HIDEN
A N A L Y T I C A L

EQP and EQS Analysers

Summary

Hiden Analytical EQP and EQS analysers are capable of monitoring ions from an external source, or neutrals which are ionised by the internal ion source. The EQP is used where the external ion source is a plasma, and the EQS where the source is, for example, a bombarded surface or laser induced ionisation at high vacuum. All the diagrams in the following document apply to both analysers, the only difference being in the components used to form the ion extraction system.

The probes for these analysers are comprised of 5 sections, extractor, RGA source, energy analyser, quadrupole mass filter and detector. Figure 4 shows the relative positions of the components, and figure 5 the electrical connections.

Figure 6 shows the relationship of the potential and kinetic ion energies during transit through the probe.

Figure 7 shows the variation in acceptance angle of the probe vs. ion energy. This graph was calculated using the SIMION programme.

Extractor

EQP analyser

Two types of extractor system are available, one for low temperature, the other for high temperature, both of which are suitable for positive and negative ion work. Figure 1a shows the low temperature version, and figure 1b the high temperature version.

Both types of probe are differentially pumped.

In Ion Analysis mode (labelled SIMS in the control menus), ions are extracted from the Plasma via a sampling orifice, behind which is the extractor electrode itself and lens 1. The orifice diameter is small (50µm - 200µm) and depends on the process pressures. Lens 1 focuses the ions into the following ion transport system.

In RGA operation, the extractor potential is normally set to prevent the ingress of ions from the plasma to ensure that only neutrals are analysed.
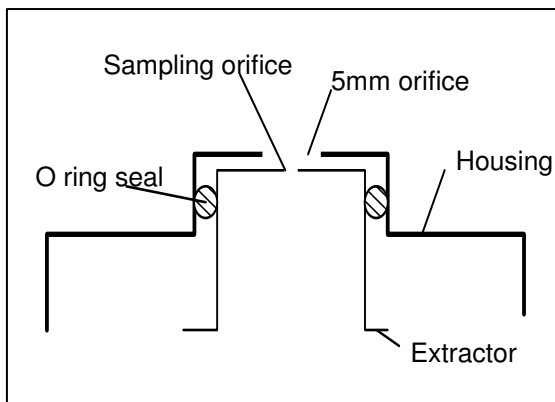
Low Temperature version

Figure 1a Low temperature extractor

In the low temperature version, the sampling orifice is situated in the extractor itself, behind a 5mm hole in the probe housing. Sealing for differential pumping purposes is provided by a Viton O ring between the extractor and the housing.
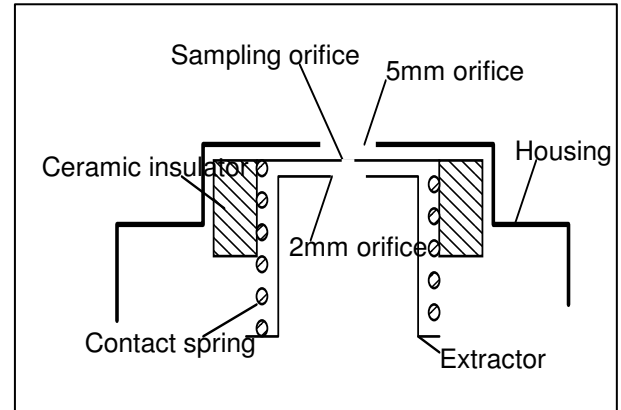
High Temperature version

Figure 1b High temperature extractor

The high temperature version dispenses with the O-ring seal and uses a ceramic insulating ring carrying an orifice plate to provide the differential pumping seal. The orifice plate is electrically connected to the extractor by a spring.

EQS analyser

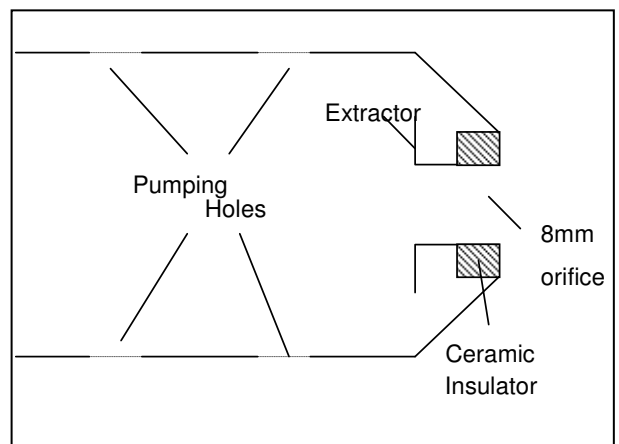Figure 2 shows the EQS extractor system.

Figure 2 EQS extractor

The EQS extractor is simply a larger diameter hole, insulated from the probe supporting body. Since the probe is expected to operate in high vacuum

systems, there is no need for differential pumping, and holes are cut in the sides of the support tube to allow internal components to be pumped.

RGA source

In Ion Analysis and SIMS modes, the RGA source is held at AXIS+ENERGY potential, so the ions pass straight through.

In RGA mode, the source is referenced to 0v (ground), except in the high voltage EQP where it is referenced to REF potential. In this mode it acts as an electron impact ion source.

Energy analyser

A 45º Sector field energy filter with inner and outer plate radii of 68mm and 82 mm is used to provide ions to the mass filter, and is preceded by a D.C. quadrupole lens to correct for the different focal length of the sector analyser in x and y axes. This lens is connected to the QUAD, VERT and HORIZ potentials. The PLATES potential appears across the energy filter plates, with the mid-point connected to the AXIS potential. At the exit from this filter, the ions are focused for presentation to the mass filter by a series of lenses, the first of which also defines the resolution of the energy filter. By default the aperture in this lens, which is held at AXIS potential, is 3mm, giving a nominal resolution of ±0.75eV. This aperture can be changed if other resolutions are required. An instruction sheet is available for this operation.

Quadrupole mass filter

The Hiden high transmission triple filter mass analyser with Molybdenum rods is used here.

