Plasma Analysis
Application Note 256

EQP

Investigation of Plasmas at Atmospheric Pressures

Summary

A Hiden HPR-60 inlet system with an EQP 1000 series mass analyser gauge was used for the analysis of atmospheric plasmas. This particular sampling inlet was specifically designed to penetrate into the plasma chamber and allow sensitive measurement of the plasma at up to atmospheric pressure. To lower the pressure from atmospheric to gauge compatible pressures, a system of three cones with small orifices was used to divide the inlet into three stages. Each stage was separately pumped. MASsoft was used to gather data about neutral, positive and negative species through the modes available through the MASsoft controlling software.

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Introduction

The Hiden HPR-60 inlet system is a molecular beam sampling mass spectrometer. This is designed for accurate process sampling for applications such as plasma etch and deposition, chemical vapour deposition, atmospheric or cluster research, combustion and nanoparticle studies and flow reactors. In many applications the process pressure must be reduced to be compatible to the operating pressure of the quadrupole mass spectrometer. This can be achieved by a design, such as in Figure 1.

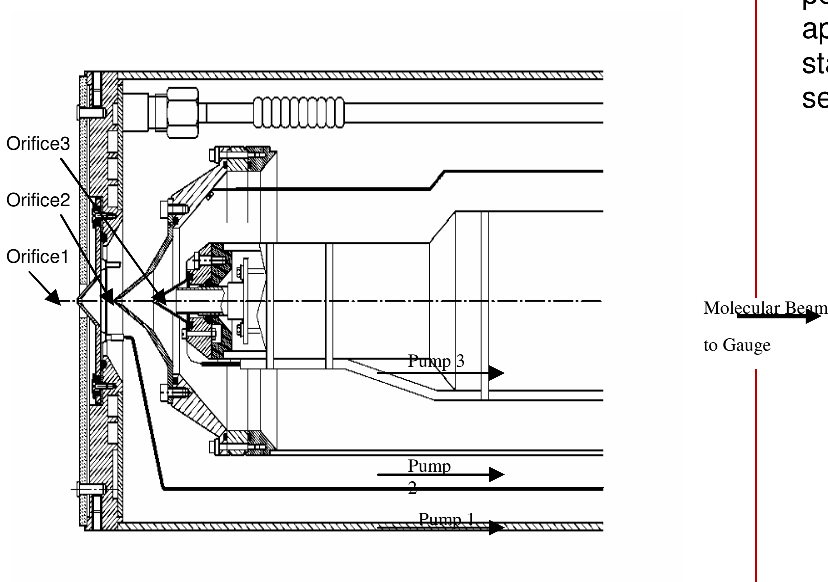

Figure 1: Schematic of inlet sampling system with orifice sizes and pumping locations.

The sampling part consists of several chambers separated by orifices. Each chamber is pumped separately to obtain a sequential pressure drop from the sampling area through the chambers to the gauge. If a suitable combination of orifice sizes and pumping rates are used, sampling can be performed at atmospheric pressures without exposing the gauge to overpressure. The pressure drop helps the sample remain as a coherent beam, reducing collisions between sample and background gases and eliminating interactions with surfaces, increasing accuracy significantly. The orifices are interchangeable and will affect the beam focus and modulation. The design will maximise the pumping efficiency to obtain the optimum beam into the mass spectrometer gauge.

In this particular application the mass spectrometer selected for this inlet system was the Hiden EQP-1000. This is a combined 45° sector field energy analyser and mass spectrometer with 9mm quadrupole rods. The mass range options can be selected from 300, 510, 1000, 2500 amu, providing the best performance to the relevant research application. The triple filter is fitted as standard, increasing accuracy, sensitivity and mass discrimination.

Gas Analysis

Argon gas was used to test the residual gas analysis (RGA) mode of the software. As the gas passes through the three stages, it cools down rapidly and dimerises. Therefore the signal analysed is the Ar_2 80 amu peak, which can be seen in Figure 2. Even for this low concentration species, it can be clearly seen that the count rate was consistently over 50,000 counts per second.

