



## Plasma Diagnostics

### The EQP Plasma Diagnostic System; an overview

#### Summary

The EQP mass spectrometer combines an electrostatic sector energy analyser with a high performance quadrupole mass filter in an instrument designed for plasma diagnostics. The spectrometer can acquire the mass spectra and energy distributions of neutrals, radicals and ions (both positive and negative). Trends in intensity can be plotted against time. Fast acquisition modes mean that transients and afterglows can be studied.

Manufactured in England by:

HIDEN ANALYTICAL LTD  
420 Europa Boulevard, Warrington, WA5 7UN, England  
t: +44 (0) 1925 445225 f: +44 (0) 1925 416518  
e: [info@hiden.co.uk](mailto:info@hiden.co.uk) w: [www.HidenAnalytical.com](http://www.HidenAnalytical.com)

## Introduction

The EQP Mass Spectrometer is a PC controlled instrument for plasma analysis and diagnostics. Spectra can be easily acquired, stored and manipulated using Windows™ based MASsoft software. The EQP uses an electrostatic sector field energy analyser for ion energy analysis, the transmission and resolution of this device making it the instrument of choice for plasma diagnostics.<sup>(1)</sup> The energy analyser is followed by a triple section quadrupole mass filter. A pulse counting electron multiplier, which can be configured for positive or negative ion operation is used for ion detection. This detector provides high sensitivity and fast response. The spectrometer design provides high dynamic range ( $>10^6$ ) for plasma ions and neutrals.

The EQP operates in one of two modes:

### PI Mode :

**Ions** can be directly extracted from the plasma. These plasma ions (PI) are formed in the plasma, extracted from it and focussed into the energy filter.

### EI Mode :

**Neutrals and radicals** are sampled from the plasma and then ionised at low pressure ( $10^{-5}$  torr ) inside an electron impact ion source. The energy of the ionising electrons may be controlled to enable the detection of **radicals**. Ions from the electron beam source are first transferred and then focussed directly into the energy filter.

The operating modes are summarised in table 1.

Mode	Analysis Mode	Ionisation Point
PI	Mass and energy analysis of ions generated in the plasma.	In the plasma volume.
EI	Mass and energy analysis of neutrals and radicals in the plasma.	In the electron impact ion source.

Table 1 Analysis Modes Summary Table

## Probe Overview

The main constituents of the EQP Plasma Diagnostic System are as follows:

### Differential Pumping:

The probe housing is differentially pumped by a turbomolecular pump. The flow rate of gas and pumping speed is arranged to give a pressure in the

manifold of the order of  $10^{-6}$  torr. An ion or penning gauge attached to the manifold is used to monitor the differential pumping action and provide overpressure protection.

### Electrical Connections:

Two multiway feedthroughs provide electrical connections to the probe.

**Chamber Mounting :**

Standard mounting is via a DN-63-CF (4.5" OD Conflat) flange. This mounting may be customised as required.

**Sampling Orifice:**

The sampling orifice is removable and is mounted at the probe tip. This may be configured in various ways which include; grounded, DC biased, heated, RF driven and electrically floating.

**Transfer Lens:**

In PI mode a transfer lens is used to refocus the ion beam from the sampling orifice into the exit aperture of the electron impact ion source. In this mode the electron impact ion source is switched off and forms a field free drift space for the PI ions. In EI mode, neutral particles emerge from the sampling orifice and travel with linear motion into the ion source.

**Electron Impact Ion Source:**

The electron impact ion source is not used in PI mode. In EI mode the dual filament electron impact ion source is used to create ions from the neutral species which diffuse into the ion source.

The selected filament emits electrons with an energy defined by the variable electron energy. The electron emission current is the current collected by the ion source cage, this current is measured and used to stabilise the filament current. The electron emission current can be user controlled.

The variable electron energy can be used to find the ionisation potential of radicals and neutrals. The electron energy is scanned with other

parameters fixed and intensity is plotted against electron energy.

**Transfer Ion Optics:**

A drift space and lens are used to transfer the EI or PI ions to the input of the energy filter. In this drift space ions are accelerated to a higher kinetic energy. A lens is used to match the ion (EI or PI) into the energy filter.

**Energy Filter:**

The energy filter is a 45 degree sector field electrostatic energy analyser fitted with fringe field correction apertures. The analyser radius is 75mm providing high resolution and transmission.

**Decelerating Lens:**

A decelerating lens reduces the kinetic energy of the ion beam before injection into the quadrupole mass filter.