Detector

A channeltron SEM detector, optimised for pulse counting, and preceded by a conversion dynode (1st DYNODE) is used as the detector, operating at 2kV+, normally on a count rate vs. voltage plateau. The conversion dynode produces electrons when struck by either a positive or negative ion, which are then attracted to, and trigger, the SEM. This arrangement simplifies the pulse detection electronics as only one particle polarity needs to be detected. The detector circuit for a positive/negative ion probe is shown in figure 3a. For positive ion detection, the 1st DYNODE is operated at a net negative potential, and for negative ions it is set to a positive potential. The voltage between the conversion dynode and the SEM (MULTHT) remains at a constant positive potential to attract the electrons to the SEM. For a positive ion only probe the circuit is as in figure 3b. In this case, MULTHT is negative.
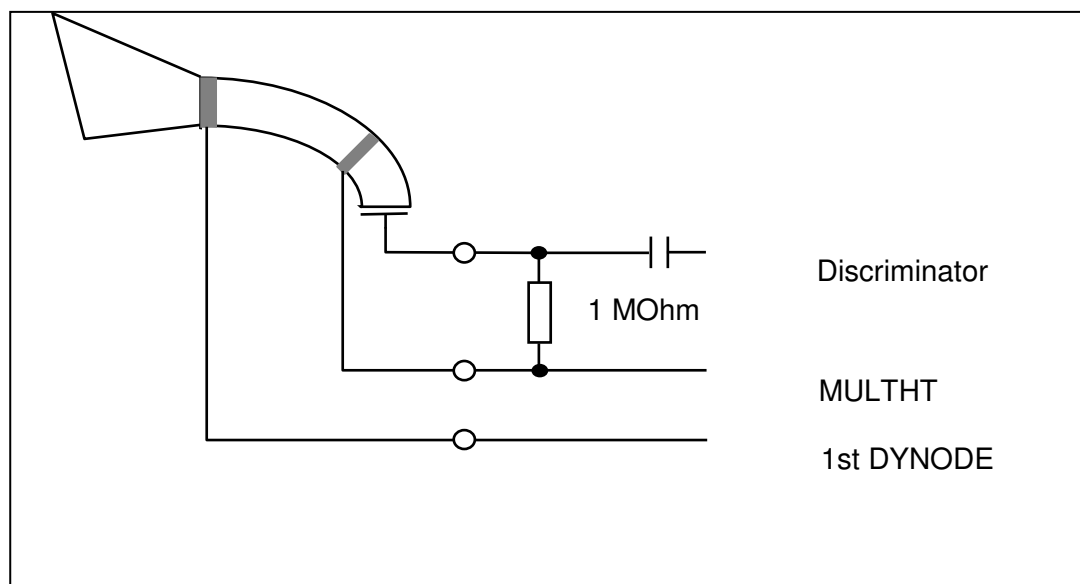
Figure 3a Positive/negative ion detector
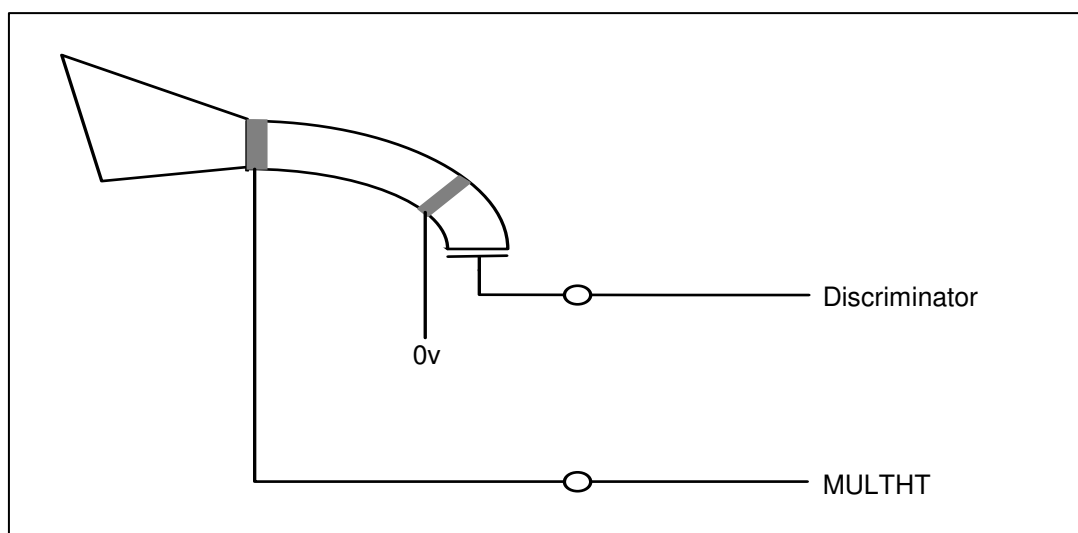
Figure 3b Positive ion only detector

A suppressor operating at a negative voltage (for positive ions) precedes the detector to prevent electrons triggering it.

Electrode descriptions

- **<u>Extractor</u>** - Set to attract ions from the external source, or set positive to prevent ions entering the probe for neutral/radical detection.
- **<u>Lens 1</u>** - Set to quickly remove ions from the extractor region and focus them into the source.
- **<u>Axis</u>** - Used to give the ions a specified energy prior to the energy analyser. Set negative for positive ions and vice-versa. Tune using the Quad, Vert and Horiz in

the Sector menu.

- **Lens 2** - Focuses the ion beam on exiting the Axis section.
- **Focus** - Focuses the ion beam into the quadrupole mass filter.
- **Suppressor** - Prevents electrons triggering the SEM.
- **Energy** - Normally scanned to determine the best setting.
- **1st Dynode** - Forms the front end of the SEM. Set for the ion type.
- **Filaments** - For neutral species, set E-ENGY to a high negative voltage sufficient to ionise neutrals in the source cage. For ions, the filament is connected to Energy.
- **Source cage** - For neutral species, set to a small positive voltage so that the ions have enough energy to reach the detector. For ions, the source cage is connected to Energy.