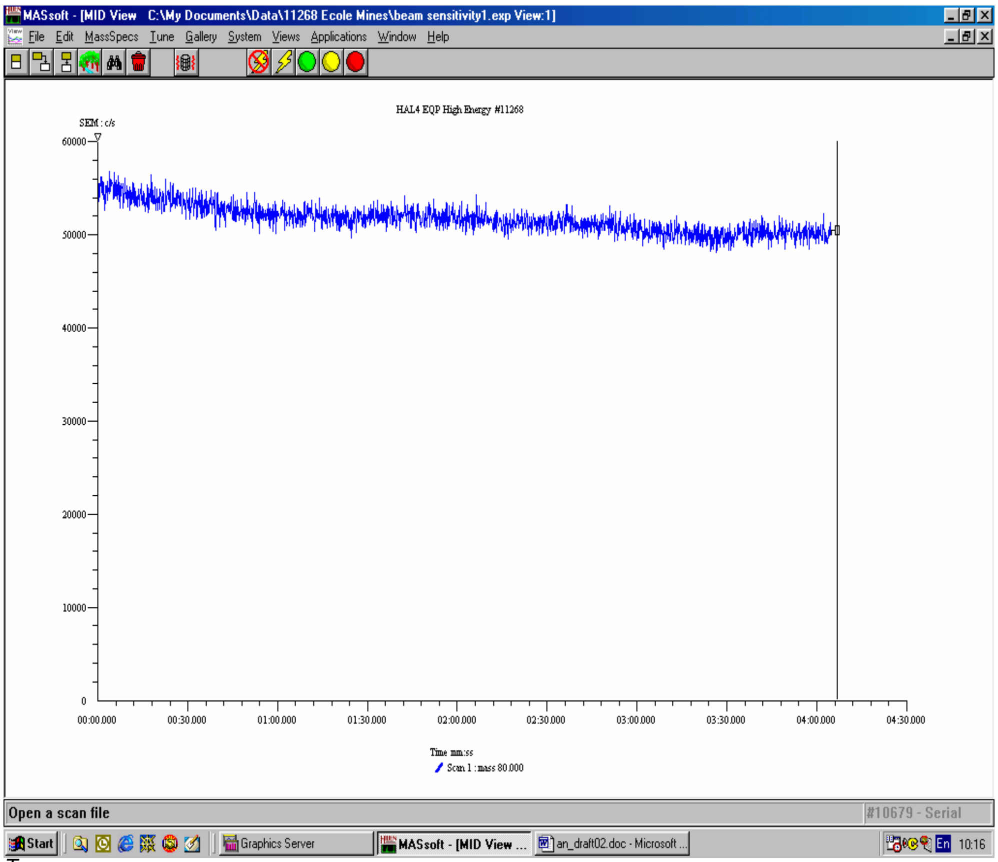

Figure Two

The alignment of the molecular beam was deemed to be good as the background level, equivalent to the beam being "off" was at a level of counts per second.

Usually this would be compared with the strongest Ar 40 peak to examine the amount of dimerisation. However, this peak was too intense for measurement. Hence the secondary Ar 36 peak was measured at a level of approx. 0.3% of that of the Ar 40 peak. The calculated ratios are summarised in Table 2. The scans are seen in Figures 3a and 3b.

Pump Location	SEM / Counts s^{-1}	Ratio to Ar 80
Ar 80	53,000	1 : 1
Ar 36 (0.3%)	2.1×10^6	40 : 1
Ar 40 (100%)	7.0×10^8 (extrapolated)	13,207 : 1

Table 2: Calculated ratios for Ar species.