**Quadrupole Mass Filter:**

The quadrupole mass filter is constructed in three sections, prefilter (rf only), main filter (rf and dc) and post filter (rf only). The mass filter resolution is electronically controlled and is software adjustable. This feature means that the mass resolution can be easily set according to the requirements of the experiment.

**Detector:**

The detector is an off axis mounted continuous dynode electron multiplier which operates in the pulse counting mode. The three variables which control the detector are; (i) the first dynode voltage-this is the voltage on the front of the detector, (ii) the multiplier HT-this is the voltage across the detector and (iii) a discriminator which is used to set a counting threshold on the pulse output

from the multiplier. The pulses from the detector can be electronically gated so that only pulses detected during the gate time are included in the energy or mass spectra. This feature can be used to study afterglows and transient phenomena.

## Probe Installation

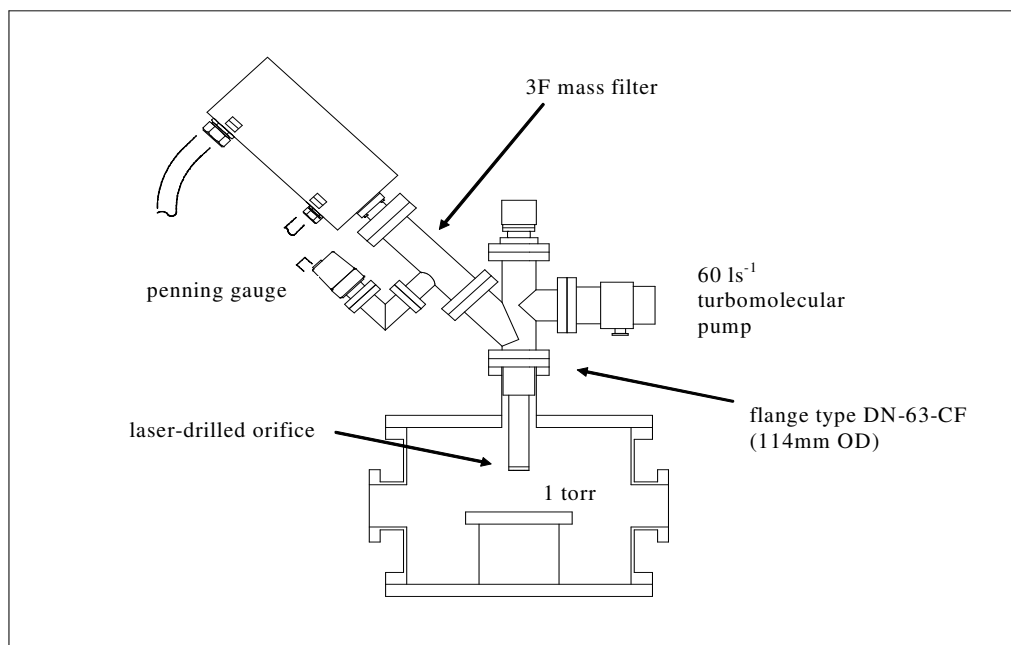
Figure 1 shows a typical probe installation for plasma pressures up to 1 torr. The probe is differentially pumped by a single stage turbomolecular pump. The probe is mounted on the plasma chamber using a 4.5" diameter conflat flange. The drawing in figure 2 shows how a probe can be configured to operate at pressures up to atmosphere.

## Magnetic Shielding:

For applications where a high magnetic field is present the probe can be magnetically screened.

In this case the addition of two further pumping stages extends the sampling range to atmosphere and is applicable to the study of gas kinetics of atmospheric reactions, clusters and high pressure plasmas. The addition of a bellows allows a small amount of mechanical movement at the probe tip. The EQP probe can additionally be configured for all intermediate pressures and mountings can generally be customised to meet the needs of the customer.