The following table indicates <u>possible</u> values for the analyser elements.

| Element	RGA	SIMS +ve ion	SIMS -ve ion
Extractor	100v	0v to -100v	0v to 360v
Lens 1	-100v	-100v to -200v	100v to 200v
Cage	3.0v	n/a	n/a
Electron energy	70v	n/a	n/a
Lens 2	-95v to -120v	-95v to -120v	95v to 120v
Quad, ,	±30%	±30%	±30%
Vert	±20%	±20%	±20%
Horiz	±20%	±20%	±20%
Plates	7.4v to 7.8v	7.4v to 7.8v	-7.4v to -7.8v
Axis	- 40v	- 40v	40v
Energy	0v to 3v	Plasma dependent	Plasma dependent
Focus	-150v to -350v	-150v to -350v	150v to 350v
Suppressor	-200v	-200v	200v
1st Dynode	-1200v	-500v to -1200v	800v
SEM	1.8kv to 2.5kv	1.8kv to 2.5kv	1.8kv to 2.5kv

Table 1 Typical operating values
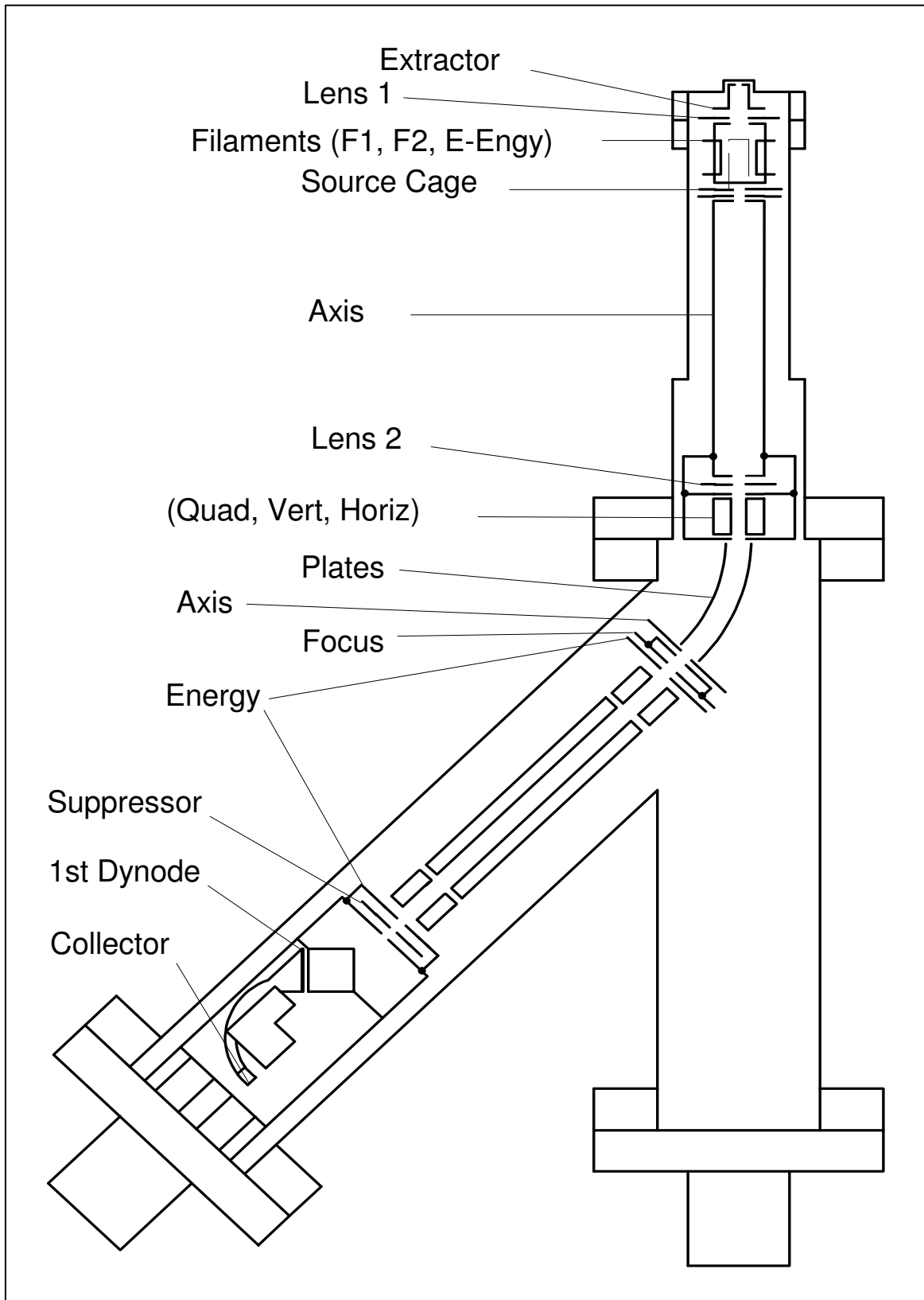
Fig 4 EQP Elements
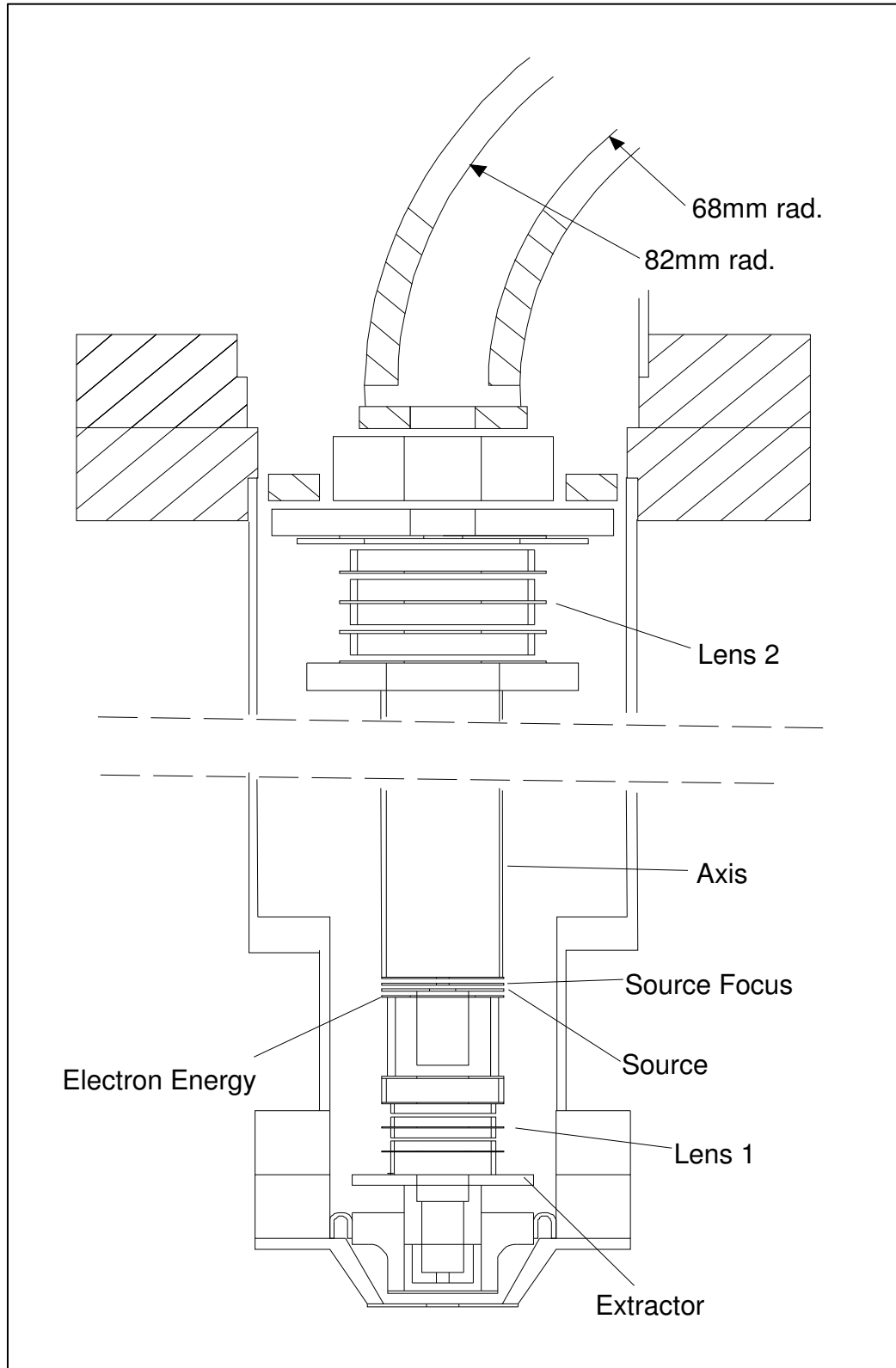
Fig 4a High temperature EQP extractor detail
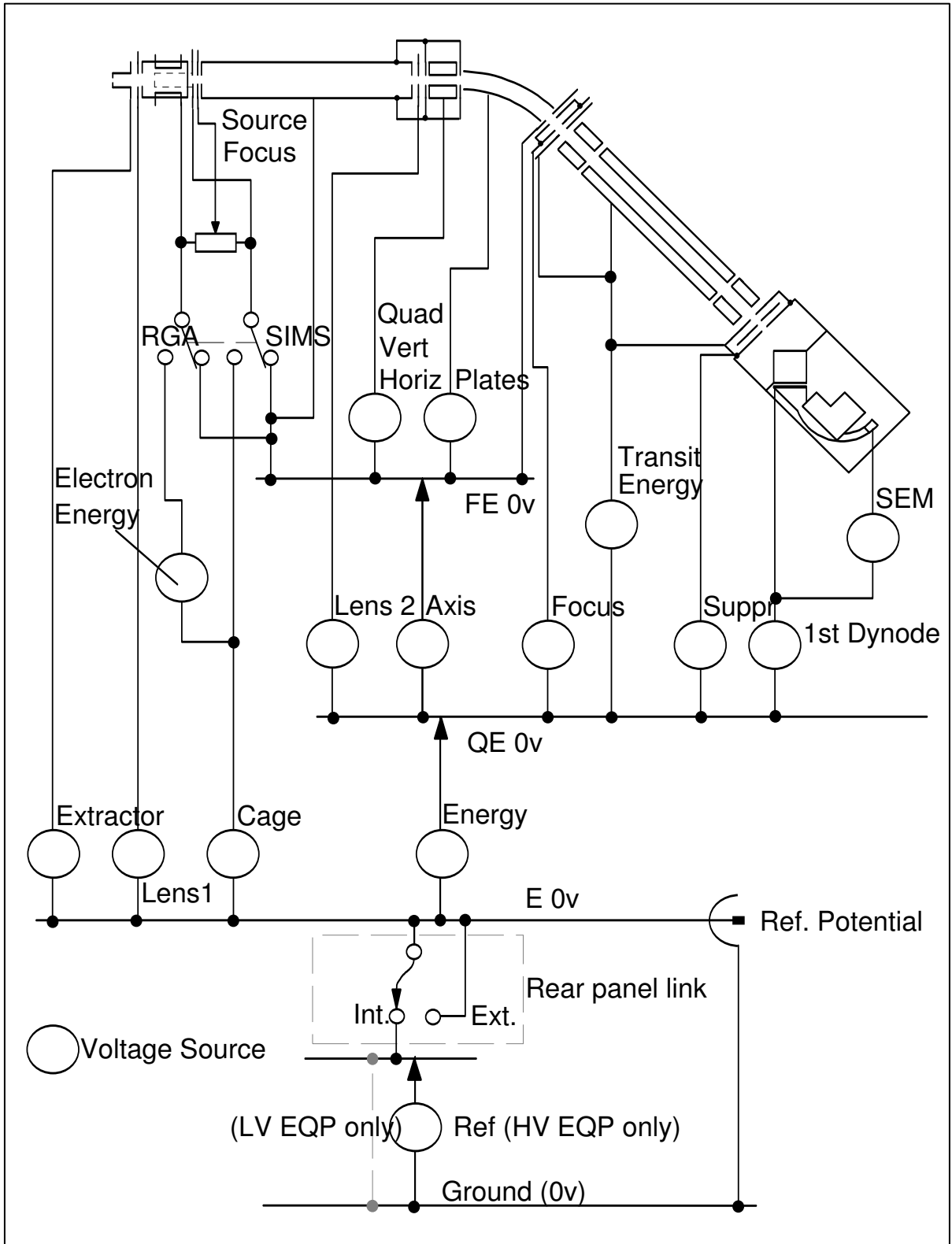
Fig 5 Lens names and references