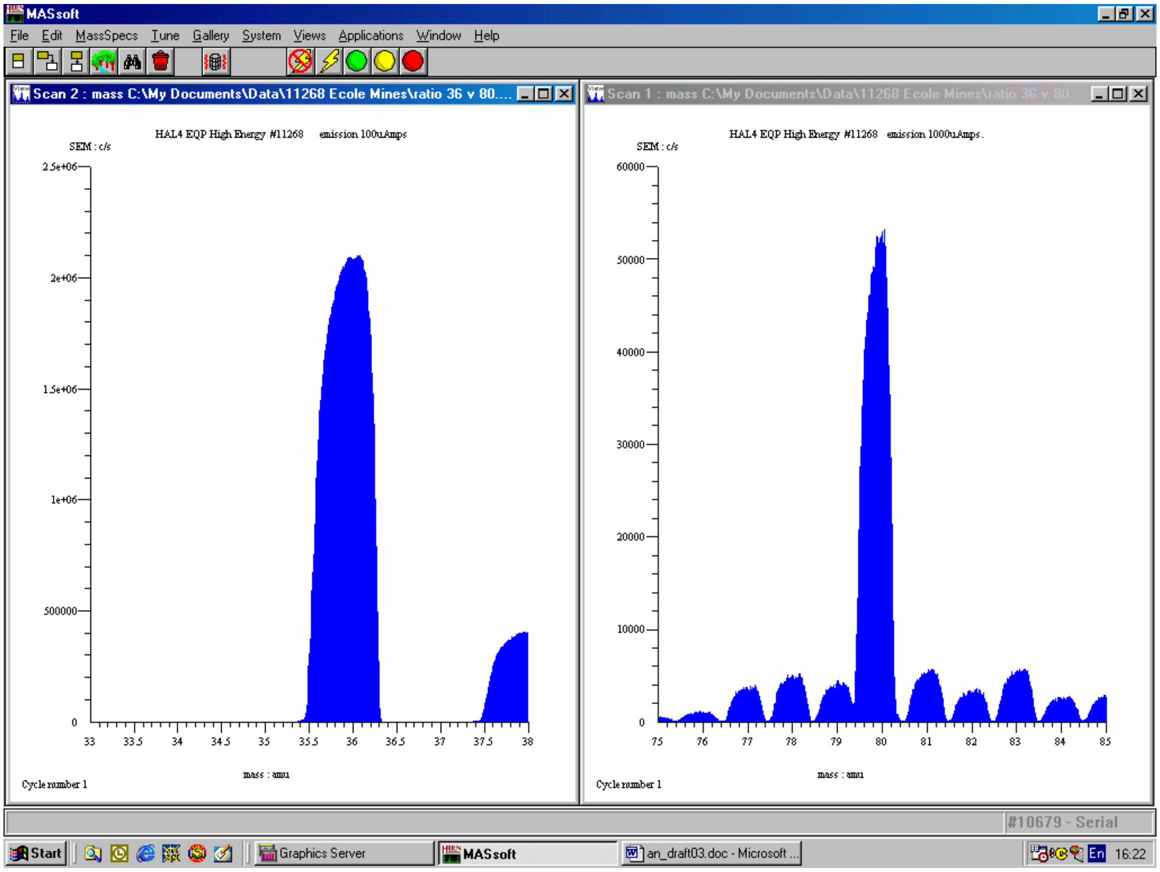

Figure 3a: Mass Scan for Ar36, RGA mode. *Figure 3b: Mass Scan for Ar80, RGA mode.*

Hence the amount of dimerisation and the beam sensitivity to these small levels of species was determined.

Often smaller species only are measured using the gauge. However, higher mass species are also investigated. To test the high mass sensitivity of the gauge, the signal from a sample of heptacosafluorotributylamine, PFBTA ($C_{12}F_{27}N$), was measured. This is a large compound with the an intense peak of mass 219, which can be seen in the mass scan in RGA mode seen in Figure 3. The peak is clearly seen, demonstrating that the mass calibration of the gauge extends up to the higher masses.