Figure 1 EQP Operation up to a system pressure of 1 torr



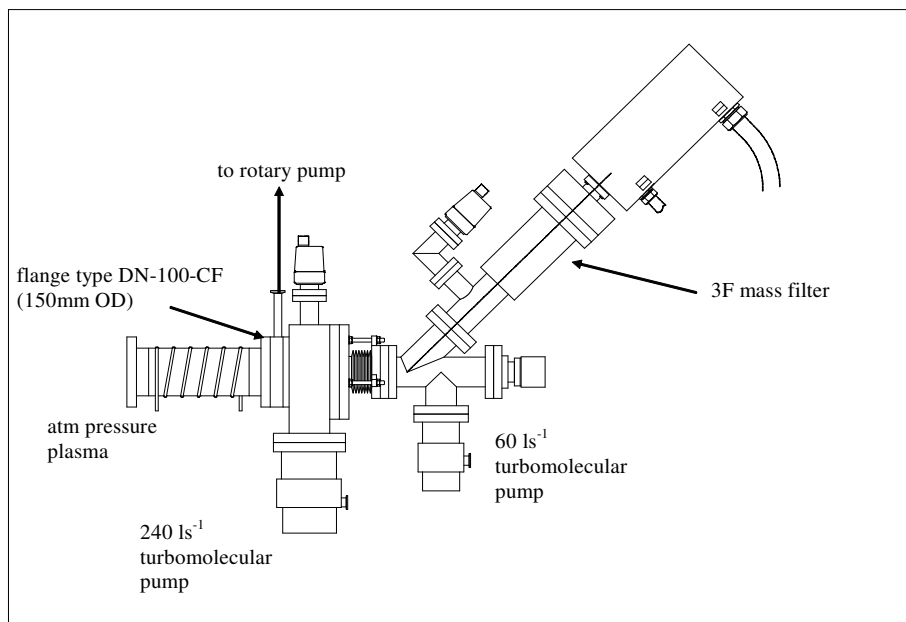


Figure 2 Operation at atmospheric system pressures

## Spectrum Acquisition and Display

The spectra acquired by the EQP can be displayed in several formats. Each format can display data from either positive or negative ions created in the plasma or from radicals or neutrals ionised in the electron impact ion source. The formats and their applications are summarised in table 1.

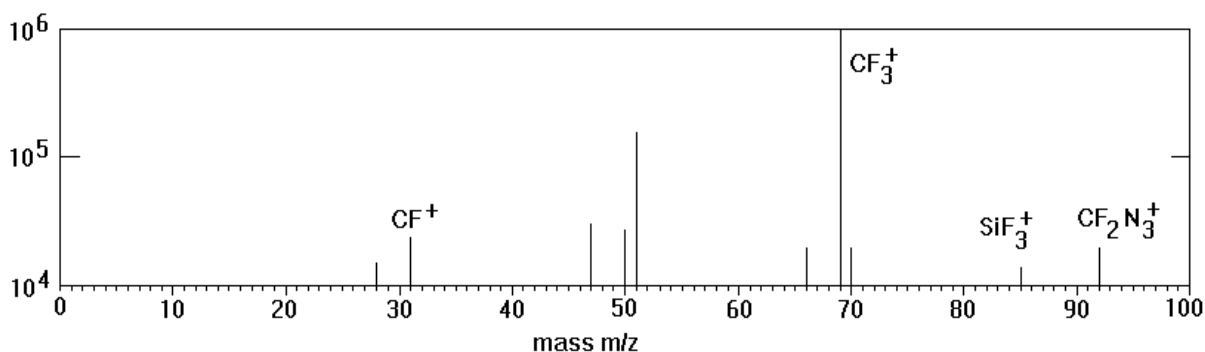


Figure 3 Energy Selected Mass Spectrum from a Freon Plasma Etching Silicon Nitride

## Bar Format

The spectrum in figure 3 shows is a bar format spectrum of positive ions from a Freon 14 plasma. The discharge was RF driven at 13.56 MHz in a Reactive Ion Etching mode. The plasma pressure was 15 mtorr and a silicon nitride substrate mounted on the driven electrode is being Reactively Ion Etched. In this case the intensity scale in counts per second is a two decade logarithmic display the intensity signal can either be displayed in a logarithmic or a linear scale. In logarithmic mode the number of decades displayed can be set between 2 and 7, this facility means that mass spectra can be acquired over a

high dynamic range. The dwell time per sample can be set so that acquisition speed can be traded for signal to noise ratio on peak height. The logarithmic and linear displays are common to the other operating formats PROFILE, MID, ENERGY, ELECTRON ENERGY, and MAP. The mass scale limits can be set to any value within the mass range of the spectrometer. The major peak in the bar mode spectrum is the  $\text{CF}_3^+$  ion at  $m/z$  69. Two interesting ions occur at  $m/z$  85 and 92, these ions incorporate etch products from the substrate and can be used for end point detection. They can be assigned to  $\text{SiF}_3^+$  and  $\text{CF}_2\text{N}_3^+$  respectively.