Energy analysis

Kinetic Energy

| Orifice | Extractor | Drift tube + ESA | Quadrupole mass filter |

Ex

Ew

Ei

Eq

Ei = Incident Ion energy
Ex = Energy in Extractor system
Ew = Energy in Energy filter
Eq = Energy in Quadrupole mass filter

Potential Energy

| Orifice | Extractor | Drift tube + ESA | Quadrupole mass filter |

0v

Vi

Vw

Vx

Vq

Vi = Incident Ion potential (assumed 0v)
Vx = Potential in Extractor system
Vw = Potential in Energy filter (= Energy + Axis)
Vq = Potential in Quadrupole mass filter (= Energy)

Figure 6 Potential and kinetic energy relationships

Energy measurement

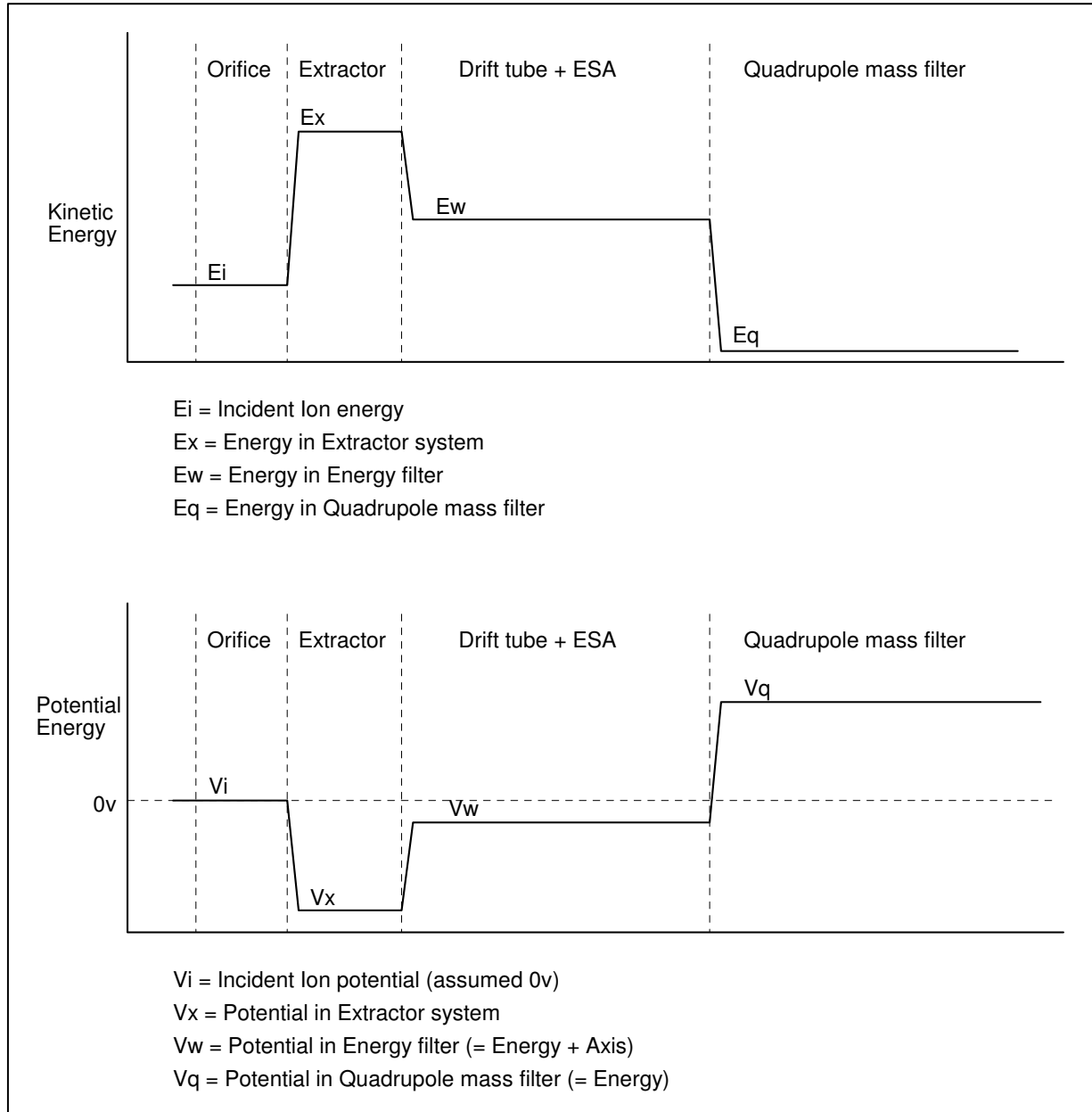Ions with incident energy E_i are accelerated or decelerated to transit the energy filter with energy E_w

$$E_w = E_i + (AXIS - ENERGY)n\varepsilon \tag{1}$$

(*ENERGY* and *AXIS* voltages are connected to have opposite polarity)

To pass through the energy analyser, an ion must have energy

$$E_w = \frac{R}{d}PLATESn\varepsilon \tag{2}$$

where R is the mean radius of the sector, d the distance between the plates, *PLATES* the magnitude of the potential between the sector plates, and ne the total charge on the ion.

Equating (1) and (2)

$$E_i + (AXIS - ENERGY)n\varepsilon = \frac{R}{d}PLATESn\varepsilon$$

$$\therefore \quad E_i = ENERGY.n\varepsilon + (\frac{R}{d}PLATES - AXIS)n\varepsilon 2)$$

or
$$E_i = ENERGY.n\varepsilon + \delta E$$

For the *ENERGY* voltage to correctly indicate the incident ion energy the δE term should be zero. However, on entering the mass filter, the ions have been slowed down from *AXIS* potential, but still require an energy δE to transit the quadrupole.

$$\delta E = E_w - AXIS.n\varepsilon$$

Substituting from 2)

$$\delta E = (\frac{R}{d}PLATES - AXIS)n\varepsilon$$

which means that *ENERGY* potential underestimates the true ion energy. For the EQP and EQS, the ratio

$$\frac{R}{d} = 5.498$$

,

so that a correction based on the *PLATES* and *AXIS* voltages can be applied. These

voltages are not normally altered, so this correction need only be calculated once.