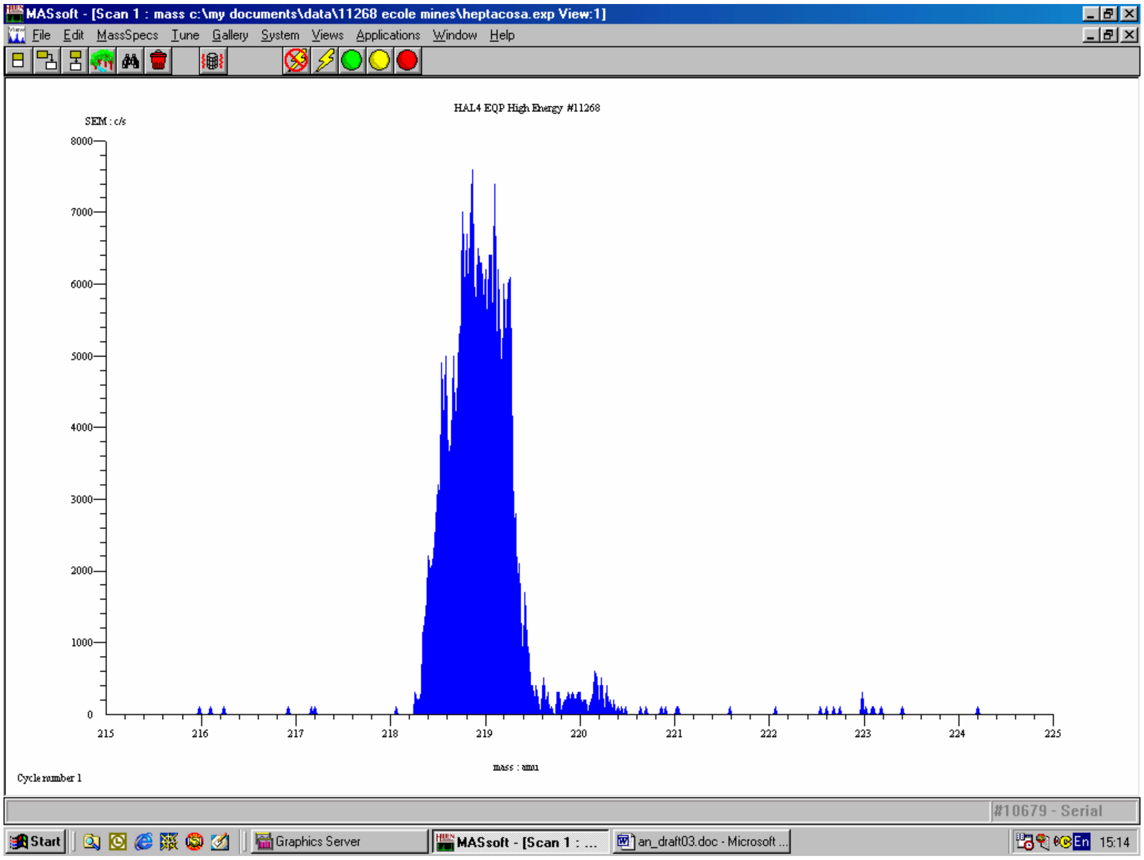

Figure 3: Mass Scan for PFBTA, RGA mode.

To test the detection limits of the gauge, air was sampled. The levels of krypton, Kr, and Xenon, Xe, were extracted by looking for their main peaks, at mass 84 and 132 respectively. The two scan results can be seen in Figures 4a and 4b.

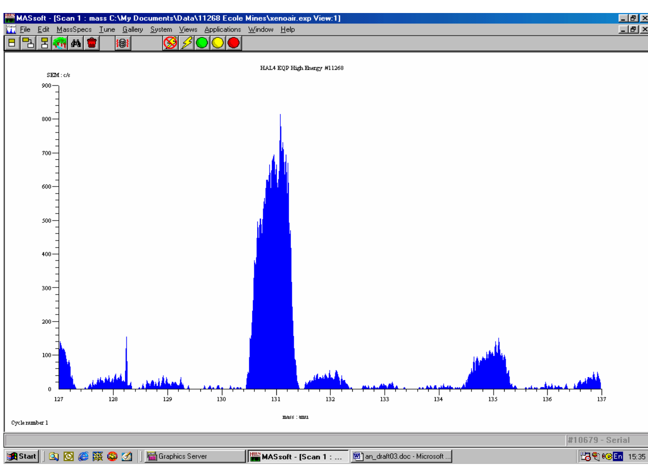

Figure 4a: Mass Scan for Xe, RGA mode.

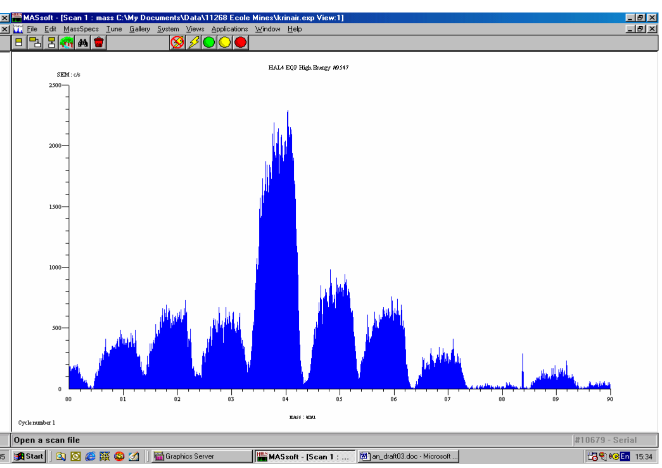

Figure 4b: Mass Scan for Kr, RGA mode.

Again the counts rates give an indication of the concentration in air. These are 700 and 2300, correlating approximately with the rarities in normal atmospheric air of 1ppm and 87ppm respectively, after the relative sensitivity of the mass gauge has been taken into account.

The electron attachment energy was also measured for N_2O in negative ion RGA mode. The scan is seen in Figure 5 with the strongest signal for negative oxygen ions, mass 16, which is a source of this ion.

