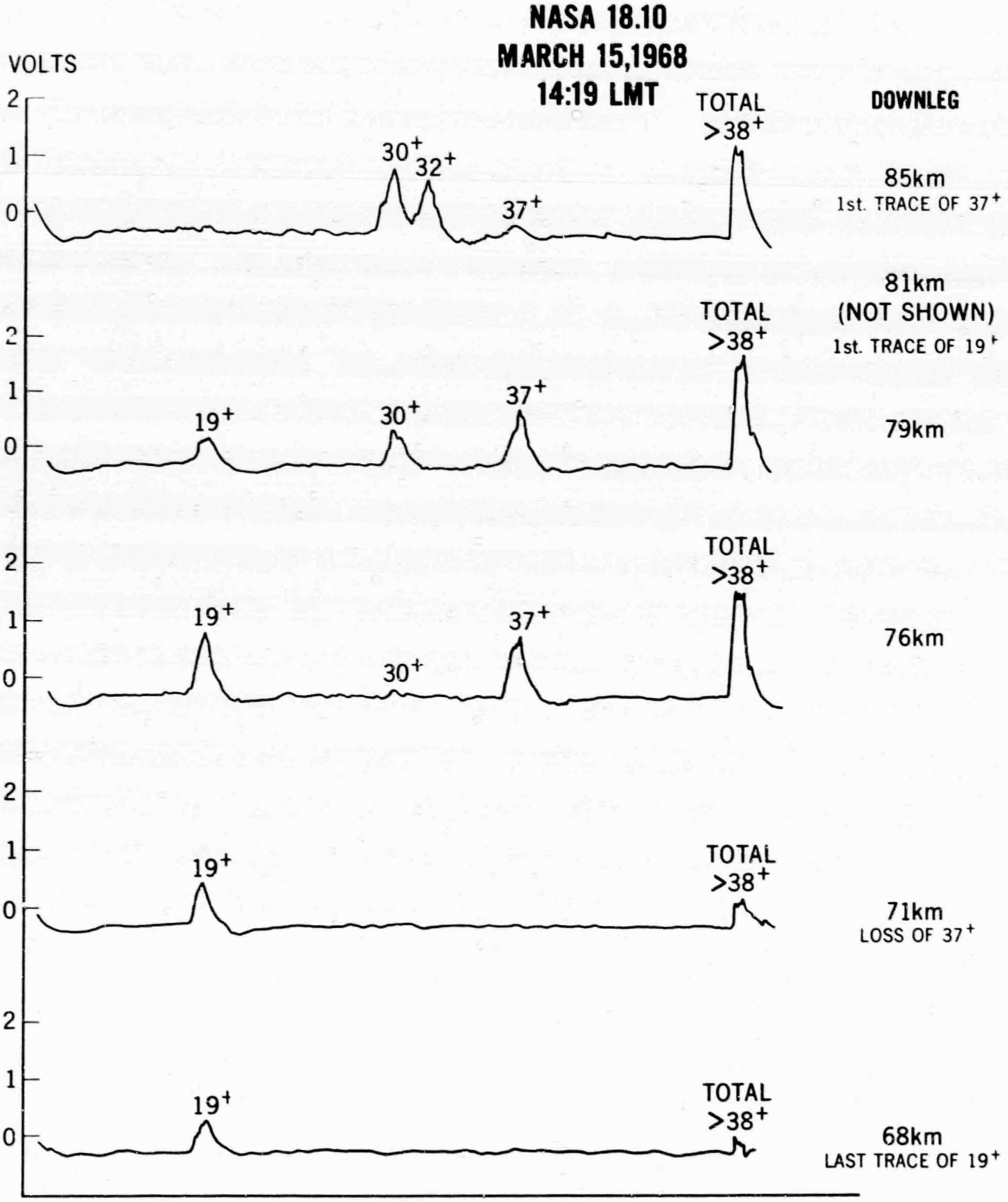

Figure 4 — Raw data spectra taken during downleg D region penetration.