Applying this correction in terms of electrode voltages

$$\delta V = \frac{R}{d} PLATES - AXIS$$

so that the true ion energy for singly charged ions is given by

$$ENERGY + \frac{R}{d} PLATES - AXIS$$

Hiden Analytical acknowledge, with gratitude, the work by Dr. Alan Howling on which this analysis is based.

*Note that instruments with **Transit Energy** available do not need this correction applied, as the flight energy through the mass filter is set independently.*

Energy resolution

For a sector energy analyser, the energy resolution is given by

$$\Delta E = \frac{w.TE}{R(1 - \cos\phi) + L\sin\phi}$$

Where ΔE = Energy resolution

    w = aperture diameter

    TE = Transmission Energy

    R = sector mean radius

    ϕ = sector angle

    L = distance to exit aperture

w = aperture diameter
R = mean radius of sectors
↗ = sector angle
L = distance from sector to exit aperture
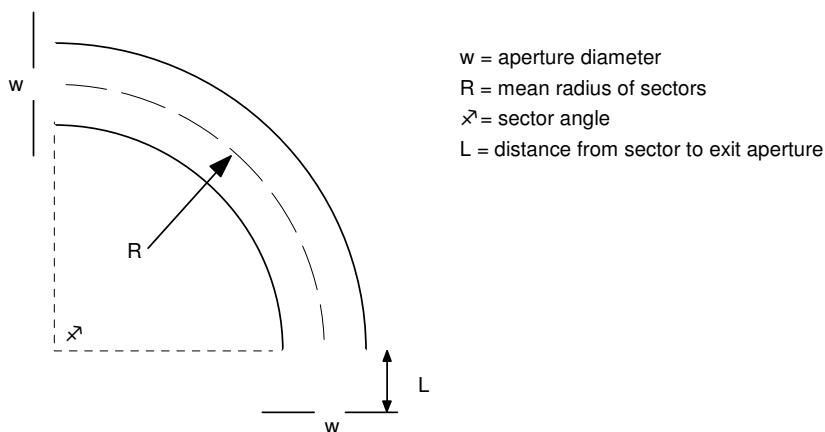
In the EQP/EQS, the following values are used:-

w = 3 mm standard, adjustable as required

R_1 = 68 mm

R_2 = 82 mm

R = 75 mm (average of R_1 and R_2)

ϕ = 45 degrees

L = 35.4 mm

TE = 40 eV standard, user adjustable as required

TE	w				
	5 mm	3 mm	2 mm	1 mm	0.5 mm
5 eV	0.53	0.32	0.21	0.11	0.053
10 eV	1.06	0.64	0.43	0.21	0.11
20 eV	2.13	1.28	0.85	0.43	0.21
30 eV	3.19	1.91	1.28	0.64	0.32
40 eV	4.26	2.55	1.70	0.85	0.43

Table 2 Energy resolution for given pass energy and aperture sizes

(Default aperture size and pass energy are shown shaded).

Transmission Energy

The pass energy for the sector analyser is given by:-

$$TE = \frac{\Delta V}{\dfrac{R_2}{R_1} - \dfrac{R_1}{R_2}}$$

where ΔV = potential difference between sector surfaces.
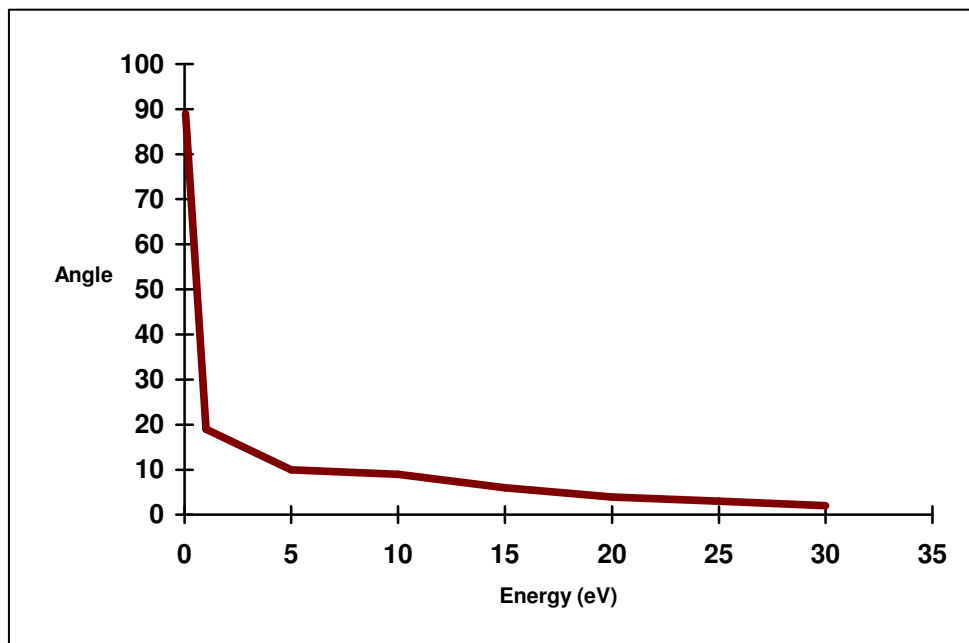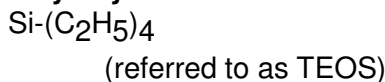
Probe acceptance angle

Figure 7 Probe acceptance angle vs. Ion energy

The acceptance angle data is derived from SIMION trajectory calculations on the ion transport system, and not on experimental data.
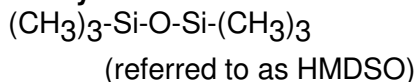
Mass Discrimination

To determine the mass discrimination of the instrument, four substances were used to find the response of the analyser and detector. The compounds used were:

Tetraethyloxysilane

$Si-(C_2H_5)_4$

(referred to as TEOS)

Hexamethyldisiloxane

$(CH_3)_3-Si-O-Si-(CH_3)_3$

(referred to as HMDSO)

Tetramethyldivinyldisiloxane

$(CH_3)_2-Si-O-Si-(CH_3)_2$

C_2H_5 C_2H_5

(referred to as VMDSO)

Hexamethyldisilazane

$(CH_3)_3-Si-NH-Si-(CH_3)_3$

(referred to as HMDSN)
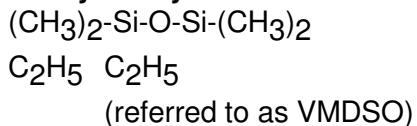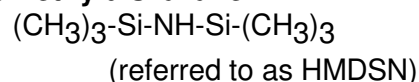
Spectral data for these compounds was taken from the "Eight Peak index of Mass Spectra" (Unwin Brothers, 1977) and is shown in the "Library Data" columns of Figure 9 below. The "Measured Data" columns show normalised data measured in RGA mode with 70eV electron energy, and the "Corrected Data" columns show the data corrected and referred to mass 28, which is given a value of 100%. Figure 8 shows the mass dependency graphically.