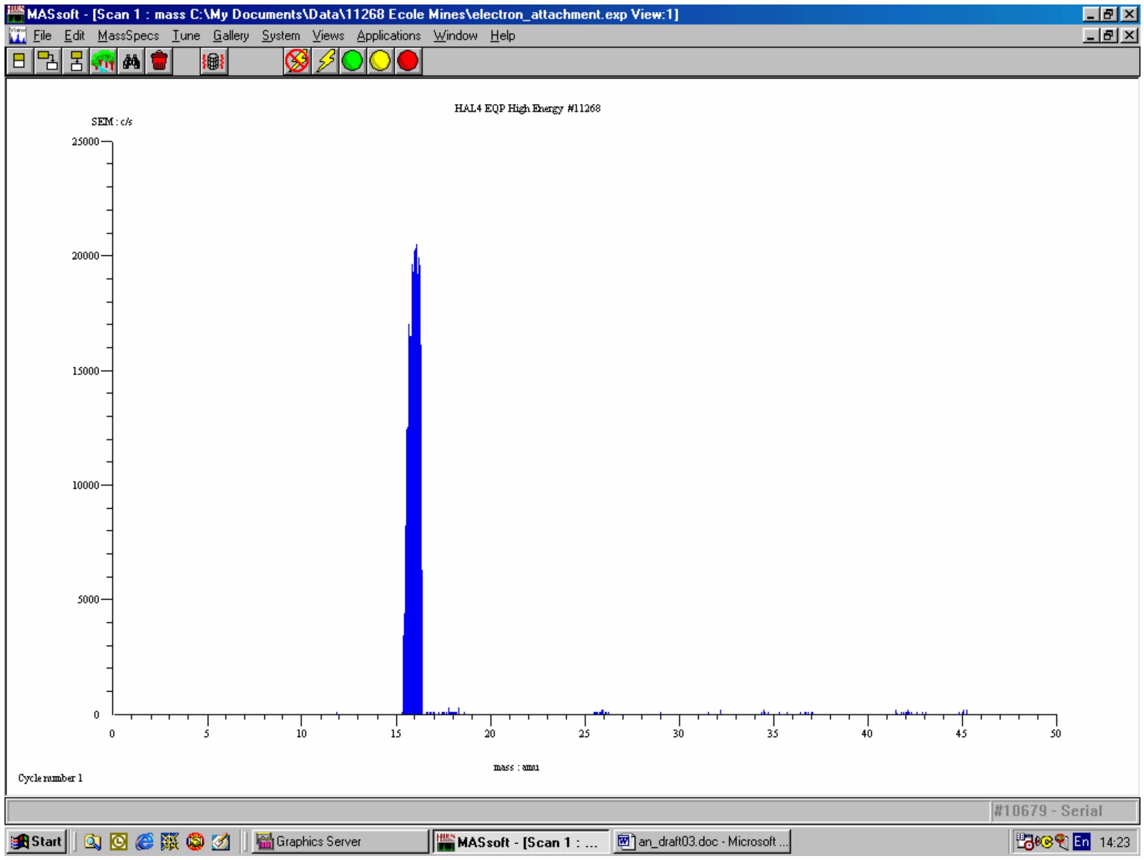

Figure 5: Electron Attachment Scan for RGA mode.

Plasma Characterisation

To use the ion analysis ability of the gauge, the sample was changed from neutral gas to plasma. For this experiment the “atmospheric plasma” was generated in the form of a propane flame. The tip of the flame was positioned close to the first cone orifice for optimal sampling. The bias voltages were set on the cones and pressure readings taken. These set conditions for the instrument are summarised in Table 2.

Stage	Diameter / mm	Bias / V	Pressure Reading / Torr
Sample	--	--	9.0×10^{-1} (atmos.)
Orifice 1	0.1	+30	7.5×10^{-1}
Orifice 2	0.4	-6	4.9×10^{-4}
Orifice 3	0.6	-30	2.0×10^{-7}

Table 2: Conditions for Ar gas experiment.

The voltages were tuned to obtain the required beam through the cone inlet, allowed detailed analysis. Optimisation of the voltages enhances the acceleration effect of the pressure drop and ensures good focus on the source.

Figures 6a and 6b shows the mass data and the energy data respectively, obtained from the gauge in positive ion SIMS mode. Hence only ions produced in the plasma and transported down the molecular beam path will be analysed.