Name	Format	Application
<b>Bar</b>	Histogram display of mass spectra	Fast survey of a complete mass spectrum. Mass assignment of major and minor peaks. Finding the ion or neutral species involved in process phenomena.
<b>Profile</b>	Continuous display of mass spectra	Survey of a small range of a spectrum, checking instrument resolution and abundance sensitivity.
<b>MID</b>	Multiple Ion Detection. Display of selected ions intensity against time (Trend Analysis) either in tabular or graphical mode.	Following variations in time varying signals. End point detection.
<b>Energy</b>	Continuous display of mass selected energy spectra.	Determination of plasma potential or ion energy distribution. Determination of collisions in sheaths. Measuring the variations in ion energy distribution with mass.
<b>Electron Energy</b>	Continuous display of mass selected ion intensity as a function of electron energy.	Determination of appearance potential, differentiation of radicals from a parent stable neutral molecule. For example measuring the intensity of $\text{CH}_3$ in a $\text{CH}_4$ background.
<b>Map</b>	Continuous display of ion intensity as a function of ion optical tuning parameters.	Check instrument "tuning" parameters and that the transmission of the instrument is optimised.

Table 1 Acquisition and Display Formats

## Profile Format

Figure 4 is a profile format mass spectrum of positive ions from the plasma. Plasma conditions are similar to those described in figure 3. The profile spectrum is an analogue display of intensity against mass. The mass window is variable over the mass range of the instrument and the dwell time per sample can be set. The spectrum shows a high resolution scan of a 40 mass unit window. The most significant ion in the

spectrum is  $m/z$  114 assigned to  $C_2F_4N^+$ , this ion provides a high intensity signal and is suitable as an end point detection signal when layers of silicon nitride are etched. Other ions in the spectrum correspond to etch products and ions formed by ion molecule reactions in the plasma, for example: 85 -  $SiF_3^+$ , 92 -  $CF_2N_3^+$ , 119 -  $C_2F_5^+$ .

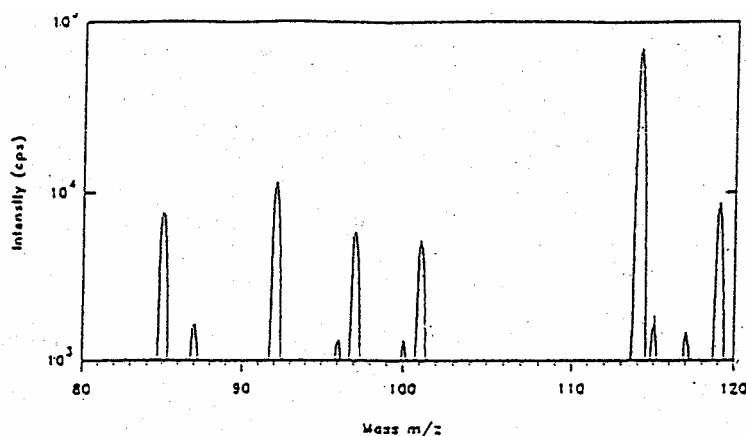


Figure 4 Profile Mass Spectrum of ions from a Freon Plasma

## Mid Format

In Multiple Ion Detection format the intensity of many ions can be monitored as a function of time. Up to 100 mass channels can be simultaneously monitored. The ion intensity can then be displayed using one of two methods.

**Graphical mode** - plots the intensity of each ion against time on the screen.

**Tabular mode** - prints the intensity of each ion in counts per second in tabular form on the screen.

In either mode the dwell time per peak can be defined. The acquired data is displayed in real time on screen and automatically saved allowing for post processing by any Windows™ supported software package.

Figure 5 shows the variation of ion intensity of the ion at 114  $m/z$  shown in figure 4. In this spectrum the plasma is reactively ion etching a 2000 angstrom layer of silicon nitride on a gallium arsenide substrate. The intensity of the nitrogen containing molecule at  $m/z$  114 can be used as an end point indicator.