Figure 8 Mass Dependency

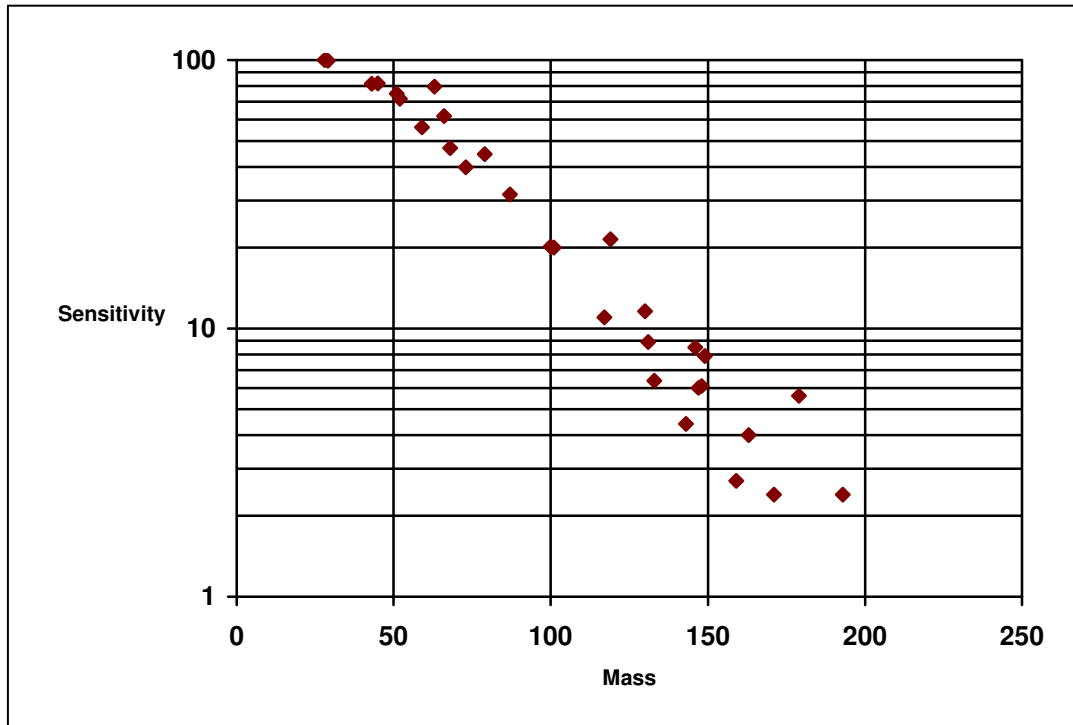| Mass	Library Data				Measured Data				Corrected Data				Transmission
	TEOS	HMDSO	VMDSO	HMDSN	TEOS	HMDSO	VMDSO	HMDSN	TEOS	HMDSO	VMDSO	HMDSN	%
28		5.0			100.0	70.0	100.0	35.8		100.0			100.0
29	30.0	3.0			26.4	10.1	6.1	3.3	99.5				99.5
43		3.5			11.5	25.3	23.9	25.9		81.7			81.7
45		6.9	9.0	6.7	41.6	77.7	38.9	77.7		80.4	66.8	98.6	81.9
51		2.0			6.4	13.6	14.4	2.8		74.9			74.9
52		3.0			2.5	49.6	21.1	8.8		71.8			71.8
59		6.5	25.0	6.0	3.6	54.8	81.7	40.8		60.2	50.4	57.9	56.2
63	31.0				21.9	0.1	0.8	0.0	79.6				79.6
66		9.8		13.0	2.1	100.0	4.7	21.5		72.9		50.7	61.8
68		9.0			1.0	0.2	0.4	0.0		47.0			47.0
73		13.5	18.0	11.7	4.5	73.7	47.3	55.2		39.0	40.6	40.2	39.9
79	56.0				22.1	0.1	1.8	0.0	44.6				44.6
87		2.5			0.5	5.5	4.2	1.8		31.6			31.6
100				7.0	0.1	0.1	0.3	16.6				20.2	20.2
101				8.0	0.1	1.5	0.7	2.4				20.0	20.0
117			80.0		1.9	3.8	57.0	0.7			11.0		11.0
119	36.0				6.8	0.9	8.6	0.0	21.5				21.5
130				27.3	0.2	0.1	0.6	37.1				11.6	11.6
131		5.1			0.3	6.4	4.6	5.4		8.9			8.9
133		2.0	11.0		0.5	2.0	4.1	0.4		7.1	5.8		6.4
143			48.0		0.4	0.5	13.7	0.0			4.4		4.4
146				100.0	0.4	0.2	0.4	100.0				8.5	8.5
147		100.0		26.7	1.3	89.7	0.1	17.3		6.4		5.5	6.0
148		15.8		10.5	0.2	13.2	0.0	7.7		6.0		6.2	6.1
149	85.0	9.0			8.1	6.5	0.0	0.8	10.7	5.2			7.9

159			11.0		0.1	0.1	1.9	0.0			2.7		2.7
163	69.0				2.4	0.0	0.0	0.4	4.0				4.0
171			100.0		0.5	0.06	15.4	0.0			2.4		2.4
179	26.0				1.3	0.0	0.0	0.0	5.6				5.6
193	100.0				2.1	0.0	0.0	0.0	2.4				2.4

Figure 9 Mass Discrimination data

EQP Operating pressures

For a single pumped EQP with a 60 l/s turbomolecular pump fitted, the following orifice sizes are required for the stated plasma chamber pressures.

Maximum process pressure - mTorr	Orifice Diameter microns
500	50
100	100
40	150
20	200
15	250
10	300

(Source HAS-027-008 issue A)

Probe transit time

For certain modes of operation it is important to know the time taken for ions to transit the probe.