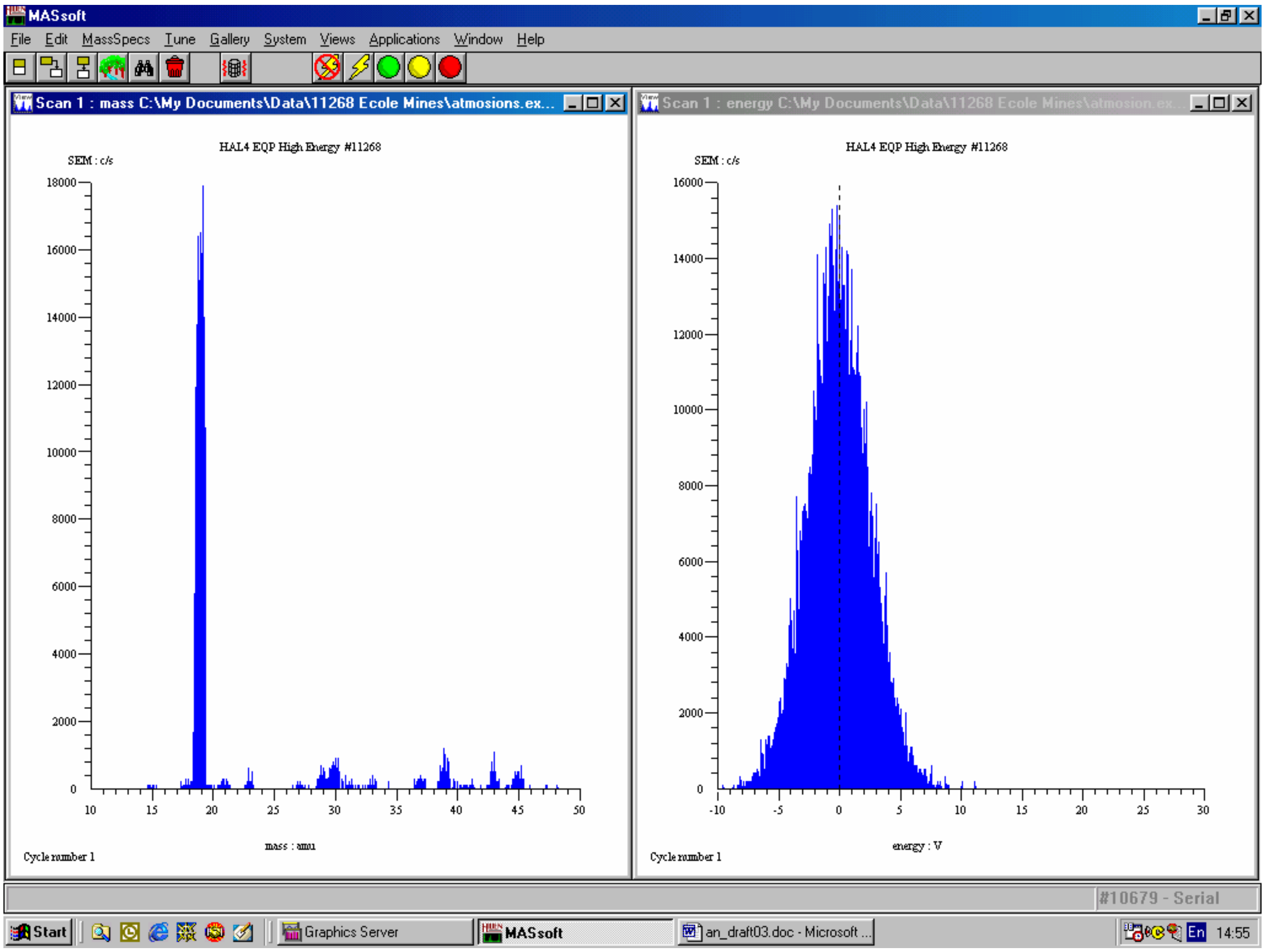

Figure 6a: Mass Scan for atmospheric plasma. *Figure 6b: Ion Energy Scan of atmospheric plasma.*

Figure 6a clearly shows the mass 19 peak. The count rate of 18,000 suggests good focus along the molecular beam path. Other peaks are at a smaller level, but resolved from the noise. Figure 6b shows the energy is centred at zero volts with a peak of 15,000 counts per second. The characteristics of the plasma are therefore defined.

Conclusions

The gauge operates in all the required modes of RGA detecting positive, neutral and negative species. Furthermore the inlet system allows the secondary ions from plasma to be analysed at up to atmospheric level.

Also appearance potential, ion energies and other plasma characteristic can be examined. As well as reducing the pressure through the stages of the inlet system, the three cones can have individual potentials applied to them. This feature is useful for beam studies.

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