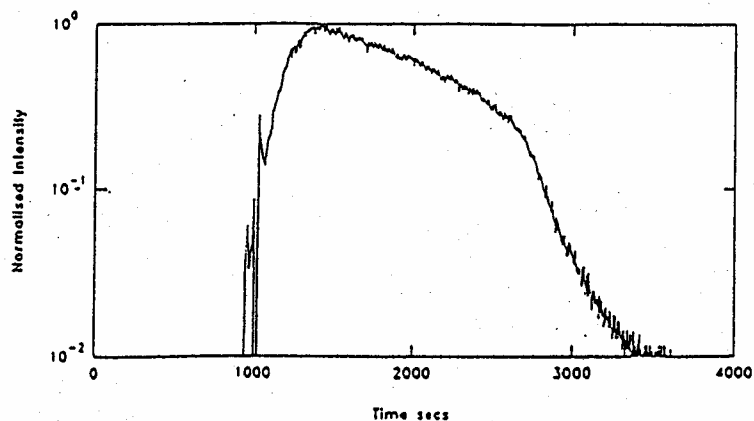


Figure 5 Time Variation of the Intensity of m/z 114 - End Point Detection

## Energy Format

An energy spectrum from a freon plasma is shown in figure 6. The spectrum shows the energy distributions for two ions-  $\text{CF}_3^+$  at m/z 69 and  $\text{CF}^+$  at m/z 31. The energy range of the instrument is 100 ev with an option for

1000ev operation. The energy spectrum can be used to determine the plasma potential, tailing on the energy spectra is an indication of collisions taking place in the plasma sheath.

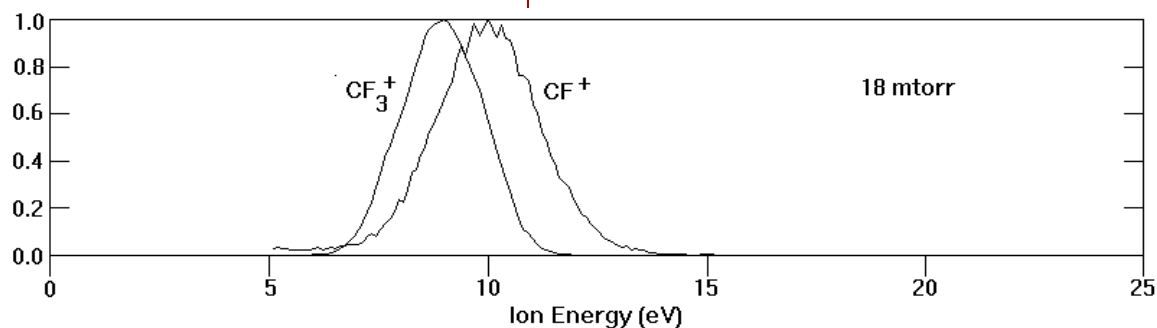


Figure 6 Energy Spectra from a Freon Plasma

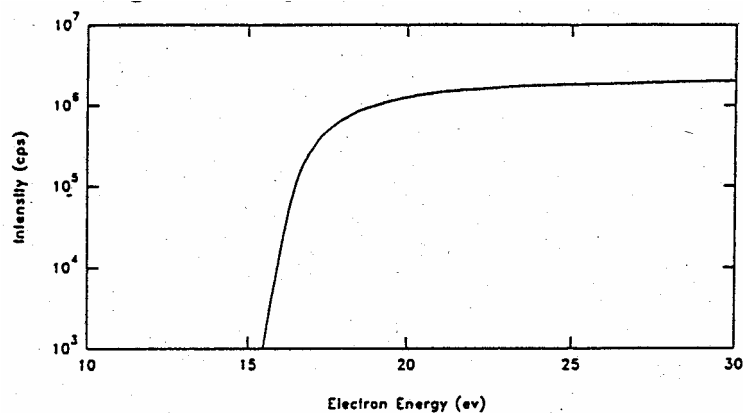


Figure 7 Ionisation Potential of Argon by scanning the electron energy



## Electron Energy Format

In the EI mode of operation neutrals and radicals are sampled from the plasma and ionised in the electron impact ion source. The kinetic energy of the electrons is controlled by the variable electron energy. When this variable is scanned as in figure 7, it is possible to determine the ionisation potential of the ion selected by the mass

filter. Figure 7 shows an electron energy spectrum of argon with an ionisation potential of about 15.5 volts (the mass filter is set to transmit  $m/z$  40.)

The electron energy scanning technique can be used to differentiate radicals from ions formed by the electron fragmentation of a parent gas molecule<sup>(2)</sup>.

## Map Format

In Map format the lens voltages can be scanned and intensity plotted against lens voltage. This mode is a useful

diagnostic for checking instrument tuning. Displays are similar to energy or electron energy scans.

## References

1. PLASMA DIAGNOSTICS Volume 1 Ed. Auciello and Flamm, ACADEMIC PRESS.
2. PLASMA MONITORING WITH HIDEN ANALYTICAL DIAGNOSTIC EQUIPMENT. Chatterton and Smith.