In this section, EQS and EQP probes are treated as identical. The difference in the extractor geometry will not significantly affect the analysis.
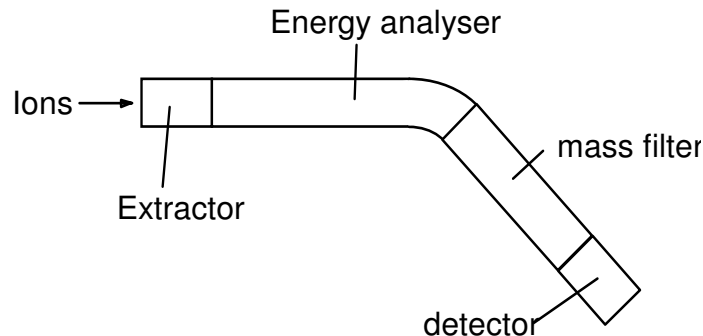
The analyser can be divided into four sections for transit time analysis:

- Extractor region
- Energy analyser region
- mass filter region
- detector region

Ions transit each of these regions at different energies.

The total transit time for the analyser is

$$t = t_{ext} + t_{en} + t_{mass} + t_{det} \qquad (1)$$

This transit time is for ions carrying a single charge. Where a multiple charge is carried, the effective mass of the ion is the real mass divided by the number of charges.

Extractor region

Ions are assumed to enter this region with an energy derived from their method of formation or the potential at which they are formed. The potential at the entrance to this region is the sum of the extractor voltage V_{ext} plus the reference voltage V_{ref}, and the exit potential is the sum of the axis voltage V_{axis}, the energy voltage V_{en}, and the reference voltage V_{ref} (see the voltage reference diagram above). This region is treated as a drift space (which will give a small error) as otherwise the analysis becomes very complicated.

Put $V_{in} = V_{ref} + V_{ext}$

If the ion is formed with zero kinetic energy at a potential of V_{form}, then it will enter this

region with energy

$$E_i = e(V_{form} - V_{in})$$

so that

$$\frac{mv^2}{2} = e(V_{form} - V_{in})$$

giving the entry velocity

$$u = \sqrt{\frac{2e(V_{form} - V_{in})}{m}}$$

For an ion to transit the energy filter in the following region, it must have eV_{axis} kinetic energy. The exit velocity from this region is therefore

$$v = \sqrt{\frac{2eV_{axis}}{m}}$$

If the length of the region is s_{ext}, we can say

$$s_{ext} = \frac{u + v}{2} t_{ext}$$

where t_{ext} is the transit time.

Therefore

$$t_{ext} = \frac{2s_{ext}}{\sqrt{\dfrac{2e(V_{form} - V_{in})}{m}} + \sqrt{\dfrac{2eV_{axis}}{m}}}$$

$$t_{ext} = s_{ext}\sqrt{\frac{2m}{e}}\left(\frac{1}{\sqrt{V_{form} - V_{in}} + \sqrt{V_{axis}}}\right)$$

and substituting for V_{in}

$$t_{ext} = s_{ext}\sqrt{\frac{2m}{e}}\left(\frac{1}{\sqrt{V_{form} - V_{ext} - V_{ref}} + \sqrt{V_{axis}}}\right) \qquad (2)$$

Energy analyser region

To pass through the energy analyser, Ions must transit this region with a kinetic energy of eV_{axis}, which gives a velocity

$$v_{en} = \frac{s_{en}}{t_{en}} = \sqrt{\frac{2eV_{axis}}{m}}$$

$\therefore$ transit time

$$t_{en} = s_{en}\sqrt{\frac{m}{2eV_{axis}}} \qquad (3)$$

Mass filter region

Ions transit this region with an energy defined by the *Transit Energy* voltage V_{te} which is adjustable. Their transit time is therefore

$$(4)$$

$$t_{mass} = s_{mass}\sqrt{\frac{m}{2eV_{te}}}$$

Detector region

Ions enter this region with 3 eV energy and are accelerated to detector potential before they strike the conversion dynode (1st dynode). For this analysis, the entry energy is taken as zero, which will not cause a significant error.

Since ions are converted to electrons at the first dynode, they will only be accelerated by V_{dyn}, and the force on the ions is

$$\frac{eV_{dyn}}{s_{det}}$$

and the acceleration of the ions is given by

$$f = \frac{eV_{dyn}}{ms_{det}}$$

using

$$s = ut + \frac{ft^2}{2}$$

where s is distance and u initial velocity.

Assuming the initial velocity is zero and substituting gives

$$s_{det} = \frac{eV_{dyn}t_{det}^{\,2}}{2ms_{det}}$$

$$\therefore \quad t_{det} = s_{det}\sqrt{\frac{2m}{eV_{dyn}}} \qquad (5)$$

Transit time

Substituting (2) (3) (4) and (5) in (1)

$$t = s_{ext}\sqrt{\frac{2m}{e}}\left(\frac{1}{\sqrt{V_{form}-V_{ext}-V_{ref}}+\sqrt{V_{axis}}}\right) + s_{en}\sqrt{\frac{m}{2eV_{axis}}} + s_{mass}\sqrt{\frac{m}{2eV_{te}}} + s_{det}\sqrt{\frac{2m}{eV_{dyn}}}$$

Using e = 1.6x10^{-19} C, proton mass = 1.6x10^{-27} kg and M in amu gives

$$t = \frac{\sqrt{M}}{10000}\left(s_{ext}\frac{\sqrt{2}}{\sqrt{V_{form}-V_{ext}-V_{ref}}+\sqrt{V_{axis}}} + s_{en}\sqrt{\frac{1}{2V_{axis}}} + s_{mass}\sqrt{\frac{1}{2V_{te}}} + s_{det}\sqrt{\frac{2}{V_{dyn}}}\right)$$

For EQP/EQS probes, the flight distances s_n are

Distance	Standard probe	Extended (321) probe	xtended (400) probe
s_{ext}	0.0588 m	0.0588 m	0.0588 m
s_{en}	0.2646 m	0.3716 m	0.4506 m
s_{mass}	0.1778 m	0.1778 m	0.1778 m
s_{det}	0.0415 m	0.0415 m	0.0415 m